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Scientific International Journal of the World Academy of Materials and Manufacturing Engineering publishing scientific monographs in Polish or in English only

Published since 1998 as Studies of the Institute of Engineering Materials and Biomaterials

Volume 6, 2011

Anna D. DOBRZAŃSKA-DANIKIEWICZ (ed.)

Materials surface engineering development trends

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Patronage

World Academy of Materials and Manufacturing Engineering

Association of Computational Materials Science and Surface Engineering

Institute of Engineering Materials and Biomaterials of the Silesian University of Technology, Gliwice, Poland

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Publisher

INTERNATIONAL OCSCO WORLD PRESS

International OCSCO World Press
Gliwice 44-100, Poland, ul. S. Konarskiego 18a/366
e-mail: info@openaccesslibrary.com

Bank account: Stowarzyszenie Komputerowej Nauki o Materiałach i Inżynierii Powierzchni
Bank name: ING Bank Śląski
Bank addres: ul. Zwycięstwa 28, 44-100 Gliwice Poland
Account number/ IBAN CODE: PL 76105012981000002300809767
Swift code: INGBPLPW
Gliwice

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SOURCE OF FUNDING:

The monograph has been published in relation to the project POKL.04.01.01-00-003/09-00 entitled "Opening and development of engineering and PhD studies in the feld of nanotechnology and materials science" INFONANO, co-founded by the European Union from fnancial resources of European Social Fund.

HUMAN CAPITAL
NATIONAL COHESION STRATEGY

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EUROPEAN UNION
EUROPEAN
SOCIAL FUND

ISSN 2083-5191
ISBN 83-89728-95-8
EAN 9788389728951

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Abstract

Purpose: *The purpose of the monograph is to provide scientific workers and students with comprehensive knowledge on the original methodology of computer aided prediction of development trends in materials surface engineering together with the examples of own materials science-heuristic research used for reviewing the correctness of the newly developed methodology.*

Design/methodology/approach: *The overall methodology of the integrated computer aided prediction of development trends in materials surface engineering embraces also the methodology of interdisciplinary materials science-foresight-IT research including a group of originally matched, but commonly known analytical methods and tools as well as an original methodological concept, allowing to pursue further research, that encompasses context matrices, the Critical Technologies Book of materials surface engineering and the neural networks-aided creation of alternative scenarios of future events.*

Findings: *The substantive assumptions of the methodology of computer aided prediction of materials surface engineering development and the examples of reviewing such methodology's correctness, performed under own works together with a state of the art review, with special consideration given to its importance for the research performed related to the integrated information technology including: virtual organisation, web platform and neural networks and importance of the concept of technology e-foresight and technology e-transfer.*

Research limitations/implications: *The newly established methodology has the broad prospects of future applications. It can be applied in all kinds of technology, thematic and environmental foresights, as well as in other areas of computer aided knowledge and information management as an approach enabling to utilise the currently available economic, system-related, technological, financial and social potential for fulfilling the strategic development objectives.*

Practical implications: *The purpose of the newly developed concept of technology e-foresight is to disseminate the results of technology e-foresight of materials surface engineering in SMEs lacking the funds to undertake own research in this filed.*

Originality/value: *The monograph describes the substantive assumptions of an original methodology of integrated, computer aided prediction of materials surface engineering development and the examples of reviewing such methodology's correctness made under own materials-science and heuristic works. An original value is also represented by the concept of integrated information technology, technology e-foresight and technology e-transfer.*

Keywords: *Materials surface engineering; Development trends; Computer integrated development prediction methodology; Technology e-foresight; Technology e-transfer*

Reference to this monograph should be given in the following way:

A.D. Dobrzańska-Danikiewicz (ed.), Materials surface engineering development trends, Open Access Library, Volume 6, 2011, pp. 1-594.

1 Introduction on the materials surface engineering development trends

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This extensive monograph is comprised of more than ten cohesive chapters concerning technology foresight for materials surface engineering and foresight-materials science research and is currently made available to scientific workers and students in order to disseminate the methodology of this innovative approach for technology development forecasting. The chapters were previously published in 2009-2011, as research monographs, as the reference journals of the Archives of Materials Science Engineering and Journal of Achievements in Materials and Manufacturing Engineering included in the Directory of Open Access Journals.

The first part of the monograph presents the general approach to the issues analysed, with particular consideration given to the scientific and utilitarian objectives of the relevant tasks. The tasks are fulfilled as part of the research conducted and are defined for achieving milestones. Special consideration is also given to socio-economic conditions, methodological aspects and the expected indirect and final effects of the research efforts undertaken. The development state analysis is carried out separately and includes issue state assessment, technology review and a strategic analysis with integrated methods. The concept of **e-foresight**, i.e. the process of foresight research conducted by means of the Internet and information technologies embracing a virtual organisation, web platform and neural networks is also analysed separately. This approach organises, streamlines and modernises the foresight research pursued on a large scale.

The custom **computer integrated development prediction methodology** is of fundamental importance for all the issues presented. The data indispensable for performing the foresight part of the research has been gathered by surveying experts to collect the experts' explicit as well as implicit knowledge. In line with the assumptions adopted, such knowledge should be converted, through the research, into explicit knowledge. The experts were assessing the technologies and the micro- and macro-environment influencing such technologies using a universal scale of relative states being a single-pole positive scale without zero, where 1 is a minimum rate and 10 an extraordinarily high rate. According to the methodology established, a strategic position of each of the analysed critical technologies of materials surface engineering is presented graphically using the **matrix of strategies for technologies** made up of sixteen fields. The matrix presents, graphically, a position of each group of technologies according to its value and environment influence intensity together with the forecast strategic development tracks and identifies a recommended action strategy. The matrix contains the results of the expert investigations visualised with the dendrological and meteorological matrix transformed by means of software created for this purpose. The methodological structure of the both matrices is referring to the portfolio methods commonly known in management sciences, and first of all to BCG matrix enjoying its unique popularity due to a reference to simple associations and intuitive reasoning, becoming an inspiration when elaborating methodological assumptions for the both matrices. A four-field **dendrological matrix of the technology value** includes the expert assessments for the relevant technologies according to the potential being the actual objective value of the specific technology group and to attractiveness reflecting the subjective perception of the relevant technology group by its potential users. A four-field **matrix of environment influence** presents, in a graphical manner, the results of how the external positive (opportunities) and negative (difficulties) factors influence the technologies analysed. A presentation of the methodology assumptions was backed up with a theoretical example illustrating the practical application of a set of matrices with reference to laser remelting, passivation, detonation spraying and PVD multilayer coatings deposition. Classical materials science investigations into the structure of surface layers and mechanical, tribological and functional properties, performed with specialised diagnostic and research apparatuses, represent an inherent part of the materials science and foresight research conducted. The relevance and adequacy of the assessments performed according to the methodology developed is ensured by the synergic interaction of the materials science research and foresight methods. Technology roadmaps developed according to the custom setup are a tool of comparative analysis and they are technically supplemented by technology information sheets.

A series of **materials science and heuristic investigations** was carried out to verify whether the methodology developed is correct. The investigations were concerned, respectively, with the following critical groups of materials surface engineering technologies: laser alloying/cladding of carbide and oxide particles into the surface of casting magnesium alloys, laser alloying of alloy tool hot-work steels, physical vapour deposition (PVD) of coatings onto the brass substrate, physical and chemical vapour deposition (PVD/CVD) of coatings onto sintered tool materials, the selected thermochemical steel treatment technologies, texturisation of polycrystalline silicone, production of graded tool materials with the conventional powders metallurgy method, the selected modification technologies of polymeric materials surface layers. The results of detailed investigations into the structure and properties of the materials subjected to surface treatment are presented for each of the technology groups mentioned, as well as the results of foresight research including a set of foresight matrices with the recommended strategies of long-term development and strategic development tracks established for the three alternatives: optimistic, neutral and pessimistic. The results of the foresight–materials science research conducted constituted reference data serving to create technology roadmaps being a tool of a comparative analysis enabling to select technologies or a group of technologies best for the criterion chosen. The **technology roadmaps** developed with a custom concept are a very convenient tool of a comparative analysis enabling to select the technology best in terms of the specified criterion. Besides, their flexibility is their undisputed advantage, and, if needed, additional sub-layers can be added to or expanded for the maps according to the circumstances of the industry, size of an enterprise, scale of the company's business or an entrepreneur's individual expectations. **Technology information sheets,** containing technical information very helpful in implementing a specific technology in the industrial practice, especially in the SMEs lacking the capital allowing to conduct own research, are detailing and supplementing the technology roadmaps.

A framework bridging this monograph is a presentation of the **neural networks aided future events scenarios** shown by an example of laser surface treatment. The scenarios of future events concerning the development of materials surface engineering are considered at the three levels of generality corresponding to the extent of the phenomena analysed, taking into account their number and the intensity of influence on the other phenomena. The scenarios, at a macro scale, are developed according to the three alternatives: optimistic, neutral and pessimistic. **Neural networks** were used in a novel and experimental manner to cross impacts analysis. The analysis serves to identify how the key mezofactors of surface engineering development (e.g. collaboration between science and industry, number of specialised laboratories and R&D

institutions, continuous improvement and high quality of technology, transparent and friendly legislation, international co-operation and EU funds) and the relevant thematic areas analysed (e.g. laser technologies, thermochemical technologies, nanotechnologies) may influence the occurrence of each of the macroscenarios. A data set elaborated according to the results of survey investigations was divided randomly into the three sub-groups: learning, validation and testing sub-group. The data from the learning set was used for modifying network importance in the learning process and the data from the validation set was used for network evaluation in the learning process. The remaining part of the data, as a test set, was used to determine, independently, network efficiency after completing fully the network development procedure. The following values were used as the basic indicators of model quality evaluation: an average absolute error of network forecast, a standard deviation of the network forecast error, R Pearson's correlation coefficient for the value set and for the value obtained at the neural network output. The quality evaluation indicators of artificial neural networks were calculated for each of the separated sets. The similar values of the average error, standard error deviation and correlation coefficient confirm the generalisation ability of the network, i.e. an ability to generalise the knowledge acquired in the learning process.

9 models were created altogether using artificial neural networks by adopting, as dependent (input) variables, the probabilities of the occurrence of a growth trend, stabilised trend and/or declining trend determined for the key mezo-factors conditioning the development of materials surface engineering and for the individual thematic areas for the research domain of M (Manufacturing) and P (Product). Dependent (output) variables represent the probabilities of the occurrence of each of the three macroscenarios considered. The outcomes of the simulations performed are shown in the charts created by software created for this purpose, i.e. SCENNET48 and SCENNET21. The development trends of the 14 thematic areas analysed (mezo scale) were formulated according to the results of a statistical analysis. The results were obtained based on the results of three iterations of the Delphi method and were presented as a description and as charts illustrating, in percents, to what degree the experts surveyed agree with the theses put forward. Development trends for the relevant groups of the critical materials surface engineering technologies (e.g. laser alloying) were determined at the lowest level of generality (micro scale) for which technology strategy matrices, technology roadmaps and technology information sheets were created. Development trends were also formulated for specific technologies (e.g. laser alloying of alloy tool hot-work steels using powders of titanium

carbides), subjected to the foresight and materials science research using the custom methodology described earlier in the introduction. The extensive approach proposed, pursued at the different levels of generality, is a inverse task. A synthesis consists of seeking a solution being such a combination of micro- and mezofactors which, with the greatest probability, contributes to the emergence of an optimistic or at least neutral macroscenario of future events.

By making this monograph available to the Readers I believe it will contribute to an improvement in the standard of methodological knowledge among students and scientific workers, and will encourage them to initiate their own work and research in the future in the field of technology foresight devoted to the specific areas of material engineering and production other than surface engineering. I also believe that, on the other hand, it will contribute to enhancing the skills of using the results of the foresight research performed for supporting the decision-making process for the development of an individual enterprise, industry or overall economy. A general improvement in the culture of decision-making by utilising the area of knowledge management presented in this monograph, with this area being, unfortunately, sometimes underrated by decision-makers in the field of economy and technology, may have considerable input into economic development at a an enterprise-wide, national or international scale.

I wish to express my kind appreciation to the Group of my Friends and Colleagues who have performed, together with me, the research discussed in this monograph as this multi-faceted and long-term task would not have been implemented without their assistance, involvement and very precious discussions. I would also like to thank the persons who have supported me in pursuing the set objective, including the Reviewers of the individual chapters and the Persons who have held discussions with me during many scientific conferences I have had an opportunity and was privileged to attend and during which I was able to present the outcomes of the works carried out. The discussions were encouraging me and the Group of Co-authors of this monograph cooperating with me to continue further work. Last but not least my appreciations go to the Persons who have supported me with their technical, editorial and translation assistance in preparations to publish this monograph.

2 The state of the art analysis and methodological assumptions of evaluation and development prediction for materials surface technologies

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Abstract

Purpose: *The purpose of the chapter is to present an analysis of the state of the art including the general development trends and most prospective areas of materials surface engineering and to describe the general methodological concept for the evaluation and prediction of materials surface technology development, with special consideration given to methods for generating a pool of critical materials surface technologies.*

Design/methodology/approach: *The chapter was prepared by reviewing the international literature devoted to the latest trends in materials surface engineering and discusses the general methodological assumptions for the research conducted under the technology foresight of materials surface engineering.*

Findings: *Presentation of the most important trends, directions and areas of materials surface engineering and of the general methodology for the evaluation and prediction of materials surface technology development.*

Research limitations/implications: *The state of the art analysis and the methodology presented form constituent part of the technology foresight of materials surface engineering.*

Practical implications: *One of the final effects of the technology foresight of materials surface engineering is to establish the Critical Technologies Book comprising technology roadmaps and technology information sheets. The Book is characterising, in a harmonised fashion, the critical materials surface technologies, which is a convenient tool of comparative analysis, especially for SMEs lacking the funds sufficient to pursue their own research in this field.*

Originality/value: *The chapter is presenting the general development trends and most prospective areas of materials surface engineering and an original, newly established customs, methodological concept for the evaluation and prediction of materials surface engineering development.*

Keywords: *Surface engineering, technology foresight, the state of the art, development trends, evaluation methodology*

1. Introduction

It is a common expectation these days that materials be manufactured possessing the properties ordered by product users [1, 2]. This is significantly influencing the development of the products material design methodology as it is expected that materials are delivered having the required structure and physiochemical properties, meeting functional requirements, matching customer demands and usable functions of a product, i.e. so-called materials on demand. The functional properties of products can often be enhanced by forming the structure and properties of engineering materials surface layers [3-7], as their structure and properties are often decisive for the applicational prospects of many products. The mere transmission of mechanical loads through the entire active section of a part or its physiochemical properties are frequently not of overriding importance only. Other requirements relevant for surface properties, i.e. notably their smoothness or texture and roughness, colour and coating aesthetics, transparency, reflectivity or the absorptivity of light radiation, heat or sound waves, ability to absorb or not to absorb liquid or air, wear resistance, tenacity, ability to imitate other materials, e.g. wood, leather or glass [8] − apart from investigations into the structure of materials and their basic mechanical properties − are to a large extent decisive for the selection of technological processes for both, structural parts, tools, and other products made of, respectively, all groups of engineering materials.

An exhaustive overview of the contemporary treatment technologies decisive for the formation of the structure and properties of engineering materials surface layers, including also those exhibiting a nanometric structure, together with a general view on the current state of the art on the basis of analyses into the basic literature data and prior own research [9-17] is presented in an own book publication [18]. The publication [6] presents, on the other hand, the evaluation of the relevant material surface treatment technologies. The monograph [19] presents, however, the applications of materials science-foresight methods for predicting the development of the selected engineering materials groups for which selected surface treatment methods have been used, carried out to some extent as part of own research [20]. The culmination of all such efforts is an own methodological monograph [21] pertaining to the computer integrated development prediction methodology in surface engineering area.

This chapter describes the state of the art in the field of surface engineering and discusses a method of evaluating the state of the art of the technology in order to develop basic information enabling to pursue foresight research.

2. General development trends and most prospective areas of materials surface engineering

Surface layers can be classified as surface layers limited with the area of the part treated and encompassing the area of the material, with their structure and properties differing from the properties of the core, or as coatings being a layer of a material differing most often in its phase and chemical composition and the structure from the core, deposited permanently onto a substrate surface. Six groups are differentiated between for the surface layers production technologies according to the surface formation method, i.e. [3, 4, 18]:

- mechanical methods connected with the cold reinforcement of a surface layer or production of a protective coating on a cold substrate;
- thermomechanical methods employing heat influence and pressure together;
- thermal methods connected with the change of material structure as a result of heat impact both, in the solid state as well as by changing the state of concentration from solid into liquid and inversely;
- thermochemical methods as a result of the aggregated impact of heat and a chemically active centre;
- chemical and electrochemical methods connected with the deposition of a metallic or non-metallic material onto part surface, removal of the damaged surface layers, thermal and chemical solidification of film formers or curing of chemically setting resins;

- physical methods, achieved through the adhesion deposition of coatings and with the presence of diffusion joints.

Surface layers may have varied intended uses, exhibiting the required physical, mechanical and tribological properties, electrical or heat conductivity and temperature resistance, anticorrosive properties, including such representing a diffusion barrier; decorative and protective-decorative properties and such providing an aesthetic appearance and the required surface texture of products. The most beneficial properties of the core and the surface layer of the part produced, ensuring its required functional properties, can be obtained by selecting such core material and technology that ensure the part's properties (e.g. heat treatment or heat and plastic treatment) and by choosing at the same time the surface layer technology decisive for the functional properties of many products and parts thereof.

The tailoring of properties of different items produced by means of different engineering material groups, including structural, tool, functional and biomedical materials, to operational requirements with surface engineering methods has been used more and more often in many industries [2, 22-31]. The industries include the machine and tool construction, mechatronic, automotive, power engineering, aviation, polymer material processing, construction, medical equipment, sanitary fittings, electrical engineering electronic and jewellery sector. The scientific institutions around the globe are expressing their on-going and intensifying interest in this field, e.g. [32-46]. The basic literature data concerning the state of the art for the formation of surface layers structure and properties was analysed, therefore. The state of the art of the classical surface layers structure and properties formation technologies for the relevant groups of engineering materials has been well known and described in the basic domestic [7, 47-53] and foreign [8, 54-59] handbooks on material engineering and surface engineering. Moreover, numerous monograph papers and reviews [60-73] have been published. Research projects and numerous publications [74-93] in this thematic domain discuss a great deal of details concerning the phenomena and mechanisms in different materials being an outcome of surface treatment and its impact on the properties of diverse products. The information available in the literature relates to over 500 specific surface treatment technologies and their numerous technological variants with respect to all the basic groups of engineering materials. This aspect is of major economic importance and does not only relate to the avant-garde technologies implemented in leading, large corporations, but requires first of all that the quality and durability of most market products is improved by applying a broad array of available surface engineering technologies by the majority of industrial manufacturers, and especially by small- and medium-sized enterprises. For this reason the technologies used are identified along with their desired direction of development and products are indicated for which they should be applied. In addition, the development trends of the priority innovative technologies of engineering materials surface structure and properties formation and the directions of strategic research in this regard is of fundamental importance for economic growth in the coming decades and is decisive for the competitiveness of the domestic economy. Surface layers, for technological considerations, can be grouped as given in Table 1 and Figure 1.

Table 1. *Classification of surface layers according to technological considerations [18]*

Possible types of surface layers and coatings or processes occurring in the substrate surface		
monolayer	multiphase	amorphous
multilayer	graded	nanocrystalline
multilayer (>100 layers)	composite	hybrid
phase transformations of substrate surface layers	change of chemical composition of substrate surface layers	physical processes on substrate surface

Whilst analysing, in the course of own works [9-17, 94-108], the current state of the art for the development of the surface engineering technologies described in the literature against the macro- and micro-environment, the technologies were classified according to several thematic areas defined in an arbitrary manner, according to the phase of their development within the technology life cycle. The technologies analysed were classified as: base, key, experimental or embryonic technologies. A ranking was established of potentially critical technologies evaluated in a ten-degree universal scale of relative states by their attractiveness and potential. The selected critical technologies meeting at the same time in at least 50% the requirements of attractiveness and potential were considered only in the further literature analyses, and some of them were chosen on such basis to conduct own, supplementary research in the field of materials science.

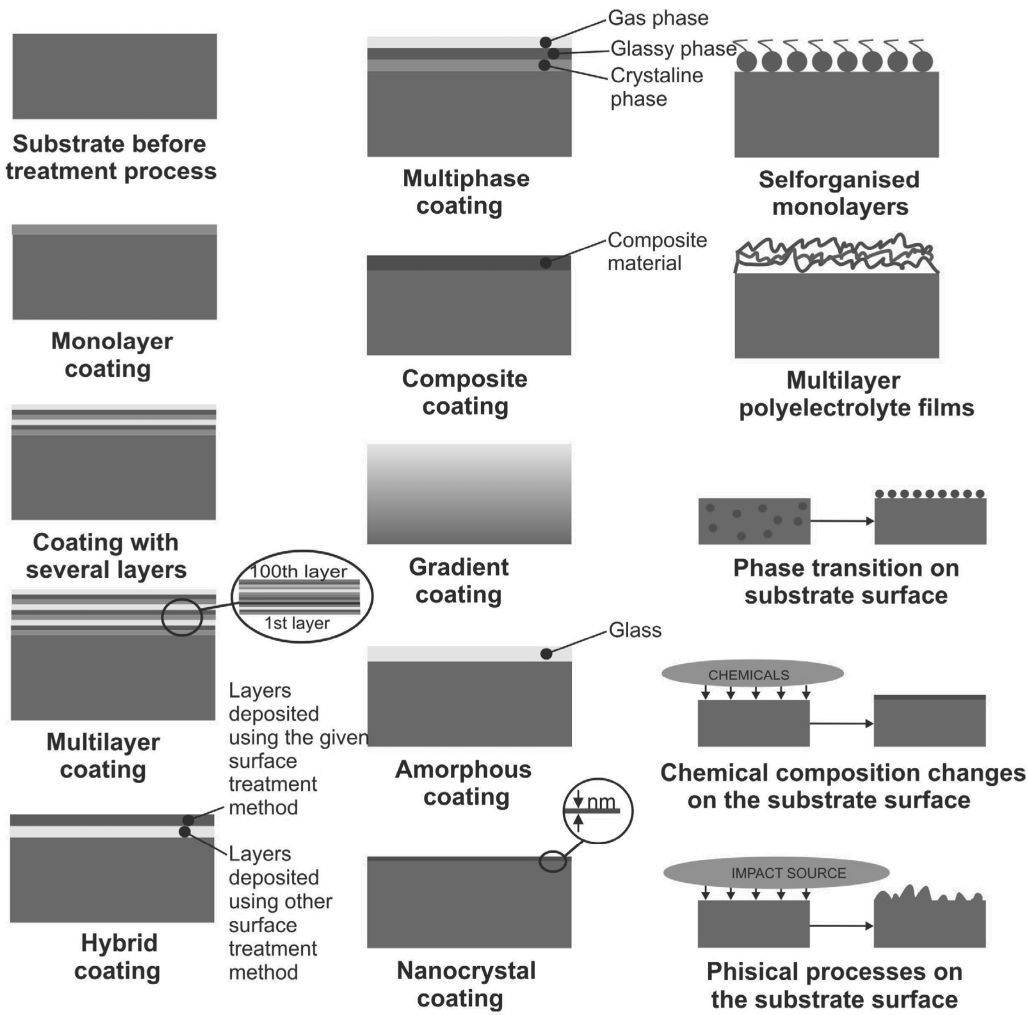

Figure 1. *Schematic examples of surface layers and processes occurring on the substrate surface according to technological conditions [21]*

Avant-garde technologies and such featuring extremely high development prospects are becoming especially vital in this context. This group includes a continuously developed concept of functionally graded materials (FGM) and tool graded materials (TGM) [15, 85-86, 109-115], new coating formation nanotechnologies for coatings several millimetres thick, sometimes composed of several dozens to several hundreds of layers, including multilayer and graded deposition with the PVD and CVD methods, but also composite coatings with a polymeric matrix reinforced with carbon or mineral nanotubes [116-120]. Extensive development prospects are also true for hybrid technologies ensuring the best possible properties of surface layers deposited subsequently on one another [121-124], as a result of combining some classical surface structure and properties formation methods (e.g. thermochemical treatment) with avant-garde technologies (e.g. laser alloying, remelting or cladding, powder injection moulding and physical vapour deposition- PVD). The fabrication of layer structures is also note-worthy, in particular with thermochemical treatment methods [74-77, 125], by producing composites [126-127] and through monolayer deposition with the PVD [128, 129] and CVD methods, and also cladding or sputtering the hard layers with the thermal sputtering method, laser surface treatment technologies (including alloying, remelting and cladding ensuring very high quality of the bond with the substrate and production of graded layers with high corrosive and wear resistance, high heat resistance and hardness and ductility, plasticity and fatigue strength) [35, 130, 131] as well as the laser texturisation of surface of parts of solar cells [14, 108, 132].

The permanent works conducted in foreign entities [109-110, 133-136] and developed also in Poland under the research projects [137-139] have laid the groundwork for the development of functional graded materials (FGM) with their properties changing in an abrupt way or continuously according to the position due to changes to chemical composition, phase composition, structure or due to harmonising atoms in a crystalline network. A lot of technologies were used for achieving the above structural outcomes, including powder metallurgy [86, 140-142], welding methods and laser treatment [34, 35, 87, 130]. An improvement in properties, by combining the very high resistance of tools surface to abrasive wear with large core ductility, is secured by tool graded materials (TGM). This group is becoming increasingly independent and more and more often considered as a separate group of engineering materials, developed to the same extent as functional graded materials (FGM), despite still being rarely used in the industrial practise [3, 4, 144]. The surface layers of graded tool materials with varying chemical or phase composition have the thickness of 10^{-3}-10^{-6} m, depending on the production technology.

One of the basic groups of technologies of producing graded tool materials is represented by the physical vapour deposition (PVD) of 3-5 μm thick graded coatings and changing constantly one or several of its components starting with the substrate up to the surface, onto the substrate made of conventional or sintered tool materials [33, 36, 142, 144, 145]. PVD coatings, sometimes only graded coatings, are used not only for depositing tool materials [146-150], but also in machine construction, automotive, aviation, space, construction, power and housing sector, optics, microelectronics and biomedicine [13, 26, 104, 151], to enhance durability, limit the rate of wear, improve resistance to high temperature, lower thermal conductivity and reduce corrosive processes [144, 152-154]. The PVD methods are also used as metal implants [155]. The following types of PVD coatings are distinguished:

- monolayer simple or complex coatings, including multicomponent coatings with a sublattice of one element partly filled with another element, usually as continuous solid solutions of carbides and nitrides;
- multilayer coatings produced by depositing the layers of different materials onto each other, usually simple coatings with different properties [156],
- multiphase coatings representing a mixture of different phases, characterised by high resistance to abrasive wear;
- graded coatings as a variant of multilayer coatings with their chemical composition and properties changing in a constant manner across the thickness [146, 147], including gradient wear-resistant coatings (GWRC) acting as gradient thermal barrier coatings (GTBC);
- composite coatings as a variant of multiphase coatings in which another phase is dispersed in one continuous phase, metastable with combined properties distinctive for materials featuring metallic and convalescence bonds as a result of solid solution strengthening of coatings.

High-temperature PVD techniques ensure the better quality of a coating's diffusion-adhesion joint to the substrate. PVD processes can be grouped into the techniques of classical deposition of pairs of metal in a vacuum or in the atmosphere of non-ionised gas on a clean and cold substrate, where the pairs of metal with low energy settle on the substrate, thus creating coatings with low density, considerable contamination and poor adherence as well as the techniques of ion vacuum deposition, most often reactive on a clean but cold or heated substrate, where the substrate is bombed with a stream of ions with their energy causing sputtering and the related shallow implantation, thus forming coatings with their high density, with no contaminants and with good adherence to the substrate. The very good adherence of PVD coatings to the substrate is caused by the adhesion and diffusion of the elements forming the coatings to the substrate in the direction of the substrate and of the elements contained in the substrate material (e.g. carbon and nitrogen) in an opposite direction, i.e. to the coating. As adhesion and diffusion occur at the same time, it is possible to produce a hybrid graded surface layer with the total thickness of up to 100 μm on the substrate made of, e.g. high-speed steels or tool alloy steels using the PVD technology with prior classical thermochemical treatment, e.g. ionising nitriding [3, 4].

Graded tool materials are also produced through chemical vapour deposition (CVD), when the coatings of carbides and nitrides of metals are formed on the surface of the treated product. They are made of the components of the gaseous atmosphere consisting of volatile halide of a diffusing element and of hydrocarbon, nitride, hydrogen or inert gas, e.g. argon, that are activated with heat or plasma, with the presence of substrate components, in a seal-tight reactor as a result of inhomogeneous, chemically and physically catalysed reactions on the substrate surface at the temperature of approx. 900-1100°C and at the pressure of $1 \bullet 10^5$-$1.35 \bullet 10^3$ Pa. The high temperature of a CVD process causes limitations to its applications to include the plates made of sintered carbides or sintered ceramic materials, which, historically, are deposited most early with single layers, then dual layers, and now usually multilayers [24, 157]. Sintered carbides deposited with multilayers are characterised by much higher wear resistance as compared to conventional grades with just slightly deteriorated ductility [148, 158, 159].

Graded materials, including tool materials, but also such with the substrate made of alloys of light metals, i.e. magnesium [9, 106, 107, 160] or aluminium, may be produced using appropriate laser remelting, cladding and alloying [143, 161-163]. The materials treated this way are reinforced by means of one or several mechanisms working in a synergy. One of the mechanisms is crystallisation and distribution of the structure of the surface layer remelted by laser and undergoing remelting hardening in the zone. The material, after hardening from the liquid state, is characterised by its dendritic morphology and the martensitic structure and the martensite laths are 1.5 to 2 times smaller than after conventional hardening [143]. Other mechanisms contributing to the reinforcement of materials as a result of laser treatment include: the dispersion hardening of the surface layer by melted or partially dissolved particles of the phases introduced; enriching the surface layers with alloy additions coming from the dissolving phases and precipitation hardening by the newly precipitated phases, e.g. carbides in case of tools steels and carbide steels [162].

This aspect is related to the new directions of laser formation of the structure and properties of light metals alloys surfaces, in particular multicomponent low density alloys of magnesium that different industries are interested in, among others, the aviation, automotive, power, electronic, chemical, nuclear, sports equipment, office devices and household appliances industry. The attractive technological options include the treatment of such alloys' surface ensuring the formation of an approx. 0.5 mm thick layer remelted by laser and next subjected to fast micro- or nanocrystallisation enabling to achieve the structure and properties the same as those achieved with the Rapid Solidification Processing (RSP) method. The laser treatment of such alloys' surface also enables to clad dispersive ceramic particles into the remelted surface layer, hence ensuring its properties corresponding to metal matrix composites (MMC) with a magnesium alloys matrix [106, 160, 164] and is considerably enhancing the durability of the products fabricated with such technology [107, 164].

Another promising laser technology requiring the use of low-power lasers is the texturisation of polycrystalline silicon used for producing solar cells. Laser texturisation is employed to develop the surface of polycrystalline silicon in the parallel or grid configuration. Texturised samples must undergo wet etching in alkali solutions to obtain the expected electrical properties of solar cells produced of silicon. The other methods of poly- and monocrystalline silicon surface treatment include: Reactive Ion Etching (RIE), Anti-reflective Coatings (ARC), including Plasma Enhanced Chemical Vapour Deposition (PECVD) and classical Chemical Vapour Deposition (CVD), thermal oxidisation [14, 108, 165, 166], sol-gel methods [167] and Atomic Layer Deposition (ALD). One of the more promising possibilities of fabricating photovoltaic cells is a thin silicon layers technology, which is several millimetres thick, with transparent oxide ZnO layers dispersing light and conductive with the thickness of several hundred nanometers. The layers can be obtained with the chemical vapour deposition or physical vapour deposition method in the conditions of a lower pressure or with the aid of plasma, produced notably using the Hall accelerator being a source of low-temperature and thin plasma [168].

The welding methods of material surface layers formation include the deposition of a layer of the hardfaced metal onto the surface or edge of the substrate material with different chemical composition, using gas and arc technologies (MMA, GTA, SSA, GMA and PTA), energy of beam of electrons and arc thermal spraying or plasma spraying. The welding methods allow to produce − on a surface made of relatively cheap, readily available materials not subjected to structural changes or plastic deformation – coatings made of metal alloys, ceramic materials, materials based on intermetallic phases, cermets, carbides or polymer materials. Such coatings ensure anticorrosive protection, surface hardening, surface porosity, decorative effects, electrical conductivity, reflection of light and/or repair of surface recesses [3, 4, 18, 64]. The coatings sprayed thermally, containing Co, Ni, Cr, Al, Y, Si or Hf or phases containing some of such elements and thermal barrier coating (TBC) protect high-temperature nickel alloys by creating heat insulation [169]. Surface layers can also be deposited by detonation [5, 6, 18].

One can also use graded tool materials produced with the traditional powder metallurgy method. The method enables to, in particular, manufacture tool graded materials based on the high-speed steel matrix with the tungsten carbide reinforcing phase and carbide steels based on the cobalt matrix with the tungsten carbide phase. By using the powder metallurgy method, a surface layer can be provided with high abrasive wear resistance ensured by the hard carbide, nitride and/or oxygen phases or powders of higher-alloy high-speed steels whilst maintaining high core ductility typical for high-speed steels and traditional carbide steels at relatively low costs. Such material structure allows to formulate properties freely depending on the tool working conditions. A layer of hard material is used for example in those locations at the tool being exposed to wear while high ductility is ensured in those locations of the tool material that are susceptible to impact [15, 85, 114, 136, 170]. Considering the techniques of formulating the powder mixtures of functional graded material, classical pressing should be distinguished in a closed mould by filling moulds sequentially with the mixtures of powders, with the presence of hard ceramic phases rising towards the surface of the tool. The other techniques applied include: pressing in a uniaxial, one-sided mould, isostatic cold pressing, low-pressure moulding of polymer and powder slurry, vibration moulding and densing in a closed mould, sedimentation moulding and low-pressure moulding of polymer and powder slurry [171].

Composite surface alloy layers on the cast steel or cast iron parts of machines can be produced immediately in the process of flooding dies with the appropriate liquid cast steel or cast iron with a composite material situated in such moulds by matching the dimensions, sprigging or attaching it in the designated parts of the cavity interior. After filling a mould with liquid metal, the metal undergoes infiltration into a composite material that may occur as perforated inserts made of powders of the selected alloys or carbide or oxide reinforcing phases. Another possibility is to use a self-fusible composite with epoxy resin, organic limited solvent or water glass bond [172]. A composite can also be placed in a mould without the presence of bond by means of a magnetic field [173]. The casting surface composite alloy layers are used in the industry on casts resistant to abrasive wear, working in the conditions of dry abrasion caused by, e.g. hard coal, lignite and construction materials. A method combining the casting techniques (infiltration process) and powder metallurgy (preparation of porous sintered skeletons) is the pressure infiltration of porous sintered skeletons, being one of the fastest developing composite materials manufacturing methods [174, 175], enabling, in particular, to reinforce casts locally at their surface.

Surface treatment with polymer materials is especially significant. The treatment relates to the polymer coatings made of thermoplastics, hardening plastics and elastomers, deposited with various technologies on different other materials, e.g. on metal alloys and textile materials.

The treatment also concerns the formation of the structure and surface properties of different polymer materials, including the fabrication of layer composites, including polymer material laminates and polymer layer materials obtaining the desired usable properties of the surface [176]. Polymer coatings can be deposited by evaporating polymers dissolved in solvents, by sputtering polymer powders onto a hot substrate and through electrostatic spraying onto the substrate with a negative electrical potential. The other known methods include: electrolytic deposition from aqueous polymer solutions, deposition of immersion particles heated in steam in a fluid bed, flame spraying by depositing the powder of a polymer material melted by hot gases onto a heated substrate and plasma polymerisation. The following types of polymer layers with different properties can be distinguished: hydrophilic and hydrophobic layers preventing the deposition of sediments; adhesive or anti-adhesive layers; light protective layers; antidiffusive layers and layers resistant to scratches [67]. The formation of the structure of surface properties of polymer materials is most often preceded with pre-treatment, e.g. in case of polyolefin (e.g. polyethylene or polypropylene), polytetrafluoroethylene or polyacetals. In order to achieve the beneficial values of surface free energy, oxidisation with acids, flame activation or corona discharges with high-energy ions in a high voltage field under the atmospheric pressure of air is used prior to polymer bonding, painting, printing or metallisation. Activation in low-pressure plasma at the temperature of 60-100°C in reactors with the atmosphere of oxygen, nitride, fluorine or precious gases is used for thermoplastics and hardening plastics [67], with simultaneous ultraviolet radiation [177]. Ionising radiation, γ radiation of the C^{60} cobalt lamp or high-energy electron radiation is used [66], respectively, for the surface crosslinking of polyethylene for pipes, tapes, foils, for sterilising disposable polypropylene syringes, for polytetrafluoroethylene degradation, for changing the properties of insulation of polymer power cables and for improving the properties of rubber car tires. The surface properties of polymer materials in the electronic industry or in the manufacture of medical devices are formed through laser radiation [178]. Protective or decorative functions are fulfilled by painting the surface of polymer materials and by printing and decorating on printing machines, by electrostatic flocking, by metallisation or by rubbing antistatic agents, silicone oil or bactericides into polymer materials [67]. The mechanical properties of polymer surface layers can be improved by using carbon nanotubes, whereas the anisotropy in the arrangement of such nanotubes supports the improvement of such properties [179]. Composite polymer layers containing carbon nanotubes represent an active part of vacuum level and pressure nanosensors [180], in which changes to the material volume is directly impacting the electrical properties of an active element. Ultrathin surface films containing carbon nanotubes are developed in multiple applications, e.g. in gas sensors and biosensors [181, 182], differing in their thickness, the bonding material used and protecting nanotubes, and also in their structure (single-walled or multiwalled ones), in their purity and in the method of arrangement (random, ordered). Conductive or semi-conductive nanostructural coatings are produced as a result, applied in medicine, environmental protection, agriculture, automotive and food industry, and in industrial production [183]. The value of electrical conductivity is decisive for the application of a given composite as a component of surface layers with their insulation properties or properties allowing to evacuate an electrical charge safely. The value in some cases is higher than the electrical conductivity of the individual components of the composite, as e.g. in case of a polyaniline and multiwall carbon nanotubes of composite. Due to high thermal conductivity, surface layers made of polymer composites containing carbon nanotubes are used by the space and aviation industry as thermal shields. The conductive polymer coatings applied onto the wings of aeroplanes allow deicing during harsh weather conditions, and they replace traditional liquid heat evacuation systems on the external parts of cosmic crafts. Stresses can be determined by means of the Raman spectroscopy by introducing carbon nanotubes into a polymer matrix. It is because the shape of carbon nanotubes' spectrum (in particular the position of reflex equal to 2601 cm^{-1}) varies as a result of composite deformation [184, 185]. Polymer coatings containing carbon nanotubes with biocatalytic properties prevent foul odours, and are used for active elements of biosensors and as materials with higher biocompatibility [186-188]. Composite polymer layers containing carbon nanotubes can also be used for constructing solar cells [189]. As the conductivity of carbon nanotubes changes as a result of interacting with molecules of multiple chemical substances [190], they represent the elements of active sensors and electronic nanodevices in which sensitivity and selectivity of such detector increases as result of depositing the nanoparticles of precious metals, in particular Au, Pt, Pd, Rh onto the surface of carbon nanotubes [191-196]. A composite achieved as a result of coating the external surface of carbon nanotubes with a layer of conductive polymer can be used as a supercondenser [197]. The endeavours are also very promising in this field targeted at using polymer-matrix surface layers reinforced with mineral halloysite nanotubes as the substitutes of carbon nanotubes. As a result of depositing with conductive materials, they can also ensure a surface's thermal and electrical conductivity, and the general cost of such material can turn out to be much lower [198]. A prospective example of applying carbon nanotubes in surface engineering is also their use for producing ceramic nanotubes by depositing ZrO_2 onto their surface and then by oxidising in the temperature of above 800°C, as a result of which ceramic circles with the diameter of approx. 40-50 nm are developed on the surface of carbon nanotubes, with their walls approx. 6 nm thick [199]. The works carried out in this field are intended to establish the scope of such materials' application as most probably they can also be used as the composite reinforcement of polymer-matrix surface layers.

The surface engineering methods are also frequently used for filling recesses in such elements of machines and devices that are operated for a long time or in so-called remanufacturing processes for the formation of the structure and surface properties of re-processed structural components [200]. It is also noted that the surface properties of products and elements thereof during operation are subjected to considerable degradation, due to changes to the structure of engineering materials' surface, in particular as a result of corrosion of metals and alloys, tribological wear, wear and destruction of cutting tools and wear of hot-work tools. The mechanisms of wear should be taken into consideration already at the stage of products material and technological design by approaching them as factors the same as surface treatment, because, in fact, they form surface structure and properties, having, by the rule, usually an adverse impact and intensify over the time of product usage [3, 7, 18].

The state of the art of the highly extensive detailed knowledge in the field of surface engineering does not, paradoxically, support the making of correct managerial decisions on the optimised selection of the relevant technology in each case of producing a specific product. The reason for this is the lack of synthetic, collective elaborations that would take into account the cognitive, technical as well as economic aspects of the technologies mentioned and their importance in the industrial practise, according to harmonised, standardised criteria.

3. Analysed thematic areas and evaluation research methods

In the framework of the custom evaluation research [20, 21] concerning materials surface engineering two wide research areas were considered, respectively: *Manufacturing* (**M**) and *Product* (**P**). The first research field (M) reflects a manufacturer's point of view and encompasses the production processes determined by the state of the art and a machine park's manufacturing capacity. The *Manufacturing* (**M**) area is divided into narrower following research scopes:

M1: Laser technologies in surface engineering;

M2: PVD technologies;

M3: CVD technologies;

M4: Thermochemical technologies;

M5: Polymers surface layers;

M6: Nanostructural surface layers;

M7: Other surface engineering technologies.

The second research field (P) is determined by the expected functional and usable properties resulting from the client's demands and concentrates on the product and the material it is made of. The *Product* (P) area is divided into narrower following research scopes:

P1: Surface engineering of biomaterials;

P2: Surface engineering of structural metallic materials;

P3: Surface engineering of structural non-metallic materials;

P4: Surface engineering of tool materials;

P5: Surface engineering of steels used in automotive industry;

P6: Surface engineering of glass, micro- and optoelectronic and photovoltaic elements;

P7: Surface engineering of polymers.

The research tasks, intermediate goals and milestones in Table 2 are presented. The realisation scheme of methods used during the evaluation research taking into consideration, respectively: source data, monitoring spheres, current state, used foresight methods and final results in Fig. 2 is presented.

Table 2. *Research tasks, intermediate goals and milestones*

No.	Research task	Intermediate goal	Milestone
1.	Current situation analysis in the field of technology development and socioeconomic factors	Preparation of a report determining current situation in the field of technology development and socioeconomic factors in the scope of the foresight subject matter	Report *Current situation analysis*
2.	Research using a heuristic metho6d. e-Delphix method with experts participation	Achievement of research results using e-Delphix method and preparation of a report including results of three survey iterations	Report *e-Delphix method. Research results*
3.	Research using an artificial intelligence method, i.e. neural networks	Creation of artificial neural networks in order to determinate impacts between future trends and events	Neural networks

No.	Research task	Intermediate goal	Milestone
4.	Materials science- heuristic and foresight research	A series of materials science-heuristic and foresight research to verify development trends of the selected surface engineering technologies for selected engineering materials	Practical experimental verification of the computer integrated development prediction methodology developed
5.	Open public debate and social consultations	Open public debate and social consultations concerning research realisation and results	The International Conference
6	Generation and characterisation of priority leading technologies and strategic research directions in the scope of the foresight subject matter	Creation of technology roadmaps and technology information sheets for industrialists and three scenarios of possible future events: optimistic, neutral and pessimistic ones	The Critical Technologies Book Optimistic, neutral and pessimistic future events scenarios

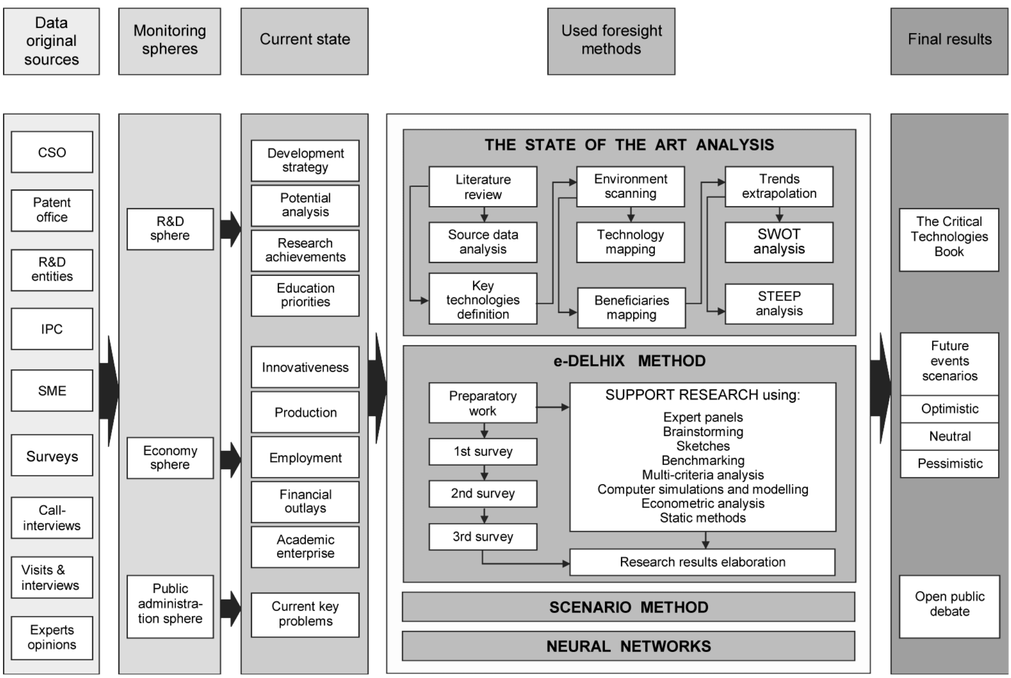

Figure 2. *The scheme of surface technologies evaluation methods [102]*

The following data from original sources during the evaluation research were used: Central Statistical Office (CSO), patent office, research and development (R&D) entities, Information

Processing Centre (IPC), small and medium enterprises (SME). The original data were gathered using survey questionnaires, call-interviews, visits and interviews in enterprises and institutions as well as expert opinions. The activity leaded within the framework of the research was concerned to three spheres: R&D, economy and public administration ones. In the R&D sphere development strategies, potential analysis, research achievements and education priorities are the most important. In the economy sphere especially innovativeness, production, employment, financial outlays and academic enterprise were considered. In the public administration sphere it is the most important to identify current key problems. Generally, the used evaluation research methods are typical for that kind of works and they are recommended as right and justifiable in foresight projects by National Foresight Programme "Poland 2020" [201], United Nations Industrial Development Organization and Polish Agency for Enterprise Development [202]. An exception is an innovative and experimental idea concerning the application one from artificial methods, i.e. neural networks into the analysis of impacts between future trends and events. All evaluation research were carried out parallel with reference to each one from the defined research scope pool.

4. The state of the art analysis for materials surface technologies

The research intermediate goal is to prepare a report characterising current situation in the field of technology development and socioeconomic factors in the materials surface engineering area. The state of the art analysis of surface technologies against a background of macro- and microenvironment realised by the key experts concerns each from the fourteen-element set of determined research scopes. Within the confines of the state of the art analysis the following works were carried out [102]:

- issue state assessment;
- technological review;
- strategic analysis using integrated methods.

Issue state assessment is a first part of the state of the art analysis. It is a set of elaborations connected with the consecutive research scopes. The following methods were recommended to key experts for the preparation of issue state assessment: literature review, source data analysis, environmental scanning, benchmarking (comparing to a leader), brainstorming, expert panels and trends extrapolation. Each elaboration concerning a given research scope includes the following elements: research scope general characteristics, statistical data, main trends and development directions as well as references. General characteristics concerning a given research scope includes main definitions, an outline of the knowledge development history and detail research fields included in the wide defined main research scope. Statistical data concerning issue state assessment in tables, graphs, charts and comparative tabulations were presented. Moreover, statistical data mainly shown within the confines issue state assessment are following [101]:

- domestic and/or European and/or world demand;
- domestic and/or European and/or world consumption;
- domestic and/or European and/or world production;
- the biggest domestic and/or European and/or world producers and sellers (amount produced/ sold by them);
- domestic and/or European and/or world export/import;
- domestic production capacity;
- production amount in given world countries;
- shares of given countries in European/world production;
- production amount changes in given years in a defined period.

The key experts in order to complete statistical data used many different source data. First of all the following raw data were utilised: Central Statistical Office (CSO), patent office, research and development (R&D) entities, Information Processing Centre (IPC), small and medium enterprises (SME). The raw data were gathered using survey questionnaires, call-interviews, expert opinions, visits and interviews in enterprises and institutions. Moreover, the key experts often in order to prepare the bibliographical as well as Internet review used the statistical comparisons. The next part of issue state assessment concerns main trends and development directions in the given research scope. The time horizon is strategic, it means a long-term one, i.e. the next 20 years. Especially, theories which are controversial, untypical as well as non thoroughly confirmed and investigated in that part of the elaboration were considered. The issue state assessment ends with references. The consecutive literature sources are arranged in an alphabetical order. The newest and English ones play the most important role between them.

The technological review is a second part of the state of the art analysis. It is a set of fourteen pieces from which every one is connected with a given research scope. The following methods were recommended to key experts for the preparation of technological review: critical technologies definition, technology mapping, literature review, environmental scanning, trends extrapolation, brainstorming, benchmarking and expert panels. Each part of a technological review concerning a given research scope includes the following elements: formulation of a technologies list included ones used in a given research scope; determination of the technology lifecycle phase in which are consecutive technologies from the previously defined technologies pool and identification of a preliminary critical technologies pool.

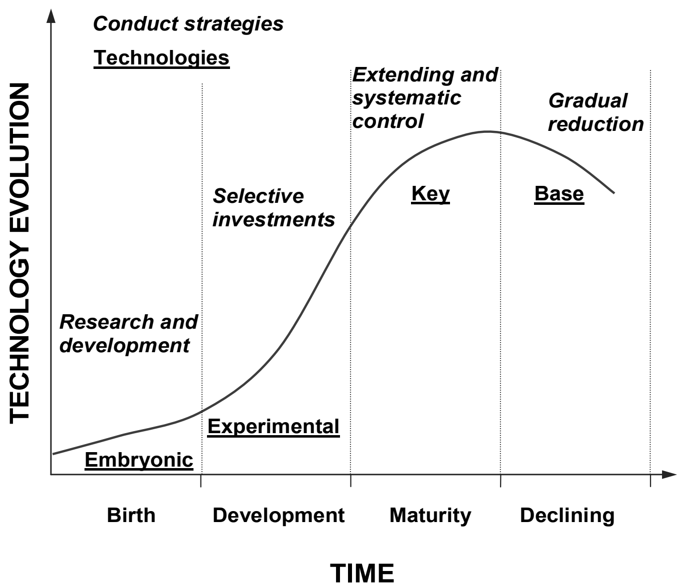

Figure 3. *Technology lifecycle [101]*

According to an issue called technology lifecycle all technologies were assigned to one from the following groups (Fig. 3) [101]:

- basic technologies; they are common available and used very often, their competitiveness decreases or is little yet; they are slowly falling into disuse;
- key technologies; they are the basis of the product competitiveness, their mastering is the key factor determining the enterprise success; the using perspective is ten years;

- experimental technologies; their application is not wide yet; their often are in testing or prototype building phase; a glorious future is predicted for them, because they should be key technologies in the future; they are very strong protect against competitors;
- embryonic technologies; they are in research development phase; works concerning outworking and implementation of prototypes are carried out, but prototypes do not exist yet; they are very strong protect against competitors the same like experimental technologies.

Into the next research step were qualified the key technologies which are not common used in the country as well as experimental and embryonic technologies. The assessment scale was five-steps, as follows: (1) really little, (2) little, (3) medium, (4) quite high and (5) high. There are two main criteria of technologies assessment which are defined in order to carried out research realisation, respectively: the (A) attractiveness and the (P) potential [101]. Moreover, the following six detailed criteria were outworked and used in carried out research:

- the (A1) economic attractiveness; significance of a given technology for the country and/or World economic development;
- the (A2) social attractiveness; significance and beneficial impact of a given technology for society;
- the (A3) ecological attractiveness; significance and beneficial impact of a given technology for natural environment state;
- the (P1) creative potential; the possibility of creation of new research and application directions;
- the (P2) applied potential; the possibility of a given technology application in implementation sphere;
- the (P3) research and development potential; the possibility of carried out of research and development works.

The $\overline{O_i}$ average mark concerning a given technology was determined using the (1) formula.

$$\overline{O_i} = \frac{\sum_{n=1}^{3} A_n + \sum_{m=1}^{3} P_m}{6} \qquad (1)$$

where:

$\overline{O_i}$ – the average mark concerning the i-th technology; i=1,2,…,a; a – a number of analysed technologies;

A_n – an assessment of a given technology attractiveness according to defined criterion; n=1,2,3;

P_m – an assessment of a given technology potential according to defined criterion; m=1,2,3.

The set of possible critical technologies consists of the technologies with the highest average marks which fulfilled the (2) condition.

If $\overline{O_i} > 3$, then the *i-th* technology belongs to a possible critical technology pool. (2)

where:

$\overline{O_i}$ – the average mark concerning the i-th technology; i=1,2,…,a; a – a number of analysed technologies.

A technology ranking according to attractiveness and a potential parameters in Fig. 4 is presented. The next stage of that research part concerned to verification the preliminary critical technologies pool and generation the final pool of 10 critical technologies in each thematic area.

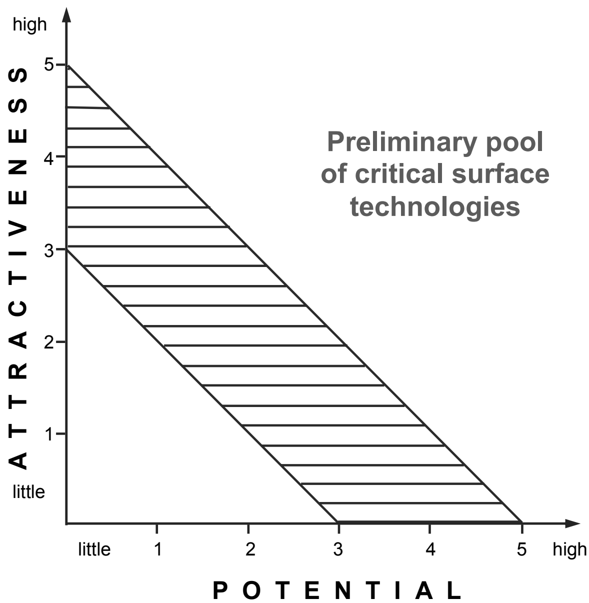

Figure 4. *A technology ranking according to attractiveness and potential parameters [101]*

Strategic analysis using integrated methods is a last part of the state of the art analysis. All from fourteen pieces connected with a given research scope fit together into a whole. Each part of strategic analysis concerning a given research scope includes the following elements:

STEEP analysis, SWOT analysis, conduct strategies choosing. The supporting role during preparation of strategic analysis by key experts played the following methods: multi-criteria analysis, trends extrapolation, beneficiaries mapping, environment scanning, brainstorming, expert panels.

According to the **STEEP analysis** assumptions consecutive outside remarks reflecting macroenvironment situation were considered [203, 204]. The macroenvironment characteristic feature is that it strong determines entities functioning conditions, but those entities can not directly influence on macroenvironment. Because of that, macroenvironment must be penetratingly observed. Particularly, arose opportunities should be used as well as shown weaknesses should be overcame. The outside positive and negative remarks according to the STEEP analysis are divided into the following remark groups (Table 3): **(S)**: Social; **(T)**: Technological; **(E)**: Economic; **(E)**: Ecological; **(P)**: Political.

The STEEP analysis results were directly utilised into the research carried out using the **SWOT analysis** rules. SWOT analysis is a key analytic tool using in order to categorise significant inside and outside remarks which can influence in a positive or negative way on forecasted future events [202, 205]. SWOT analysis enables to determine strengths and weaknesses of future events as well as opportunities and threats coming from environment, which can help or interfere with some appearing events. The results of carried out SWOT analysis were used in followed e-Delphix method. The first stage in SWOT analysis concerns the remark list formulation. The given remark list is assigned to the given research scope. There is a clear division of presented remarks into four groups, as follows: **(S)**: Strengths, inside positive remarks; **(W)**: Weaknesses, inside negative remarks; **(O)**: Opportunities, outside positive remarks; **(T)**: Threats, outside negative remarks. All analysed remark groups divided according to two criteria in Fig. 5 are presented. Within the confines of SWOT analysis realised for research needs the most important ten strengths and ten weaknesses of the given research scope were chosen. Similarly, also the most important ten opportunities and ten threats of the given research scope were completed. Next, each remark impact was defined. The sum of the impacts determined for each from four remark groups is equal 1. The consecutive remarks using the ten-steps universal relative scale was assessed. The best mark is (10) meaning very high level and the worst mark is (1) meaning very low level. The next stage requires weighted average calculation as a product of impact and assessment. The research results concerning each technology from the given research scope in four tables were presented. Each table correspond to each remark group.

Table 3. *Characteristics of different macroenvironment types*

Symbol	Macroenviron-ment type	Characteristics
S	Social environment	Social environment includes two remark groups: demographical and cultural ones. The demographical remarks are connected with the society size and people age structure, what in an important way determine profitability and development of some industry trades. However, the cultural remarks mainly include fashions, lifestyle and people tastes. They influence on some products and services popularity in certain social groups.
T	Technological environment	Technological environment signals increasing speed of technological changes, no limited innovations possibilities as well as high research and developments budgets. It pays attention also to little facilitates against huge inventions and the increasing number of legal articles concerning technological changes. Fast technological changes can contribute to decline some industry trades as well as to create another ones. Those events depended on entity activities profile can be an opportunity or a threat for the given entity.
E	Economic environment	Economic environment includes one of the most important and influenced group of remarks. It is determined by the domestic economy situation. The most important economic environment remarks are following: • the economic growth rate; high economic growth rate ensures the consumer expenses increase and from that arises higher development changes and lower competition on the market; • the interest rates; low level of the interest rates cause cheaper credits and ensure high demand level for the given products; • the exchange rates; they create competitiveness on the word markets; as an example: if given country exchange value is low comparing with other countries, then product import from that country is profitable and similarly if given country exchange value is high comparing with other countries, then product export to that country is profitable; • the inflation level; high inflation destabilises economy, limits growth rate, discourages investors from capital location and prevents from effective planning; the opposite of inflation is deflation; deflation causes reduction in goods and services prices and also in production and employment because of money flow limitation; too little money on the market generates stagnation what is unprofitable for domestic economy; the most profitable for domestic economy is little inflation reaching a few percent level; and also the consumption rate, unemployment level and public debt.
E	Ecological environment	Ecological environment is connected with natural factors and environment protection. The key issues concerning that environment are, as follows: raw materials deficiency, increasing energy costs and increasing environmental pollution level. The important factor concerns the increasing level of people ecological consciousness.
P	Political environment	Political-legal environment by the Government, binding legal acts and international situation is determined. Domestic government stability, transparent tax system and advantageous customs regulations encourage investors to investment of capital in the given country.

Next, overall statement including total weighted average of each remark group were shown. For each technology one of four possible conduct strategies were preliminary chosen. The strategy depends on inside and outside remark groups dominated with reference to the analysed research scope. Recommended conduct strategies in Fig. 6. is illustrated.

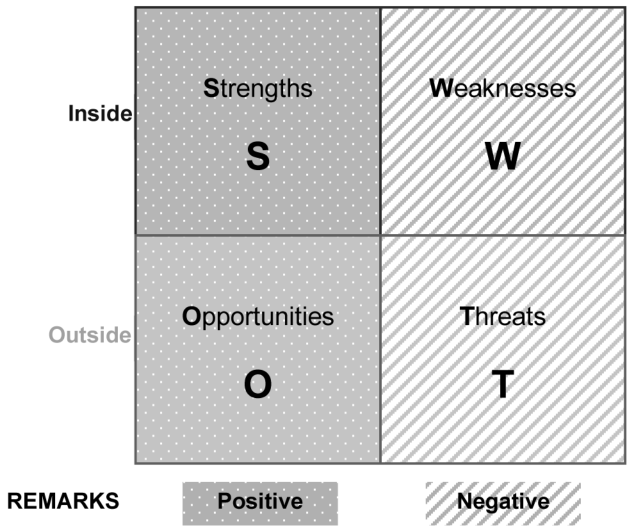

Figure 5. *Remark groups by SWOT analysis [101]*

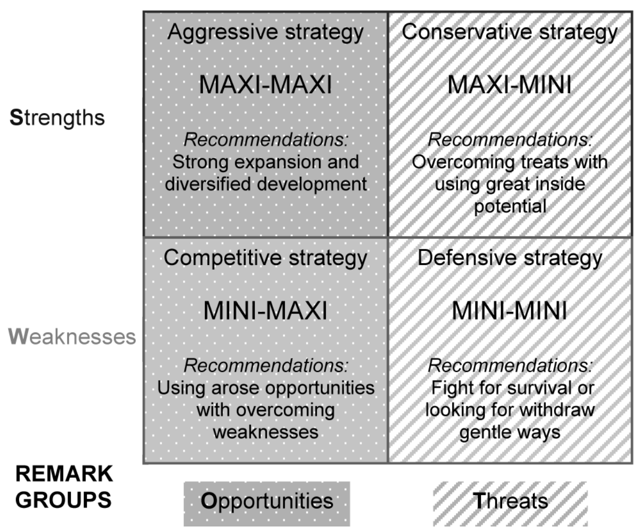

Figure 6. *Recommended conduct strategies [101]*

5. The computer integrated development prediction methods

The consecutive research intermediate goal is the achievement of research results using e-Delphix method and a preparation of a report including results of three survey iterations [20]. The results of carried out the state of the art analysis including, respectively: issue state assessment; technological review and strategic analysis using integrated methods is the pool of critical surface technologies. A collection of 140 critical technologies, 10 for each thematic group, was selected for the above 14 thematic groups from the initially inventoried approx. 500 specific technology groups.

The critical technologies generated as a result of the state of the art analysis next underwent expert studies performed with three iterations of the e-Delphi method according to the e-foresight concept. The e-Delphix method is a modified version of the classical Delphi method [206, 207], differing from the original method mainly in that experts are surveyed electronically and in that the level of generality for the questions asked to the experts is growing along with the subsequent iterations of the research. The relationships between the state of the art analysis and the e-Delphix method are given in Fig. 7. Over 300 independent experts from many countries representing scientific, business and public administration circles have taken part in the technology foresight [20]. The experts have completed approx. 650 multi-question surveys and held thematic discussions during 10 expert panels. Moreover, the scientific and research methods supporting main expert research realised by the e-Delphix method were parallel used, including: brainstorming, sketches, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and static analysis.

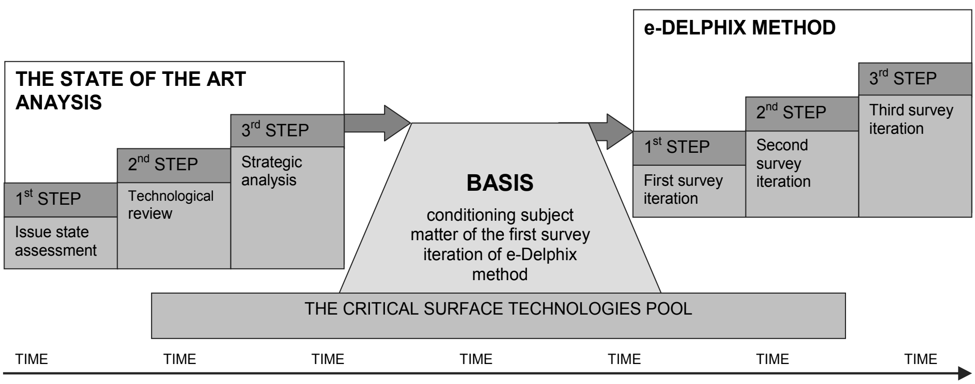

Figure 7. *The state of the art analysis versus e-Delphix method [101]*

The results of the expert studies laid the groundwork for further research targeted at identifying the position of the relevant critical technologies using contextual matrices, for preparing alternative scenarios of future events for materials surface engineering and for creating the Book of Critical Technologies of materials surface engineering consisting of technology roadmaps and technology information sheets. A very important element constituting a basis of the newly developed research methodology concerning the prediction of development of the relevant critical technologies is to perform a series of materials science and heuristic and foresight research to verify the development trends of the selected surface engineering technologies for the selected engineering materials described in a series of own works [9-17, 94-108] and in the next chapters of this book. The verification of the established methodology's correctness according to materials science criteria, made for the selected specific technologies of materials surface engineering, is necessary to ascertain that the reasoning used is correct and that the approach proposed is universal. The outcomes of the materials science research coupled with the results of heuristic research of strategic management of knowledge, show a full picture of the issue, allowing to characterise the analysed groups of technologies in terms of harmonising materials science, technological and economic criteria setting a basis of a comparative analysis of such technologies. The same place and time is not indispensable with reference to the implementation of materials science and heuristic research and does not affect the correctness of the reasoning conducted. The synergic influence of the materials science and heuristic research methods guarantees, therefore, that the evaluations made according to the methodology of computer-aided prediction of development of materials surface engineering is correct and adequate. The research was made using a customs e-foresight method detailed in the following papers [21, 94-97] and in one of the chapters of this book that follow.

The research main goal realisation should lead to achieve the following **final results**:

- The Critical Technologies Book preparation;
- Three alternative future events macroscenarios formulation and description,
- Open public debate on the foresight subject matter animation.

The Critical Technologies Book including technology roadmaps [208-210] and technology information sheets on the basis of the results of the materials science-heuristic research concerning critical technologies were prepared (Fig. 8).

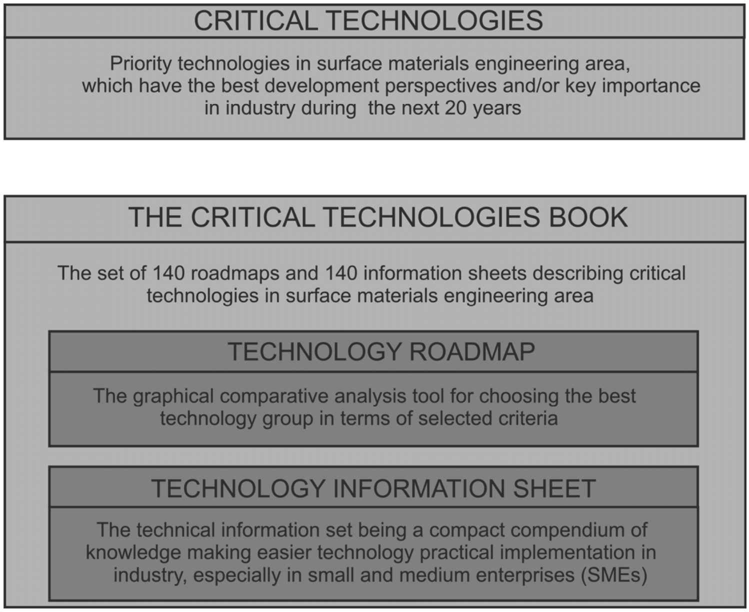

Figure 8. *The Critical Technologies Book content [13]*

The framework of the custom **technology roadmap** corresponds to the first quarter of the Cartesian system of coordinates. Three time intervals for the years: 2010-11, 2020 and 2030 are provided on the axis of abscissa, and the time horizon for all the results of the research applied onto the roadmap is 20 years. Seven main layers were applied onto the axis of coordinates of the technology roadmap: time layer, concept layer, product layer, technology layer, spatial layer, staff layer and quantitative layer, made up of more detailed sub-layers. The main technology roadmap layers divided into more detailed sub-layers were hierarchised starting with the top, most general layers determining all-social and economic reasons and causes of the actions taken, through the middle layers characterising a product and its manufacturing technology, to the bottom layers detailing organisational and technical matters concerning the place, contractor and costs. The middle layers of the technology roadmap are subject to two types of influence – pull from the top layers and push from the bottom layers. The relationships between the individual layers and sub-layers of the technology roadmap are presented with the different types of arrows representing, respectively, cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow [12, 13].

Technology information sheets, containing technical information very helpful in implementing a specific technology in the industrial practice, especially in SMEs are detailing and supplementing the technology roadmaps. The technology information sheets provide, in particular, a description of the technological process progress and a characteristic of a physiochemical phenomenon accompanying the technological processes, the advantages and disadvantages of the relevant technology, the most prospective detailed technologies and substitute / alternative technologies. They also contains the types of a coating / surface layer that may be deposited or the processes occurring at the substrate surface, as well as the specific properties of coatings / surface layers / substrate surfaces as a result of technological processes. A special heed was paid also to the general physiochemical conditions of technological process implementation, substrate material preparation methods, research instrument type / kind and possible specific accessories. Besides, the research results acquired with an expert research method have allowed to provide the following details in the developed sheets determined with a universal scale of relative states: the impact of technology application on the predicted and expected material properties, the efficiency of preventing the consequences of wear, industry section acc. to the PKD classification having the highest technology applicability, the applicability of computer modelling and steering methods and the development prospects of the individual analysed technologies. In addition, each technology information sheet provides a general or example diagram of the considered production process and a three-part list of the recommended references [12].

Three alternative future events macroscenarios, respectively: optimistic, neutral and pessimistic ones were formulated and described. The references [211-215] indicate that there is no one correct and generally accepted method of creating the scenarios of future events or a management algorithm recommended for implementation in the scenario creation process. In fact, the algorithm is created each time from the scratch by the practicians implementing a specific project [216]. The same refers to building the scenarios presenting the forecast future of materials surface engineering where a methodological challenge exists of combining skilfully the presentation and description of the phenomena characterised by varied generality and to capture the cause and effects relationships existing between them. In order to solve the so formulated research task, all the analysed phenomena are divided into the three groups: macro-, meso- and micro- ones. Three scenarios of future events are considered at the macrolevel. The mesolevel is grouping 16 key factors influencing the development of materials surface engineering and 14 thematic areas analysed under the foresight research. The microlevel is represented by 140 groups of critical technologies. Specific technologies with an unknown *n* number can be distinguished between for them, often differing only in details determining their applicability in the industrial practise. In a novel and experimental manner to impacts analysis between future events and development trends **neural networks** were used [16, 96]. The analysis serves to identify how the key mesofactors of surface engineering development (e.g. collaboration between science and industry, number of specialised laboratories and R&D institutions, continuous improvement and high quality of technology, transparent and friendly legislation, international co-operation and EU funds) and the relevant thematic areas analysed (M1-M7, P1-P7) may influence the occurrence of each of the macroscenarios. Nine models were created altogether using artificial neural networks by adopting, as dependent (input) variables, the probabilities of the occurrence of a growth trend, stabilised trend and/or declining trend determined for the key mesofactors conditioning the development of materials surface engineering and for the individual thematic areas for the research domain of M (Manufacturing) and P (Product). The experts were evaluating the occurrence probability of the relevant scenarios by dividing the total value of probability (of 100%) by the three possible variants of future events. The (output) dependent variables represent a probability that each of the three macroscenarios considered, i.e. optimistic, neutral and pessimistic, occurs [16].

Open public debate on the foresight subject matter is also the expected project result. Domestic and foreign representatives of science, economy, public administration and society are the main groups invited to join the debate. Open public debate initiated and animated in groups interested in a foresight subject matter should contribute to better cooperation between R&D and economy spheres and facilitate labour flow between them. The utility consequence of that is better competitiveness of Polish economy and science at the background of other European and world countries. The realisation of the research concerning the technologies of surface structure and properties formation of products and their elements should contribute significantly to predicted development. The best technological solutions taking into considerations the enterprises competitiveness increasing and improvement of product utility properties, stability and reliability should be founded. The expected practical project results are long-term ones. Identified and probably applied leading technologies and connected with them strategic research directions should be important minimum during the next ten or fifteen years. That is an average period of the amortisation of technological equipment in manufacturing sectors using those technologies. The introduction level of technological news in the field of selection of a material element and processes determining its structure and properties as well as selection of surface layer kind and technology ensuring expected utility properties currently is not satisfying. Special consideration should be given to small- and medium-sized enterprises encompassing

99.8% of all domestic companies and generating 68% of GDP in which the level of the priority innovative technologies introduction is insufficient. Because of that, an absolute need for increase average level of technologies realisation by producers' statistic majority takes place. It is very important for quality and stability of products on the market and it decides about Polish economy competitiveness. Thus, presented issues have great economic significance. The trade of surface properties formation of engineering materials and biomaterials is one from the most dynamic [3]. It requires the continual introduction of technological news into industrial entities working in almost all divisions of industries. Generally, SMEs have not considerable financial outlays allotted for development. It forces necessity of their activity directions determination. The research results will create good conditions for making objective decisions concerning innovativeness development and research financing. The additional effect will be knowledge propagation in scientific and industrial group of people interested in the foresight subject matter and activation of open public debate on the considered theme.

6. Conclusion

In order to diagnose the crucial scientific, technological, economic and ecological aspects of material surface engineering and to identify the directions of their strategic development and decision-taking, the appropriate instruments of forecasting and identifying the priority innovative technologies must be used. It is purposeful, therefore, to undertake systematic scientific research, notably using foresight methods, for predicting and formulating the directions of desired development of leading surface layers structure and properties formation technologies for products and their elements produced of different engineering materials. The directions of strategic research, classified in the most avant-garde thematic areas, should also be set to create several alternative feasible development scenarios aimed at the improvement of functional properties, durability and reliability of products and at selecting the most effective technologies that must be popularised in the industry which – in terms of their modernity and price to quality ratio – are most suitable for effective implementation in the industry. Foresight research in the field of material engineering allow, in particular, to identify the priority innovative technologies of materials surface engineering and to determine their strategic development directions. The dissemination of such research and the related public debate and the broadening awareness of entrepreneurs within the scope considered is tangibly translating into statistic growth in the quality of the technologies implemented industrially, into sustainable development and into the strengthening of a knowledge- and innovation-based economy. The development directions of the most advantageous technological solutions of surface layers structure and properties formation of products and their elements produced using materials surface engineering technologies considered as critical were indicated as part of own materials science-heuristic and foresight research [20] pursued together with top-notch, internationally recognised experts. The selected critical technologies of materials surface engineering understood as the priority technologies with the best development prospects and/or of key significance in the industry over the assumed time horizon were subjected to own research [6, 18, 20] in order to evaluate their value according to objectivised criteria against the micro- and macroenvironment and to identify their development prospects over the nearest 20 years. In this chapter the state of the art as well as evaluation and development prediction methodological assumptions for materials surface technologies is presented. The next chapters of this book present the detailed results of materials science and foresight investigations in relation to the selected groups of specific technologies [9-17] forming part of the practical verification of correctness of the established computer integrated development prediction methodology in surface engineering area, presented in general in the monograph publication [21].

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3 E-foresight of materials surface engineering

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Abstract

Purpose: *The purpose of this chapter is to present the concept of e-foresight and the possibilities of its implementation in practice using the Computer Aided Foresight Integrated Research Management (CA FIRM), Virtual Organisation for Foresight Integrated Research Management (VO FIRM) and Web Platform for Foresight Integrated Research Management (WP FIRM).*

Design/methodology/approach: *The proposed methodology of the Computer Aided Foresight Integrated Research Management (CA FIRM) describes the steps to be taken within the framework of technological e-foresight in order to carry it out it in an organised, efficient, and modern manner.*

Findings: *Methodology of conducting foresight research allowing it to be carried out in an organised, efficient and modern manner.*

Research limitations/implications: *Methodology implementation allows generating a set of priority innovative technologies and determining the strategic research trends whose development will be of key importance for the country within next 20 years.*

Practical implications: *The implementation of e-foresight results into economic practice will contribute to the development of knowledge-based economy, statistical increase in the quality of technology, and continuation of sustainable development.*

Originality/value: *This chapter for the first time presents the e e-foresight idea together with associated methodology of the Computer Aided Foresight Integrated Research Management (CA FIRM), Virtual Organisation for Foresight Integrated Research Management (VO FIRM) and*

Web Platform for Foresight Integrated Research Management (WP FIRM). The "show-off effect" concept has also been introduced.

Keywords: *Manufacturing and processing; Foresight; Surface engineering*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, E-foresight of materials surface engineering, Archives of Materials Science Engineering 44/1 (2010) 43-50.

1. Introduction

The strategic development-related priorities of the European countries include the development of knowledge-based economy, statistical increase in the quality of technologies and sustainable development. The innovative technologies of engineering materials and biomaterials surface engineering considered as extremely development-oriented are one of the most promising technologies which can be the key contribution to the achievement of assumed strategic developmental priorities. Thus, it becomes justifiable to conduct scientific research within this subject area. In the contemporary world it is impossible to make use of the available economic, systems, technological, financial, and social potential without using cutting-edge information systems for knowledge and information management. Such management, in respect of priority innovative technologies and strategic research trends concerning materials surface engineering, can be carried out by means of foresight research used for scientific prediction and shaping of the future by gaining knowledge from domain experts, organising it and disseminating. On account of materials surface engineering area significance and importance it is justifiable to conduct foresight research related to this particular knowledge area in an organised, efficient, and modern manner.

2. Contemporary world trends

One of the key trends which significantly determine the functioning of the contemporary world is a growing percentage of advanced engineering and technology in an increasing number of domains of everyday life. This is manifested by the fact that the access to state of the art automation developments, computerization, or satellite communication is more and more common for an average individual, which combined with already widespread command of English across the world, results in progressive globalisation and expanding cyberspace. Described factors definitely contribute to the development of information society, which not only has access to knowledge, information and information systems but also knows how to use them for the purpose of efficient and economically sound achievement of collective and individual goals. Modern society which can effortlessly gain access to any desired information is more and more aware, as a mass, of the significance of environmental issues and necessity to avoid further pollution as well as to rectify the effects of ensuing natural environment degradation. A care for natural environment is accompanied by the concept of sustainable development understood as a process of integrating systems, economic and social actions while maintaining natural balance and stability of basic natural processes, taking into consideration the interest and future of next generations.

The most important drivers which stimulate contemporary technological progress include the concept of continuous improvement, devised and implemented in the Japanese industry conditions and promoted around the world by Deming, which says that "it is never so good that it cannot get better" [1]. The consequence of a general social approval for such an approach is both higher and higher quality in various domains of social and economic life and increasing number of innovations implemented in economy as well as statistical increase in the quality of technologies implemented in the industry.

In recent decades, major changes have been observed in the relations between separate entities which make up the supply chain. The time when materials manufacturers dictated the rules is now a remote past since, at present, materials are manufactured on customer's demand and have properties demanded by the customer. In our age, it is considered a priority to increase product functionality at the expense of unjustified attachment to the type or chemical constitution of the applied material. In order to satisfy more and more sophisticated customer's needs, modern products are required to be manufactured with materials of apparently exclusive properties. This is possible by applying layers and coating with properties which are complementary to the properties of particular material.

Contemporary world is also full of various hybrids which are the consequence of combining, linking, and compiling different approaches, methods, and techniques related to problem solving. Such approach allows acquiring a new, better quality in numerous domains of life and allows the synergy effect to take place and reinforce the achieved final outcome. As part of this trend, the tendencies towards the management and manufacturing integration have been noticed, as well as a growing popularity of hybrid technologies.

Today's world trends include also the battle against the consequences of the economic crisis and attempt to nip in the bud the behaviours which led the developed countries, thriving until quite recently, such the USA, Island, Greece, or Ireland, to serious social and economic problems. It becomes therefore necessary to use the available economic, systems, technological, financial, and social potential in the best way possible in order to take advantage of arising opportunities while avoiding at the same time the risks by applying scientific prediction and shaping of the future [2, 3, 4].

The trends which occur in the contemporary world, presented in this part of the chapter and considered most important by the Author, may be a key factor to the shaping of the world's face and further directions of development.

3. Importance of materials engineering and materials surface engineering

Materials engineering is one of several most development-oriented scientific and technological areas in the contemporary world, constituting at the same time one of the most significant elements of science, science and technology, and innovative policy of Poland within the framework of knowledge-based economy. The group of advanced engineering materials with the best prospects of development within the next 20 years certainly includes the following groups of materials: nanomaterials, biomaterials, infomaterials, light metals alloys, and graded materials [5, 6]. The asset of nanomaterials is their extremely fine-grained structure guaranteeing mechanical as well as physical and chemical properties which cannot be obtained with any other methods. The future of biomaterials is connected with the development of biomimetic materials imitating the behaviour and functioning of nature and of the materials allowing replacing natural tissues and/or human organs either directly or with the use of properly designed appliances. Informaterials, which include the most advanced intelligent and self-organising materials, also belong to the group of the most prospective materials. The materials which play a crucial role, next to the composite materials, in the designing and use of modern means of transport are light metals alloys. Functionally and tool graded materials, in which the chemical constitution, phase composition, and structure or arrangement of atoms changes gradually along with their position (in a continuous or discrete manner) also have exceptional and specific properties [7, 8].

The development of materials engineering is not only the national but first of all the European Union's developmental priority, which have been included in the European Community's 7th Framework Programme 2007 to 2013 (FP7) for research, technological development and implementations. The detailed programme *CAPACITIES* in FP7 concerns guaranteeing the European Union's competitiveness and maintenance of productive potential, essential enhancement of industrial research, and implementation of new solutions to improve the current productive potential. The detailed programme *IDEAS* in FP7, on the other hand, intends to support the most creative, interdisciplinary scientific frontier research. The main line of development of materials engineering and production methods has been covered by the subject *"Nanosciences, nanotechnologies, materials and new production technologies"* of the detailed programme *COOPERATION* in FP7. The results of research conducted as part of the Europe's technological Foresight in the 5th and 6th European Community's Framework Programme and announced in the reports on implementation of the projects *The Future of Manufacturing in Europe (FutMan)* and *Manufacturing Visions The Futures Project (ManVis)* have been used in order to define the detailed assumptions of FP7 in the scope of materials and production methods. The most important of the selected future trends include: development of new engineering materials for expected applications, simplification of engineering materials production processes, and alternative opportunities for new production processes development in respect of new engineering materials. Among the compatible or alternative methods of accomplishing the anticipated trends one can distinguish: specialisation, convergence, and integration.

The generalisation of the European Foresight research results concerning different new materials and different material processes technologies is to anticipate the production of materials with properties demanded by the customer [9]. The necessity of producing engineering materials and biomaterials on demand, arising more and more often, in order to meet the complex set of specific requirements defined by the customer, dramatically changes the method of materials designing in general and the product material designing. At present, materials with properly formed structure guaranteeing the required set of physical and chemical properties have to be provided on demand of the product manufacturers. This approach replaces traditional choice-making based on the materials with the offered structure and properties, choice of material which is closest to the expectations, however, which does not meet them whatsoever, thus being the choice of a lesser evil. The current tendencies force therefore the classification of engineering materials in respect of their functional characteristics. In this view, the type, and especially the chemical constitution of the material used, is of a minor importance, while product functionality gains greater importance. At present, the materials engineers participate in the product designing process, and the product manufacturers have to meet the imposed requirements as the effect of multi-criteria optimization of structure, properties, mass, product manufacturing and use costs, as well as ecological compatibility with natural environment, etc. It is therefore fundamental to make a change in the assessment of engineering materials role as they can no longer be perceived as the goods in themselves with applications sought for them, and the new engineering materials market can no longer be the manufacturer's market. Offering materials which are currently in stock regardless the users' needs is out of question now. The manufacturers' market has ended irretrievably. The new engineering materials and production processes are subjected to the customer's needs and product functional features. The production of materials which satisfy the needs of market product manufacturers in due time and place is the priority of new materials technologies and production processes as they are complementary base technologies used to improve the existing solutions, alternative technologies applying synergy of various solutions and original technologies aiming at developing completely new solutions.

When writing on the significance of the materials engineering in its broad context, which constitutes one of the most important elements of science, science and technology, and innovative policy of Poland, the importance of its component known as surface engineering should be emphasized. Very often, the functional properties of many products and their elements depend partially or mainly on the surface layer structure and properties and not only on their physical and chemical properties or the possibility of transferring the mechanical load through the entire cross section of the element. Therefore, as a result of proper selection of the element's material together with its structure and properties formation processes as well as surface layer type and technology, which guarantee required functional properties, it is possible to put together the manufactured element's core and surface layer properties in the most favourable manner. It is thus not surprising that materials surface engineering, including surface treatment and coating, is one of the most dynamically developing sectors of economy in many technologically advanced countries. As an example, according to reference data of 2008, 8-10% of German economy was accomplished in this very industrial sector. With a large measure of probability the analogous phenomenon will soon occur in a rapidly developing Polish economy. Surface treatment and coating in its wide context are carried out in almost each manufacturing sectors of the industry including the automotive, machine building, tool construction, mechatronic, metallurgical, electrotechnical, electronic, plastics, aircraft, medical equipment, sanitary devices, jewellery, precision, construction, and other industries. Engineering materials and biomaterials surface engineering is undoubtedly one of those domains which are promising and can potentially become a key contribution to the country's economic growth.

For the accomplishment of a long-term strategy of production which will respond in a flexible manner to continuous change in the customers' preferences, the Polish enterprises, following the example of the countries operating within the so-called old European Union, should put pressure on constant development of advanced manufacturing technologies and search for innovative solutions. The level of technological novelties implementation and increase in the quality of applied technologies within this scope is definitely unsatisfactory, especially when referring to small and medium-sized enterprises (SMEs), of which expenditures on development are inconsiderable. Thus a need arises to chart the course of action for the SMEs, which will positively contribute to their market success and consequently will be a key contribution to the statistical increase in the quality of the technologies implemented in the Polish industry. It should be highlighted that the examined issue does not concern solely the cutting edge technologies applied by model enterprises which are often referred to when discussing new technologies. It is much more important to focus on the critical need of increasing the average level of technology implementation by statistical majority of manufacturers, which is crucial for the quality and stability of a statistical majority of products launched into the market and it substantially determines competitiveness of the country's economy.

4. E-foresight

When carrying out the foresight research related to materials surface engineering in practice, the problems and difficulties have been encountered which urged the Author of this chapter to conduct scientific research aiming at organising, improving and updating the process of carrying out foresight research. Such a large scale of research planned to be conducted has become the major driving force behind the implementation of improvements. Generally, 14 subject areas have been analysed within the framework of foresight research on materials surface engineering.

At the initial research stage, about 500 technologies have been analysed, about 150 of which have been qualified for detailed analysis. As part of the research, three survey iterations have been planned, addressed to top-class experts selected from the scientific, business, and public administration environments. It has been planned that 210 filled in surveys will be obtained in each of three foresight research iterations – 630 in total [10]. The necessity has therefore arisen to develop methodology and information technology which would organise, improve, and update conducted foresight research. This is how the e-foresight idea emerged in relation to already known and commonly used concepts [11, 12]: e-management, e-business, e-commerce, e-banking, e-logistics, e-services, e-administration, e-education, which always refer to the performance of particular activities using the computer networks, especially the Internet.

4.1. E-foresight idea

E-foresight means conducting foresight research using the Internet. E-foresight is orientated to support the activities of two beneficiaries. The first group consists of foresight researchers, who can perform their work at any time and in any location, which combined with teleworking contributes to giving equal opportunities on the labour market as it allows the persons working from home, including mothers raising small children and disabled persons, to join the team of project researchers. The other group of beneficiaries are the domain experts, direct participants of the conducted survey, selected from the scientific, business, and public administration environments, who can work according to the principle: *"I participate with my laptop in the foresight research at the time and place which are most convenient to me"*, thus contributing to a quicker and more effective acquiring of indirect and final research results.

4.2. Computer Aided Foresight Integrated Research Management CA FIRM

The Computer Aided Foresight Integrated Research Management (**CA FIRM**) methodology describes the steps to be taken within the framework of the e-foresight technological research in order to carry it out in an organised, efficient, and modern manner. Such *organising* is achieved by describing in detail the order of what needs to be done and how, so that the foresight research is conducted successfully in practice, which is of great significance if we take into consideration an inconsiderable number of national publications on this subject. Carrying out the foresight research in a more *efficient* manner is achieved by speeding it up, elimination of the “show-off effect”, and occurrence of the synergy effect resulting from the integration of the foresight research. The foresight research conducted in a *modern* way is accomplished through the use of information technology, including the Web Platform, virtual organisation, databases, and neural networks, which fits into the most dominant world trends according to which the racing process of computerization in respect of newer and newer domains of life is observed.

The key question which will we will be able to answer as a result of implementing the CA FIRM methodology is following:

Which of the technologies applied in a particular research field belong to the set of priority innovative technologies and the development of which strategic research trends in a particular research field will be of key importance for the country within next 20 years?

In order to answer the above research question, it is necessary to execute the following **steps** of the CA FIRM arranged on a serial and parallel basis:

- Division of a wide research field covered by the foresight subject into detailed subject areas;
- Performing of three iterations of expert opinion research by electronic way;
- Preparation of the static statements presenting expert opinion research results;
- The dendrological matrix of technology value construction;
- The meteorological matrix of technology value construction;
- The matrix of strategies for technologies construction;
- The neural networks creation and training using data received from experts;
- Preparation neural networks aided three alternative version of future events scenarios, respectively: optimistic, neutral and pessimistic ones;
- Creation of the Critical Technologies Book including the pool of Technology Roadmaps and Technology Information Sheets;
- Open public debate initiation and animation;
- Useful assessment of the modern information technology including Virtual Organisation, Web Platform and Neural Networks into the foresight process realisation.

The CA FIRM methodology is dedicated to the computer aided foresight research on materials surface engineering. The implementation of the proposed approach allows the technological foresight on materials surface engineering future to be carried out in an organised, efficient and modern manner, and, in particular, allows determining priority innovative technologies and strategic development trends within this research field. It should be noted, however, that this methodology, due to its universal nature, can be without any difficulties implemented to carry out any technological foresight concerning the future of any domain of knowledge. The computer tool allowing the CA FIRM to be conducted from the technical aspect is the author Web Platform for Foresight Integrated Research Management (WP FIRM). In order to achieve the e-foresight objectives it is also necessary to create a Virtual Organisation for Foresight Integrated Research Management (VO FIRM) which allows gathering, organising, selecting, and managing explicit and tacit knowledge in cyberspace.

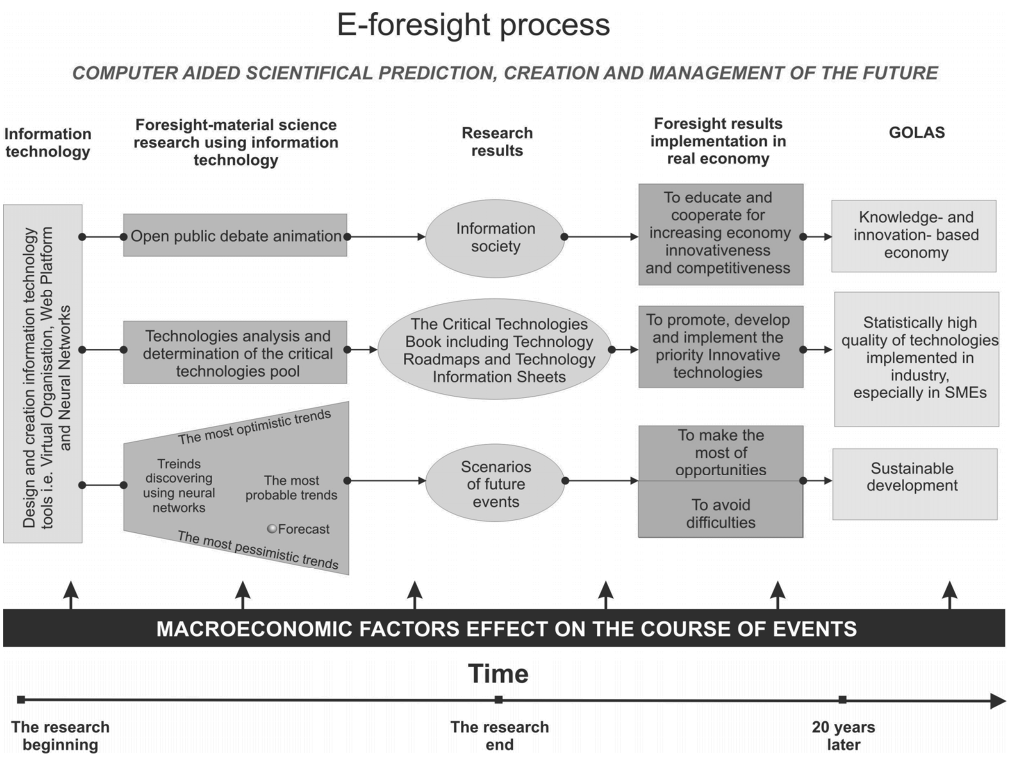

Figure 1. *E-foresight process*

The e-foresight process including the technical basis indispensable for the commencement of appropriate research, the types of the VO FIRM virtual organisation activity with the use of WP FIRM Web Platform, research results, implementation of foresight results in the economic reality, and strategic objectives that those activities are accompanied by have been presented in Figure 1. Attention should be paid to the distribution of the mentioned elements across the time scale and to the constant impact of macroeconomic factors on the course of events.

Due to the implementation of the CA FIRM methodology it becomes possible to achieve the main objectives of technological foresight in an organised, efficient, and modern manner as well as to specify which of the technologies applied in a particular research field belong to the set of priority innovative technologies and the development of which strategic research trends in a particular research field will be of key significance for the country within next 20 years. The developed methodology is an integrated approach which allows gaining explicit and tacit knowledge from the top-class experts selected from the scientific, business, and public administration environments during the performance of three survey iterations. The proposed approach uses the synergy effect and eliminates unfavourable psycho-social phenomenon called by the Author of this chapter as the "**show-off effect**" which is manifested during direct meeting of people which serves to exchange views on a specific subject and consists in the fact that people are orientated to show themselves to their best advantage and promote themselves instead of sharing their knowledge.

4.3. Virtual Organisation for Foresight Integrated Research Management VO FIRM

The concept of virtual organisation introduced in 1992 by W. Dawidow and M. Malone [13] have been modified, improved, and developed over years, which, however, has not resulted in the formulation of one commonly acceptable concept base.

The deliberations present in the literature on the subject area have resulted in the establishment of two trends reflecting different approaches to virtual organisation [14]: process-based and structural. The process-based approach presents the organisation from the functional point of view, as an operation mechanism, area of activity, or approach towards organisation management which focuses on actions and behaviours [15]. On the other hand, the structural approach, presented much more often, concerns the components of an organisation, their characteristics, and dependencies between them.

The Virtual Organisation for Foresight Integrated Research Management (VO FIRM), created for the achievement of e-foresight objectives, is part of the structural trend and signifies a system of elements, formed on the basis of voluntary principle, functioning dynamically and flexibly structuralised. This system of elements is of a task nature, orientated towards particular objectives, coordinated by means of information technology, allowing tacit and explicit knowledge to be gathered, organised, selected, disseminated, and managed in cyberspace.

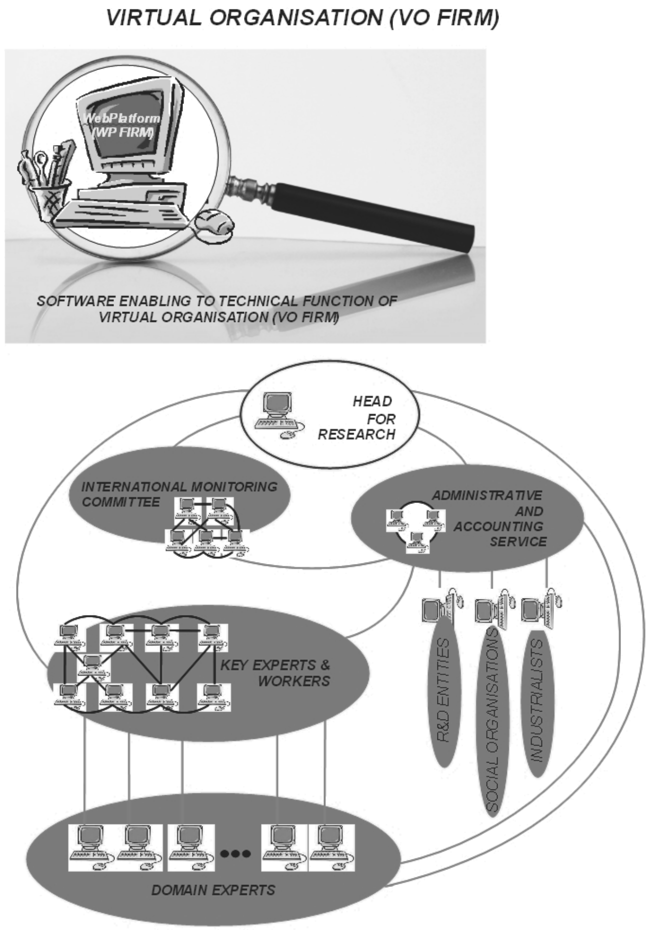

Figure 2. *Organisational chart for VO FIRM*

The VO FIRM is set up to accomplish a long-term all-society objective which is a closer relation between science and economy and development of information society, thus increasing the importance of knowledge-based economy (KBE), seeking sustainable development and statistical increase in the quality of technology. The VO FIRM is indispensable to carry out the technological foresight aiming at identifying priority innovative technologies and strategic research trends in respect of the analysed research field. A general organisational chart for the Virtual Organisation for Foresight Integrated Research Management – VO FIRM, has been shown in Figure 2.

The Virtual Organisation Management – VO FIRM as per devised concept of its functioning in cyberspace is carried out using the following modules of the Web Platform – WP FIRM:

- Experts' surveying;
- Database on experts;
- Newsletter;
- Information management;
- E-mail management;
- Finance and documentation management,
- Contract export/import from/to the university contracts system.

4.4. Web Platform for Foresight Integrated Research Management WP FIRM

The Web Platform for Foresight Integrated Research Management – WP FIRM developed from scratch on the basis of the author concept is a computer tool allowing the e-foresight objectives to be met and the virtual organisation VO FIRM to be managed in cyberspace. The Web Platform WP FIRM serves to provide the conditions for the performance of tasks by the research team of the foresight project researchers. In addition, the WP FIRM Web Platform aims at providing work comfort for the domain experts selected from the scientific, business and public administration environments, thus allowing them to work according to the principle: *"I participate with my laptop in the foresight research at the time and place which are most convenient to me"*. Thanks to the WP FIRM Web Platform it is also possible to easily disseminate information concerning the progress of conducted work and stimulate public debate on the foresight subject.

The list of activities which can be performed in cyberspace owing to technical possibilities provided by the WP FIRM Web Platform includes the following elements:

- Conducting large-scale questionnaire surveys;
- Free use/collation of information included in the database on experts;
- Receiving subscription news concerning the project realisation in the form of a *newsletter* by individuals from the world of industry, science and public administration;
- Managing information collected in the computer database;
- Quick exchange of Internet correspondence with experts whose names are in the computer database;
- Managing project finances, contracts, and documentation;
- Immediate data transfer from/to a compatible university contracts system;
- Obtaining information used for developing the database on experts and managing the data concerning particular domain experts in a quick and effective manner;
- Tracking the events concerning the Project on an ongoing basis by every outside observer, both by viewing the website as well as active participation in an online public debate.

***Figure 3.** An exemplary page of the WP FIRM*

At present the WP FIRM Web Platform can be viewed as a “living organism” as it is subjected to constant development, updates and modifications. The platform reacts dynamically to the user’ needs arising and changing over the course of foresight research realisation. The changes are implemented on an ongoing basis and aim to maximize performed tasks and functions, streamline the user interface, enhance data storage safety, increase resistance to disturbances, adapt to the existing and commonly applied IT solutions. The main page of the WP FIRM Web Platform, tested and verified for the purposes of materials surface engineering foresight, has been presented in Figure 3.

5. Conclusions

The innovative technologies of materials surface engineering as extremely development-orientated belong to the most promising technologies, which can become a key contribution to the accomplishment of strategic developmental priorities of the European countries including the development of knowledge-based economy, statistical increase in the quality of technology and sustainable development thus substantiating the research conducted in this field. When carrying out the foresight research concerning materials surface engineering in practice, the problems and difficulties have been encountered mainly due to a large scale of planned activities, which triggered scientific research aiming at organising, improving, and updating the conducted foresight research process. As a response to a justifiable necessity of devising methodology and information technology for this purpose, the e-foresight idea has emerged which signifies carrying out the foresight research using the Internet. E-foresight is orientated to support the activities of two beneficiaries: the foresight project researchers and the domain experts participating directly in the conducted questionnaire surveys according to principle: *“I participate with my laptop in the foresight research at the time and place which are most convenient to me”,* thus contributing to a quicker and more effective acquiring of indirect and final research results. In order to achieve the objectives of technological e-foresight, which come down to the identification of priority, innovative research technologies and strategic trends in respect of the analysed research field, the Computer Aided Foresight Integrated Research Management – CA FIRM methodology has been developed. The implementation of the devised methodology into practice is possible thanks to the creation of the Virtual Organisation for Foresight Integrated Research Management (VO FIRM), based on the voluntary principle, which constitutes a system of elements orientated towards particular objectives, coordinated by means of information technology, allowing tacit and explicit knowledge to be gathered, organised, selected, disseminated, and managed in cyberspace. The computer tool which enables the achievement of such defined objectives and aims from the technical aspect is the Web Platform for Foresight Integrated Research Management – WP FIRM, developed from scratch on the basis of the author concept.

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4 Foresight methods for technology validation, roadmapping and development in the surface engineering area

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Abstract

Purpose: *The aim of this chapter is to present a set of methods for evaluating the strategic development perspectives of priority innovative technologies, backed by an example illustrating the practical application of a proposed solution with regard to the selected surface engineering methods.*

Design/methodology/approach: *The chapter describes a set of validation, roadmapping and technology development methods in the part pertaining to the dendrological matrix of technology value, the meteorological matrix of environment influence, the matrix of strategies for technologies, the setting of strategic development tracks and creating technology roadmaps.*

Findings: *The presented approach allows for organizing, enhancing and modernizing the process of testing as part of the technology foresight, with special consideration paid to material surface engineering.*

Research limitations/implications: *The proposed general analyses purposefully omit the materials science part of the methodology which will be thoroughly presented in the series of publications currently prepared for print, on selected surface engineering technologies. The carried out works are part of a bigger research project aimed at generating a set of most promising surface engineering technologies.*

Practical implications *The described methods will be used to set priority innovative technologies in the field of material surface engineering; however, their universal character indicates they may be successfully used during the realisation of any technology foresight and, after a slight adaptation - also an regional foresight.*

Originality/value: *The value of the chapter is an example-supported presentation of an original authority set of methods for validation, roadmapping and forecasting the development of technologies, with special consideration paid to the field of material surface engineering.*
Keywords: *Methodology of research; Analysis and modeling; Foresight; Technology value and development; Technology Roadmapping*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, Foresight methods for technology validation, roadmapping and development in the surface engineering area, Archives of Materials Science Engineering 44/2 (2010) 69-86.

1. Introduction

Sustainable development which provides societies or their present or future citizens equal access to the environment, as well as economic growth stimulated by economy based on knowledge require a statistical increase of the quality of technologies implemented in industry [1-3]. The analysed problem pertains not only to avant-garde technologies realised by model companies but, to a larger extent, to the absolute need to increase the average level of implementing technologies by the statistical majority of manufacturers. This has very large significance on the quality and durability of the statistical majority of products introduced to the market and significantly decides about the competitiveness of economy. Thus, it is justifiable to aim at a scientific forecasting and shaping of the future [4-13], thus deviating as far as possible from the ineffective and risky method of trials and errors which may quickly lead to wasting the accomplishments of previous generations. In this situation, it seems critical to direct scientific research to the most promising scientific fields and branches which may have a large influence on the quick civilisation-economic development, the development of the IT community and the creation of an economy based on knowledge, what is the goal of many actually realised and recently finished projects [3, 14-22]. Moreover, attention should be drawn to providing the possibility of a rational practical use of conducted studies and of creating budgetary references for them. Such defined aims and targets are realised through the implementation of foresight study results in real economy, pertaining to detailed, specialist and promising thematic fields. A generalisation of Europe's technology foresight results announced in reports from the realisation of projects The Future of Manufacturing in Europe (FutMan) [14] and Manufacturing Visions The Futures Project (ManVis) [15] is the anticipation of manufacturing engineering materials of qualities ordered by product users. New engineering materials, as well as the processes of their manufacturing and processing are subordinated to customer needs and the utility functions of products. Very often, utility functions of many products and their elements depend mainly on the structure and qualities of surface layers [1-2, 23-25]. As a result of a suitable selection of the element's material and the processes which shape its structure and qualities, as well as due to the type and technology of the surface layer which provide the required utility properties, it is possible to set the properties of the core and surface layer of the manufactured element in the most favourable way. Material surface engineering, involving surface treatment and surface covering is one of the most dynamically developing economy branches in many technologically advanced countries [15] and many detailed fields, what is the subject matter of scientific works, such as [26-42]. It is anticipated that material surface engineering, as a highly significant part of broadly understood material engineering and deserving a separate and thorough analysis, will in the nearest decades be placed among the most promising advanced material technologies [14-16, 20]. Thus, it is appropriate to conduct scientific studies pertaining to strategic development directions of material surface engineering, while the analysed problem has high social-economic significance [1-5]. The strategic development directions of material surface engineering may be set in an ordered, facilitated and modernised way based on the results of classic studies of the structure and properties of surfaced engineering materials and on computer-aided foresight studies intended for anticipating and shaping the future through acquiring knowledge from experts, organizing and spreading it according to the rules of the worked out strategy. This constitutes the subject of wide-spread individual foresight-materials science research [43-48] whose main aim is the identification and critical discussion on priority innovative technologies and strategic directions of developmental studies which qualify for using industrial methods of shaping the structure and properties of engineering material surfaces whose development will be critical during the next 20 years. Generally, an analysis was anticipated of a dozen or so thematic areas, constituting in total approx. 500 detailed technologies, out of which approx. 150 technologies were qualified for further analyses as part of three iterations of surveys addressed to high-quality experts selected from scientific, business and public administration circles, in total leading to obtaining over 600 expert opinions. The analysis of this problem leads to generating a set of priority innovative surface engineering technologies which contribute to the statistical quality increase of technologies applied in industrial companies, stimulating sustainable development and strengthening economy based on knowledge. The selected aims of the undertaken individual studies include also working out a methodology of the computer-aided integrated management of foresight studies. The assumptions of the newly-created author's e-foresight methodology pertaining to the Computer Aided Foresight Integrated Research Management (CA FIRM), together with creating an accompanying IT technology including especially: a Virtual Organisation for Foresight Integrated Research Management (VO FIRM), an Web Platform for Foresight Integrated Research Management (WP FIRM), with the use of Neuron Networks for Foresight Integrated Research Management (NN FIRM), were presented in an independent publication [43]. The worked out e-foresight methodology which uses IT technologies corresponds to already known and commonly used notions [49-51]: e-management, e-business, e-trade, e-banking, e-logistics, e-services, e-administration and e-education. Each of these terms means conducting specified activities with the use of computer networks, especially the internet. The e-foresight methodology allows for organizing, facilitating and modernizing the conducted foresight research [43]. The aim of this chapter is to present an authority methodology of specifying the values of the analysed set of selected technologies for shaping the structure and properties of the surface layer against the environment, together with the recommended strategies of acting, strategic development tracks taking into account the influence of each of the used technologies of processing engineering material surfaces on the quality, structure and properties of surface layers obtained as a result of these technologies, and creating a set of technology roadmaps of the analysed technology groups. The creation and use of these tools allows for presenting, in a uniform and clear format, various types of factors which directly and indirectly characterize given groups of technologies together with the forecast and perspectives of their development in different intervals of the adopted time horizon [52-54]. Technology roadmaps are a very comfortable and practical tool of comparative analysis which facilitates the selection of technologies according to the selected criterion, and when supplemented by operation sheets with precise technological details - they enable the implementation of a given technology in industrial practice. A very large significance of technology roadmaps is their flexibility which enables their supplementing and expanding by new sub-layers depending on the arising needs.

The methodological bases described in this chapter are used in a series of foresight-materials science research which constitute a fragment of broader individual actions [4, 5, 44] aimed at selecting, testing, characterizing and specifying strategic development perspectives of priority innovative material surface engineering technologies in the process of technological e-foresight and subsequently prepared publications, including especially the results of individual research corresponding to specific surfacing technologies. However, the created methodology has a broader significance and may be applied in all types of technology and regional foresights, as well as in other areas of knowledge and information management in which the use of modern IT systems constitutes a prospective, modern and effective approach aimed at using the currently available economic, system, technological, financial and social potential for the realisation of strategic developmental aims.

2. Outworked research methodology

The conducted studies are interdisciplinary and the used testing methodology pertains mainly to technology foresight [6] being an element of a field called organisation and management and to surface engineering included in a more broadly understood material engineering. At certain stages of the conducted studies, also methods were used which come from artificial intelligence, statistics, IT technology, construction and exploitation of machines, as well as strategic [55], operational [56] and quality [57] management.

According to the adopted methodology, the carried out research include: selecting technology groups for experimental-comparative research, collecting expert opinions, carrying out a multi-criteria analysis and marking its results on the dendrological and meteorological matrix, determining strategies for technologies preceded by rescaling and objectivising test results using formulated mathematical relations, setting strategic development tracks for technologies, carrying out a series of specialist materials science experiments in experienced team [33-42] using a specialist diagnostic-measuring apparatus and the creation of technology roadmaps.

In accordance with the applied methodology of foresight-materials science research, several possibilities of homogenous groups should be singled out from the analysed technologies in order to subject them to planned experimental-comparative nature research.

The division criterion may be, e.g.: the type of tested substrate, the type or number of coatings/surface layers applied to the tested material, the manner or type of the researching apparatus for applying coatings/surface layers to the tested material.

The determination of objectivised values of specific singled out technologies or their groups is done through the dendrological matrix of technology value, while the meteorological matrix of environment influence is used to specify the positive and negative force of the micro- and macroenvironment on given technologies. The methodological construction of both these matrices refers to portfolio methods commonly known in management sciences [57-61], serving as characterisation of the portfolio of products offered to the customer by the company, allowing for a graphic presentation of the results of comparative analysis conducted based on two criteria/factors placed on the horizontal and vertical axes of the matrix, respectively. The most famous matrix of this type – the Boston Consulting Group (BCG) matrix [62] – owes its extraordinary popularity to simple associations and intuitive concluding, which has become an inspiration during the creation of methodological assumptions of the dendrological and meteorological matrices. For the purpose of evaluating technology groups with regard to their values and environmental influence, a ten-point universal scale of relative states (Table 1) was adopted, in which the smallest value 1 corresponds to a minimum level, and the highest value 10 is the level of perfection.

Table 1. *Universal scale of relative states*

NUMBER	Class discriminant	LEVEL	
			perfection
10	0.95	EXCELLENT	
9	0.85	VERY HIGH	
8	0.75	HIGH	normality
7	0.65	QUITE HIGH	
6	0.55	MODERATE	
5	0.45	MEDIUM	
4	0.35	QUITE LOW	mediocrity
3	0.25	LOW	
2	0.15	VERY LOW	
1	0.05	MINIMAL	

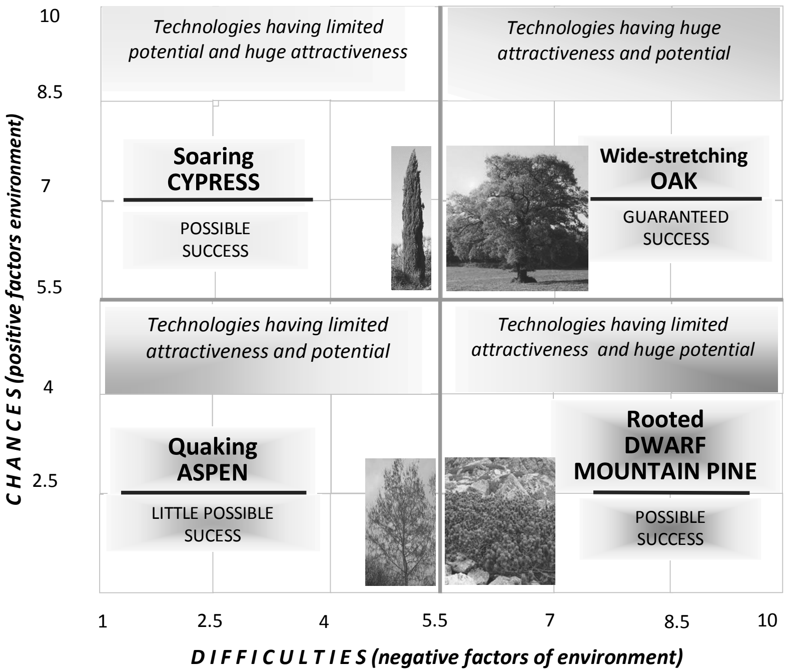

***Figure 1**. The dendrological matrix of technology value. The idea presentation*

The dendrological matrix of technology value (Fig. 1) presents graphic results of evaluating specific technology groups, with special attention paid to the potential constituting the real objective value of a given technology and to the attractiveness reflecting how a given technology is subjectively perceived among its potential users. The potential of a given technology group expressed through a ten-point universal scale of relative states, marked on the horizontal scale of the dendrological matrix is the result of a multi-criteria analysis carried out based on an expert opinions taking into account, in suitable proportions, the following types of potential: creational, application, qualitative, developmental and technical. On the vertical scale of the dendrological matrix the level of attractiveness was marked of a given technology group which is the mean weighed expert opinions based on detailed criteria corresponding to the business, economic, liberal, environmental and system attractiveness. Depending on the type of potential and level of attractiveness determined as part of the expert opinion, a given technology may be placed in one of the quarters of the matrix. The following quarters were distinguished in the dendrological matrix of technology value:

- **Quaking aspen** is a weak technology with limited potential, included within the range ⟨1, 5.5⟩ and with limited attractiveness within the range ⟨1, 5.5⟩, whose future success is unlikely;
- **Soaring cypress** corresponds to a technology with limited potential within the range ⟨1, 5.5⟩, but with huge attractiveness included in the range (5.5, 10⟩, which causes the success of a given technology to be possible;
- **Rooted dwarf mountain pine** is a technology with limited attractiveness within the range ⟨1, 5.5⟩, but with huge potential included in the range (5.5, 10⟩, thanks to which its future success is possible;
- **Wide-stretching oak** corresponds to the best possible situation in which the analysed technology is characterised by huge potential within the range (5.5, 10⟩, as well as huge attractiveness within the range (5.5, 10⟩, and this connection guarantees future success.

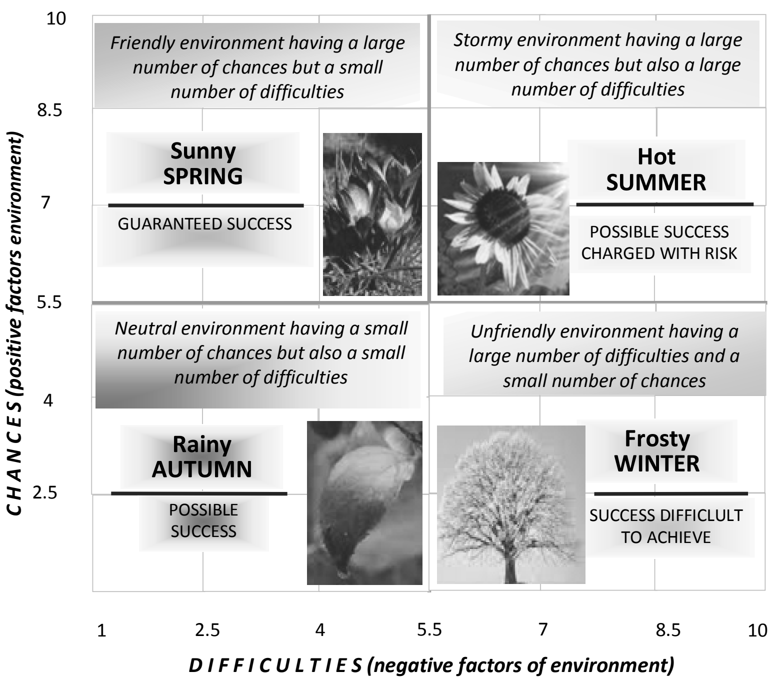

Figure 2. *The meteorological matrix of environment influence. The idea presentation*

The meteorological matrix of environment influence (Fig. 2) presents graphic results of evaluating the impact of external factors on specific groups of technologies which had been divided into difficulties with a negative impact and chances which positively influence the analysed technologies. The testing of expert opinions on the subject of positive and negative factors which influence specific technologies was carried out based on a survey comprising several dozens of questions pertaining to the micro- and macroenvironment in strictly defined proportions. 16% of the questions pertain to the competitive environment, while the remaining 84% are questions regarding specific constituents of the macroenvironment, and especially the following types of environment: technological (20%), economic (16%), social (12%), political-legal (12%), international (12%) and natural (12%). External difficulties expressed with the use of a ten-point universal scale of relative states, which are the result of a multi-criteria analysis conducted based on the expert opinions, have been placed on the horizontal scale of the meteorological matrix. On the other hand, chances, i.e. positive environment factors being a mean weighed expert opinions based on detailed criteria, were placed on the vertical scale. Depending on the level of influence of positive and negative environment factors on the analysed technology, determined as part of the expert opinions on a ten-point scale, it is placed in one of the matrix quarters. The following quarters were distinguished in the meteorological matrix of technology value:

- **Frosty winter** corresponds to the worst possible situation in which the environment carries a large number of difficulties included in the range (5.5, 10⟩ and a small number of chances within the range ⟨1, 5.5⟩, which causes the success in a given environment to be difficult or impossible to obtain;
- **Hot summer** corresponds to a situation in which the environment brings many chances included within the range (5.5, 10⟩, but which is also accompanied by many difficulties from the range (5.5, 10⟩; this causes the success of the technology under these conditions to be possible but charged with risk;
- **Rainy autumn** corresponds to a neutral situation in which there are no dangers awaiting for a given technology, which corresponds to the range ⟨1, 5.5⟩, but also the environment does not carry many chances which is reflected by the range ⟨1, 5.5⟩;
- **Sunny spring** is the best possible variant because it denotes a friendly environment with a large number of chances from the range ⟨5.5, 10⟩ and a small number of difficulties included within the range ⟨1, 5.5⟩, which will guarantee success of a given technology under such good conditions.

INFLUENCE OF ENVIRONMENT \ VALUE OF TECHOLOGY	1–2.25	2.25–5.5	5.5–7.25	7.25–10
7.25–10	STRATEGY OF AN ASPEN IN SPRING TO TAKE ADVANTAGE OF PRESENCE ON AN ATTRACTIVE MARKET	STRATEGY OF A CYPRESS IN SPRING TO TAKE ADVANTAGE OF CHANCES, STRENGTHENING TECHNOLOGY POTENTIAL	STRATEGY OF A DWARF MOUNTAIN PINE IN SPRING TO TAKE ADVANTAGE OF CHANCES, STRENGTHENING TECHNOLOGY ATTRACTIVENESS	STRATEGY OF AN OAK IN SPRING SUCCEED
5.5–7.25	STRATEGY OF AN ASPEN IN SUMMER TO ANALIZE RISK	STRATEGY OF A CYPRESS IN SUMMER TO STRENGTHEN POTENTIAL AND ESTIMATE RISK	STRATEGY OF A DWARF MOUNTAIN PINE IN SUMMER TO STRENGTHEN ATTRACTIVENESS AND ADJUST PRODUCT TO CLIENT'S REQUIREMENTS	STRATEGY OF AN OAK IN SUMMER TO ANALIZE THE MARKET AND TAKE ADVANTAGE OF CHANCES
2.25–5.5	STRATEGY OF AN ASPEN IN AUTUMN TO SLOWLY LEAVE THE MARKET	STRATEGY OF A CYPRESS IN AUTUMN "TO MILK THE COW" AS LONG AS POSSIBLE STRENGTHENING TECHNOLOGY POTENTIAL	STRATEGY OF A DWARF MOUNTAIN PINE IN AUTUMN "TO MILK THE COW" AS LONG AS POSSIBLE STRENGTHENING TECHNOLOGY ATTRACTIVENESS	STRATEGY OF AN OAK IN AUTUMN "TO MILK THE COW" WHILE SEARCHING FOR A NEW RANGE OF TECHNOLOGY APPLICATIONS
1–2.25	STRATEGY OF AN ASPEN IN WINTER TO LEAVE THE MARKET	STRATEGY OF A CYPRESS IN WINTER TO SLOWLY LEAVE THE MARKET	STRATEGY OF A DWARF MOUNTAIN PINE IN WINTER TO RESIST DIFFICULTIES	STRATEGY OF AN OAK IN WINTER TO INTENSIVELY SEARCH FOR NEW TECHNOLOGY APPLICATIONS

LEGEND

Well-portending strategies

Badly-portending strategies

Uncertain strategies which can cause success or only minimalize losses depending on circumstances

Figure 3. *The general matrix of strategies for technologies*

INFLUENCE OF ENVIRONMENT				
10	To utilize, as much as possible, weak technology using a good economic situation and, utilizing insight into the attractive market, test, develop and introduce new technologies. TO TAKE ADVANTAGE OF PRESENCE ON AN ATTRACTIVE MARKET	To test, perfect, strengthen and fully invest in an attractive technology making use of a good economic situation on the market TO TAKE ADVANTAGE OF CHANCES, STRENGTHENING TECHNOLOGY POTENTIAL	To enhance, modernize, automate, computerize and promote technology of high potential, using good economic conditions on the market TO TAKE ADVANTAGE OF CHANCES, STRENGTHENING TECHNOLOGY ATTRACTIVENESS	To develop, strengthen, implement attractive technology of a large potential in industrial practice in order to achieve outstanding success. SUCCEED
7.25	To conduct risk analysis and, depending on its results, remain on the market or withdraw from it with weak technology TO ANALIZE RISK	To enhance the potential of an attractive technology in risky environment conditions, evaluate risk and, depending on the result, fight aggressively for the customer or slowly withdraw the technology TO STRENGTHEN POTENTIAL AND ESTIMATE RISK	To try to enhance and modernize the technology of high potential, carry out marketing studies and adjust the product to the customer's requirements TO STRENGTHEN ATTRACTIVENESS AND ADJUST PRODUCT TO CLIENT'S REQUIREMENTS	To use the attractiveness and potential of a technology in a risky environment, avoid difficulties, carry out marketing studies and adjust the product to the customer's requirements. TO ANALIZE THE MARKET AND TAKE ADVANTAGE OF CHANCES
5.5	To slowly withdraw a weak technology from the market which does not provide new possibilities. TO SLOWLY LEAVE THE MARKET	To utilize the stable, predictable environment to a maximum degree in order to implement production using an attractive technology, simultaneously strengthening its potential "TO MILK THE COW" AS LONG AS POSSIBLE STRENGTHENING TECHNOLOGY POTENTIAL	To profit from the realisation of production in a stable, predictable environment using solid technology which should be modernised and intensively promoted in order to strengthen its attractiveness "TO MILK THE COW" AS LONG AS POSSIBLE STRENGTHENING TECHNOLOGY ATTRACTIVENESS	Succeed with an attractive, stable technology on a predictable market while searching for new markets, customer groups and products possible to acquire using this technology. "TO MILK THE COW" WHILE SEARCHING FOR A NEW RANGE OF TECHNOLOGY APPLICATIONS
2.25	To withdraw a weak technology from the market in which difficulties predominate. TO LEAVE THE MARKET	Based on the technology's attractiveness, make use of the environment to a maximum degree and withdraw the technology of a small potential from the market in which difficulties predominate TO SLOWLY LEAVE THE MARKET	To resist piling up difficulties coming from the environment, at the same time trying to strengthen the attractiveness of a technology with a large potential TO RESIST DIFFICULTIES	To try and wait out the difficulties and remain on the market with a solid, attractive technology, while intensively searching for new markets, customers and products possible to manufacture using this technology. TO INTENSIVELY SEARCH FOR NEW TECHNOLOGY APPLICATIONS
1	2.25	5.5	7.25	10

LEGEND

(dark grey)	Well-portending strategies
(light grey)	Badly-portending strategies
(white)	Uncertain strategies which can cause benefits or only minimalize losses depending on circumstances

Figure 4. *The detailed matrix of strategies of technology*

At the next stage of research works the results of research presented in graphic form using the dendrological matrix of technology value and the meteorological matrix of environment influence have been placed on the **matrix of strategies for technologies**. This matrix comprises sixteen fields corresponding to specific variants resulting from a combination set of four technology types and four environment types. The general matrix of strategies for technologies (Fig. 3) graphically presents the place of technologies, considering their value and environment influence and points to the strategy of conduct which should be adopted with regard to a given technology, taking into account the previously analysed factors. The detailed matrix of strategies for technologies presented in Figure 4 includes in its fields a short description of conduct recommended if a given technology with a specified value belonging to a given numeric range is applied in an environment with a value from the specified numeric range and a negative (difficulties) or positive (chances) return. To facilitate the transfer of specific numeric values from the dendrological matrix [2x2] and the meteorological matrix [2x2] to the matrix of strategies for technologies with the dimensions of [4x4], mathematical relations were formulated which enable the rescaling and objectivising of test results and, based on them, a short computer program was created to enable a quick calculation of the searched values and their placing on the chart. Thus, the following notions were introduced: the relative value of technology V_n and the relative value of environment influence E_n. With the use of mathematical relations expressed in a simplified way by the system of equations (1), it is possible to perform detailed calculations and to visualize them using the matrix of strategies for technologies.

$$\begin{cases} V_n' = c + \left(\dfrac{d-c}{b-a} \right) \left(V_n - a \right) \\ E_n' = c + \left(\dfrac{d-c}{b-a} \right) \left(E_n - a \right) \end{cases} \qquad (1)$$

where:

a − the minimum value on a scale used in the dendrological and meteorological matrix;
b − the maximum value on a scale used in the dendrological and meteorological matrix;
c − the minimum value on a scale used in the matrix of strategies for technologies;
d − the maximum value on a scale used in the matrix of strategies for technologies;
V_n' − the relative value of technology on a scale used in the matrix of strategies for technologies;
V_n −the relative value of technology on a scale used in the dendrological and meteorological matrices;

$E_n^{'}$ − the relative value of the environment influence on a scale used in the matrix of strategies for technologies;

E_n − the relative value of environment influence on a scale used in the dendrological and meteorological matrices;

n − the alphanumeric symbol of a given technology/group of technologies, $n \in \{A, B, \ldots, Z.\}$.

The next stage of research involves specifying **strategic development tracks** for specific technologies / groups of technologies which constitute a forecast of their development in time intervals corresponding to the years 2015, 2020, 2025 and 2030 in three variants: optimistic, pessimistic and most probable, and then visualizing them against a matrix of strategies for technologies.

The results of the carried out experimental-comparative research constitute source data which serve for creating technology roadmaps. The heading of each **technology roadmap** contains a basic characteristic of the technology, including: the name of the technology, the represented research field and the given catalogue number. The layout of the technology roadmap created for the purpose of the realised research corresponds to the first quarter of the Cartesian coordinate system. Three time intervals were placed on the horizontal axis, pertaining to: the situation as of today (year 2010), in ten years' (in 2020) and in twenty years' time (in 2030). The time horizon of all the research placed on the technology roadmap equals 20 years and is adequate to the dynamics of changes occurring in the analysed thematic field − the surface engineering. On the vertical axis of the technology roadmap seven main layers were placed, characterised in short in Table 2, corresponding to a specific question pertaining to the analysed scope. Each of the main layers has been additionally divided into sub-layers with a higher level of detail. The main layers of the technology roadmap were organised in a hierarchical way. The upper part of the technology roadmap contains the most general layers specifying the premises, reasons and causes of realised research which influence the layers placed under them in the process of „pull". The middle part of the technology roadmap pertains to the essence of the analysed problem by characterizing the product and technology used for its manufacturing. The lowest layers of the technology roadmap contain various details of the technical-organisational nature which influence the higher-located layers in the process of „push". In addition, the technology roadmap presents relations between its specific layers and sub-layers, with a division into: cause-and-effect relations, capital relations, time correlations and two-way flows of data and/or resources, visualised using different types of arrows.

Table 2. *Characteristics of the main layers of the technology roadmap*

Item	Layer name	Range	Question	Description
1.	Time	Order	When?	Defines the adopted time intervals and time horizon of conducted research
2.	Conceptual	Purpose	Why?	Specifies social and economic perspectives of conducted actions and the suitable strategy for a given technology
3.	Product	Subject	What?	Characterizes the product created during a given technological process, taking into account its structure and properties
4.	Technological	Manner	How?	Characterizes a given technology in terms of the following detailed criteria: lifespan, type and form of production, machinery park, automation and robotisation, quality, ecology
5.	Spatial	Place	Where?	Specifies the type of organisation and represented industrial branches
6.	Staff	Contractor	Who?	Defines the structure of the staff and expected employee competences
7.	Quantitative	Cost	How much?	Specifies capital requirements and the estimated production volume

The technology roadmap is a universal tool which enables presenting, in a unified and clear format, different types of internal and external factors directly and indirectly characterizing a given technology, taking into account the ways of influence, interdependencies and the change of specific factors over time. Such created technology roadmaps are becoming a very comfortable and practical tool of comparative analysis, facilitating the selection of the best technology in terms of the specified selected criterion, and when supplemented by technological sheets with precise technological details – they enable the implementation of a given technology in industrial practice. It should also be noted that a very important feature of technology roadmaps is their flexibility. When needed, the technology roadmap may be supplemented and expanded by additional sub-layers, adapting it, e.g. to the specificity of the carried out scientific-research studies, the requirements of a given industrial field or the size of a company.

The inherent part of the worked out methodology are detailed materials science research of surface layers structures created with the use of various surfacing methods, research of mechanical and tribological properties, as well as research of utility functions under conditions of exploitation or similar ones. The results of these research relating to technologies selected through the described technology valuating methods constitute an important premise for working out and experimentally verifying the evaluations made using methods of working out matrices of strategies for these technologies and they are necessary for creating a technology roadmaps and operation sheets. A synergic impact of the materials science and foresight testing methods guarantees the accuracy and compatibility of evaluations made according to the methodology worked out and described in this work with regard to surface engineering. Each time, carrying out such research requires supporting the forecasts based on foresight research with detailed materials science research as integral elements of the created applied methodology, which gives basis for proper concluding as part of the technology foresight. It is obvious that there is no unity of the place and time of realizing such research because proper finishing of the technology foresight requires possessing detailed results of research pertaining to the analysed technology, which could have been carried out previously. If it is impossible, it is necessary to carry out such detailed research as part of the foresight.

3. Illustrative example

The procedure, whose aim is to evaluate strategic development perspectives of surface engineering technologies in accordance with the methodology described in sub-chapter 2 of this chapter, has been illustrated with an example that presents the recommended manner of conduct step by step using specific numeric values. Thus, the presented example includes: a dendrological matrix of technology value, a meteorological matrix of environment influence and a matrix of strategies for technologies together with points placed on them which correspond to specific technologies, three alternative strategic development tracks created for an exemplary technology and one from representative technology roadmaps created as part of the carried out works [3, 44]. The conducted general analyses purposefully omit a strictly materials science part of the methodology pertaining to specific research of surface layer structures created via different methods, research of mechanical and tribological properties and research of utility functions under exploitation or similar conditions. This aspect of the analysed theme will be properly presented in the series of publications currently prepared for print, pertaining to the application of the proposed methodology in a comparative analysis of specific technologies or their homogenous groups. These groups may be created based on a division in terms of the type of tested surface, type or number of coatings/ surface layers applied to the tested substrate, the manner or type of the apparatus for applying coatings/surface layers to the tested substrate or any other clearly defined, objectively verifiable criterion.

Adopting the method of material surfacing as the division criterion in the presented example, four detailed technologies were subjected to analysis:

(A) Laser remelting,

(B) Passivation,

(C) Detonation spraying,

(D) PVD multilayer coatings deposition.

Source data constituting a basis for the performed assessments are obtained from experts who, in a survey study, answer a series of detailed questions on the potential and attractiveness of a given technology, evaluating each of them using a ten-point universal scale of relative states. Depending on the advancement level of the carried out foresight works, at the earlier stages answers may be provided by a narrow group of key experts, while at the advanced stages – by a considerably bigger group of trade experts selected among scientific, business, and public administration circles, which is realizing this aim, eg, as part of the Delphi method [6, 8, 11]. The studies especially test the creational, application, qualitative, developmental and technical potential, as well as the business, economic, liberal, environmental and system attractiveness. Using a multi-criteria analysis, the mean weighed value is calculated from the analysed criteria (attractiveness and potential), and the result obtained for specific technologies is placed on the dendrological matrix of technology value (Fig. 5).

In the presented example, PVD multilayer coatings deposition D (8.11, 8.99) was placed in the most promising quarter – the wide-stretching oak – which includes technologies with large potential and attractiveness. Laser remelting A (2.23, 7.44) turned out to be a soaring cypress with large attractiveness and a relatively small potential. Detonation spraying C (9.30, 1.94) was placed in the quarter representing the rooted dwarf mountain pine with large potential and relatively small attractiveness, while the lowest result was obtained by passivation B (3.22, 4.39), placed in the quarter referred to as the quaking aspen with low attractiveness and small potential. The evaluation of the positive and negative impact of the environment on specific technologies has been carried out with the use of a meteorological matrix of environment influence. In accordance with the adopted methodology, the matrix includes results of a multi-criteria analysis which were subjected to expert assessments during surveying, presented in Figure 6. The form used for survey studies comprises several dozen questions relating to micro- and macroenvironment forces influencing technologies in strictly specified proportions. The research results presented in the example show that in accordance with the expert opinions, the friendliest environment called sunny spring, characterised by a large number of chances and small number of difficulties, currently includes the B (9.45, 8.02) technology – passivation. In accordance with the obtained results, risky hot summer with a large number of chances accompanied by numerous difficulties is the environment of technology D (9.14, 8.92) belonging to the group of PVD multilayer coatings deposition. Neutral rainy autumn with a small number of chances and difficulties is the environment of technology A (4.10, 3.78), i.e. laser remelting, while frosty winter with its numerous difficulties and lack of chances is the environment of technology C (8.22, 2.35), i.e. detonation spraying.

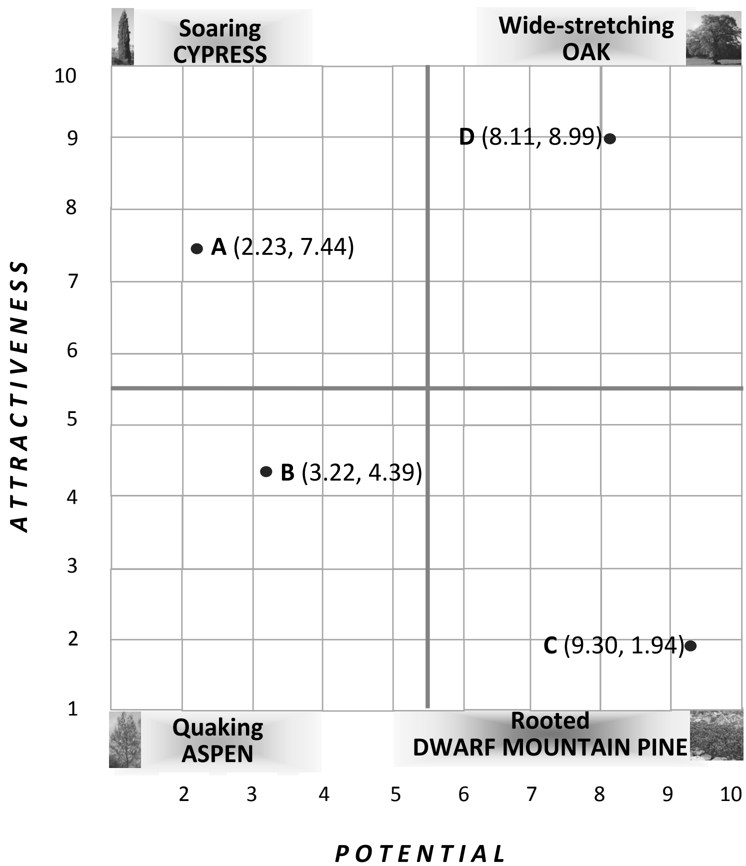

__Figure 5.__ The dendrological matrix of technology value prepared for example technologies representing, as following: (A) Laser remelting, (B) Passivation, (C) Detonation spraying, (D) PVD multilayer coatings deposition

At the next stage of research works, the results of the multi-criteria analysis which uses source data obtained from experts in the process of surveying, presented in graphic form using a dendrological matrix of technology value and meteorological matrix of environment influence, were placed on the matrix of strategies for technologies (Fig. 7). In order to transfer specific numeric values from the dendrological and meteorological matrices onto the matrix of strategies for technologies of different dimensions, formulated mathematical relations were used which allow for rescaling and objectivising research results (1). The matrix of strategies for technologies illustrates a graphic place of specific technologies, taking into account their values and impact forces of the environment, indicating a suitable acting strategy.

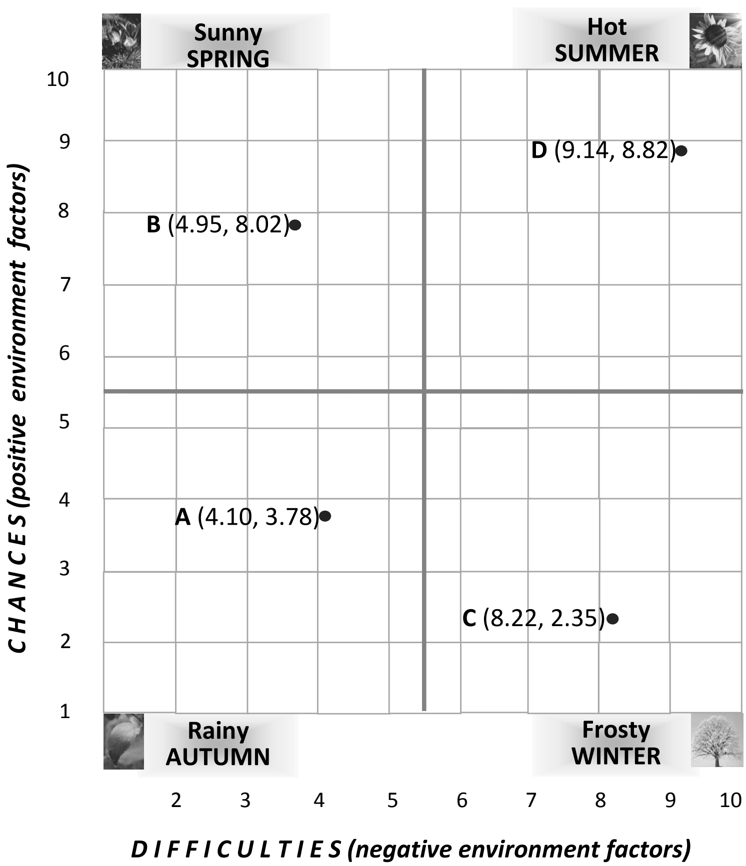

Figure 6. *The meteorological matrix of environment influence prepared for example technologies representing, as following: (A) Laser remelting, (B) Passivation, (C) Detonation spraying, (D) PVD multilayer coatings deposition*

The laser remelting technology – A (3.75, 3.87) – was placed in the matrix field corresponding to the strategy of a cypress in autumn, according to which the use of a stable, predictable environment is recommended for the realisation of production using an attractive technology with limited potential, and it should be accompanied by strengthening its potential. Passivation – B (2.01, 8.07) was placed in the matrix field corresponding to the strategy of an aspen in spring. For this strategy, it is recommended to exploit, as much as possible, a weak technology in times of good economic conditions and to test, develop and introduce new technologies relying on business contacts and insight into the attractive market. Technology C (6.07, 1.53), i.e. detonation spraying, was placed in the matrix field corresponding to the strategy of a dwarf mountain pine in winter which means that the actions recommended for it involve resisting the piling up difficulties coming from the environment, at the same time trying to strengthen the attractiveness of that technology with high potential. The last of the technologies – D (9.13, 6.33) – which represents PVD hard multilayer coatings deposition was placed in the matrix field corresponding to the strategy of an oak in summer, which involves using the attractiveness and potential of technology in a risky environment and avoiding difficulties; moreover, it is recommended to conduct insightful marketing studies and, based on them, to adjust a product manufactured using a given technology to the customer's requirements and expectations. It is necessary to emphasise that the evaluation in a scale of relative states are hypothetically presented for the use of this chapter and do not actually come from wide questionnaire surveys of experts, as well as environmental conditions for each of these discussed technologies are well established hypothetically. For example, the potential and attractiveness of passivation are low rated, what symbolizes an aspen. However, it is possible to imagine that for example in a plant equipped with technological equipment for the thermo-chemical treatment, taken over by the new owner with large capital resources, it is possible to use successfully such technology until new technological processes are worked out for new products, what is symbolised by the sunny spring in a matrix assessing environmental conditions. Mentioning these technologies is therefore an example to illustrate the mechanisms for the evaluation resulting from the newly developed methodology presented in this chapter. The presented examples, illustrating the manner of practical implementation of the proposed methodology with regard to specific technologies used in the industry, purposefully adopted various values of the analysed factors wanting to obtain the most diversified end results possible so that it will be possible to show a maximum broad spectrum of variants which may occur in the economic reality.

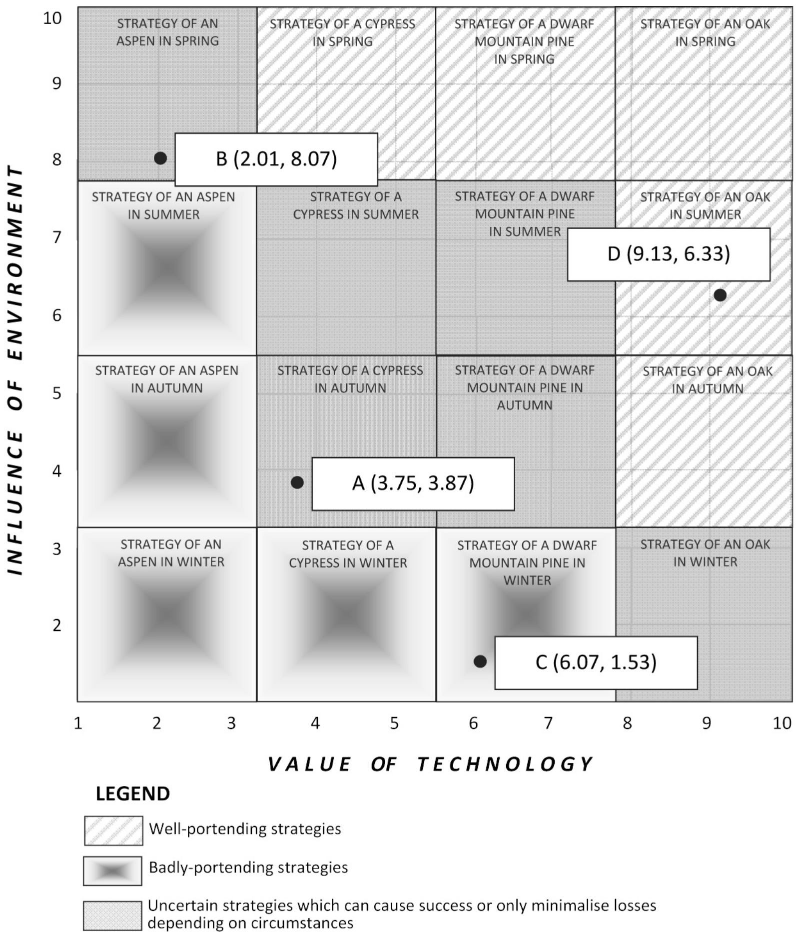

Figure 7. *The matrix of strategies for technologies prepared for example technologies representing, as following: (A) Laser remelting, (B) Passivation, (C) Detonation spraying, (D) PVD multilayer coatings deposition*

When the actual value of a given technology against the environment and the recommended strategy of conduct are known, the aim of the next step, in accordance with the proposed approach, is to specify how the technology itself and its environment will be changing in specific time horizon intervals of foresight studies. For this purpose, strategic development tracks are set in three variants: optimistic, pessimistic and most probable. For example, for the graphic delimitation of strategic development tracks being the forecast for their development in: 2015, 2020, 2025 and 2030 in three variants, another technology of gas nitriding marked as E (3.44, 3.62) was chosen.

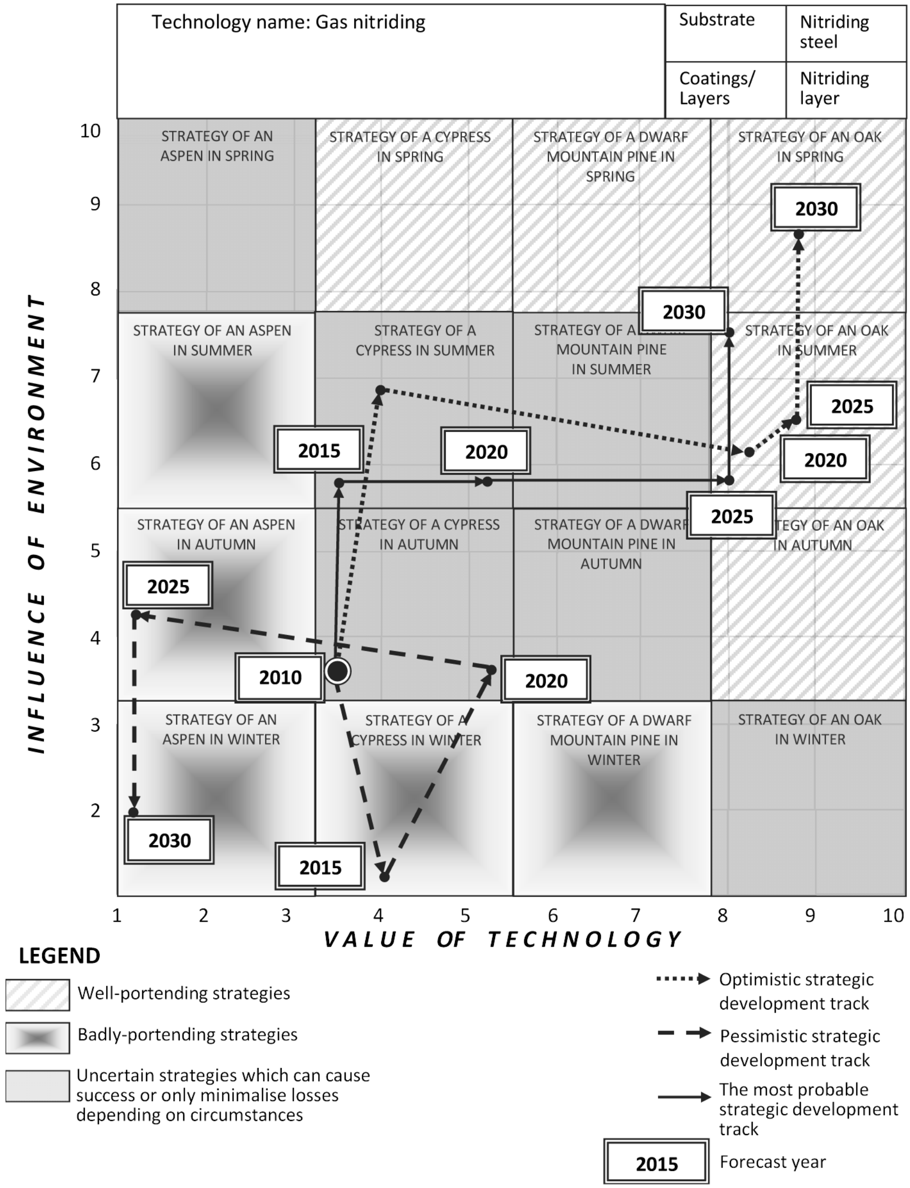

Figure 8. *Optimistic, pessimistic and the most probable strategic development tracks for example technology (E) - gas nitriding against the background of the matrix of strategies for technologies*

On the one hand the meaning of this technology becomes weak against the development of ionisation technology and in turn its development, even dynamic is possible, because of the development of hybrid technologies of conventional thermo-chemical treatment combined with

PVD coating deposition, or even laser surface treatment. Remembering of a still hypothetical meaning of presented examples to illustrate the newly developed methodology for the evaluation of future technological development, strategic development tracks, although, of course, because of the described example – still a hypothetical one were presented in Figure 8. The most probable strategic development track of this technology assumes the change of environment conditions from neutral autumn to risky summer (2015-2020) which, with simultaneous intensive strengthening of potential and maintaining high attractiveness characteristic for a soaring cypress, will allow the technology to shift to the field of the wide-stretching oak on 2025, which will be accompanied by an improvement of environment conditions (2030). The optimistic strategic development tracks of the gas nitriding assumes that the technology will be temporarily placed in a risky environment (2015); however, thanks to its high attractiveness and actions aiming at strengthening its potential it will be placed in the wide-stretching oak field already in 2020. Next, the technology potential will undergo further strengthening which, assuming its large attractiveness and increasingly favourable environment conditions, will in 2030 cause the technology to be placed in the most attractive matrix field corresponding to the position of an oak during spring. The pessimistic variant, expressed through the third set strategic development track of technology (E) assumes the exacerbation of the world crisis within a short period of time (2015) because of the unfavourably developing political and economic situation. Although actions will be undertaken aimed at strengthening the technology potential, crowned by partial success, and the environment situation will improve (year 2020), the technology will dramatically lose its attractiveness in 2025, shifting to the field of aspen during autumn. Subsequent years will lead to a further deterioration of the situation so that in 2030 technology (E) will find itself placed in the weakest matrix field corresponding to aspen during winter, which is related to withdrawing it from the market. In accordance with the adopted manner of conduct, based on the results of works carried out using matrix methods supported by materials science results of experimental research performed on a specialist diagnostic-measuring apparatus, technology roadmaps are created. The worked out technology roadmaps are universal tools which enable presenting, in a unified and clear format, different types of internal and external factors directly and indirectly characterizing given technologies, taking into account the ways of influence, interdependencies and the change of specific factors in time. An exemplary representative (F) technology roadmap prepared for a laser treatment of Al_2O_3 aluminium oxides into the substrate of Mg-Al-Zn casting magnesium alloys was presented in Table 3. The layout of the technology roadmap corresponds

Table 3. *An example (F) technology roadmap prepared for laser cladding Al_2O_3 oxide particles in the surface of Mg-Al-Zn casting magnesium alloys*

TECHNOLOGY ROADMAP		Technology name: *Laser cladding of Al_2O_3 oxide particles in the surface of Mg-Al-Zn casting magnesium alloys*	Research scope: *Laser technologies in surface engineering*	Catalogue No: *M1-19-2010*
When?	Time intervals	TODAY 2010	2020	2030
	All-society and economic perspectives	Creating scenarios of future events	Development of priority innovation technologies	Statistically high quality of technologies
		Creating the Book of information cards concerning future technologies	Using chances and avoiding difficulties	Sustainable development
		Development of information society and intellectual capital	Cooperation to increase innovativeness and competitiveness of economy and intellectual	Knowledge-based economy
Why?	Strategy for technology	Strategy of an oak in spring		
	Environment influence	Sunny spring		
	Technology value	Wide-stretching oak		
What?	Product	Elements corrosive worn, dies, punches, engine components,	MMCs composites, engine components, gearboxes, gear transmission components	Elements of vehicles, sport equipment, future applications
	Product quality at the background of foreign competitors	Moderate (6)	High (8)	High (8)
	Surface	Casting magnesium alloys Mg-Al-Zn		
	Kind of surface coatings/layers	Oxide particles Al_2O_3		
	Improved material properties	Better mechanical properties of elements (hardness), better anticorrosive properties, better tribological properties		
	Diagnostic-research equipment	Light, confocal laser, scanning electron, transmission electron, atomic force microscopes, X-ray diffractometer, X-ray microanalyzer, GDOES spectrometer, hardness, microhardness, scratch testers, profilometer, potentiostat		
How?	Technology	Laser cladding of TiC carbide particles in Mg-Al-Zn casting alloy surfaces		
	Life cycle period	Prototype (8)	Growth (7)	Base (3)
	Production type	Unit and small-scale serial	Small- and medium-scale serial	Unit and small-scale serial
	Production organisation form	Cellular	Cellular rhythmic	Cellular rhythmic
	Machine park modernity	High (8)	High (8)	Moderate (6)
	Automation & robotisation	Quite High (7)	High (8)	Moderate (6)
	Quality and reliability	High (8)	High (8)	Very high (9)
	Proecology	Quite High (7)	Quite High (7)	Very high (9)
Where?	Organisation type	Large and medium-sized enterprises, research and scientific centres, technological parks	Large and medium-sized enterprises, research and scientific centres, technological parks	Small and medium-sized enterprises, research and scientific centres, technological parks
	Represented industrial branches	Automotive industry, aviation, civil engineering, sport	Automotive industry, sport, aviation	Automotive industry, sport, aviation and others
Who?	Staff education level	Very High (9)	Medium (5)	Medium (5)
	Engagement of scientific-research staff	Quite high (7)	Quite high (7)	Quite high (7)
How much?	Capital requirements	Quite high (7)	Quite high (7)	Low (3)
	Production size determining profitability in firm	Medium (5)	Quite high (7)	High (8)
	Production size in the country	Very Low (2)	Medium (5)	Quite high (7)

LEGEND: ⇒ Cause and effect connections; ——▶ Capital connections; ·······▶ Time corelations; ◀—▶ Two-way transfer of data and/or resources to the first quarter of the Cartesian coordinate system in which time intervals are presented on the x-axis, while seven hierarchically organised main layers are organised on the y-axis. Starting from the top, these are the following layers: time, conceptual, product, technological, spatial, staff, quantitative. This enables the setting, in one place, of data on the purpose, subject, manner, place, contractor and cost in relation to the analysed technology, taking into account the changes of these parameters in time. Upper layers placed in the top part of the technology roadmap, specifying general premises, causes and reasons of activity in the process of pulling, act on the fundamental middle layers pertaining to the product and technology. On the other hand, lower layers which specify the organisational-technical details act on the middle layers in an opposite direction, which is referred to in production management as pushing.

4. Conclusions

New engineering materials and the processes of their manufacturing and processing should be subordinated to the needs of the customer and to the products' utility functions, and very often the utility functions of products and their elements depend on the structure and property of surface layers. Material surface engineering is one of the most dynamically developing economy branches in many technologically advanced countries and it is anticipated that during the next decades it will be placed among the most promising advanced material technologies [14-16, 20] which justifies carrying out scientific studies pertaining to strategic developmental directions of this group of technologies. The strategic development directions of material surface engineering may be set in an ordered, facilitated and modernised way based on the results of computer-aided foresight studies intended for scientific anticipating and shaping the future through acquiring knowledge from experts, organizing it and spreading according to the rules of the worked out strategy and to classic studies on the structure and properties of surfaced engineering materials, which constitutes the subject of widespread individual studies [44]. A dozen or so thematic areas are subjected to analysis, in total with approx. 500 technologies, out of which approx. 150 technologies were qualified for further analyses as part of three iterations of surveys studies; at the basis of these studies lies the author's individual idea of e-foresight [43] meaning conducting foresight studies with the use of IT and internet technologies.

In this chapter, methodological grounds were created for material-foresight studies in order to specify the strategic development perspectives of priority innovative technologies of material surface engineering. The worked out methodology has big significance here and may be applied in all types of foresights for formulating strategic development aims of other technologies. In the analysed case it includes especially determining the value of the analysed set of selected technologies for shaping the structure and properties of the surface layer against the environment, working out recommended strategies of conduct, strategic development tracks and creating technology roadmaps of analysed groups of technologies and operation sheets which enable their industrial implementation, taking into account the influence of each of the implemented technology on the quality, structure and properties of surface layers obtained as a result of these technologies. In accordance with the worked out methodology, the subsequently performed research include selecting technology groups for experimental-comparative research, collecting expert opinions, carrying out a multi-criteria analysis and marking its results on the dendrological and meteorological matrices, determining strategies for technologies preceded by rescaling and objectivising test results using mathematical relations, setting strategic development tracks for technologies, carrying out a series of specialist materials science research using a specialist diagnostic-measuring apparatus and creating technology roadmaps and operation sheets.

In order to perform a full cycle of research included in the worked out methodology, several homogenous groups should be singled out among the analysed technologies; the groups are subjected to experimental-comparative research according to the adopted criteria.

Referring to commonly known matrix methods, including the most famous one - BCG (Boston Consulting Group) a dendrological matrix of technology value was created for objectivising the values of specific technologies or their groups, as well as a meteorological matrix of environment influence for specifying the impact force of the micro- and macroenvironment on these technologies. For the purpose of evaluating these groups of technologies, a ten-point universal scale of relative states was adopted, with values ranging from 1 as the minimum level to 10 - corresponding to the highest value perceived as perfection.

In the dendrological matrix of technology value the quaking aspen was differentiated which symbolizes a weak technology with a limited potential and limited attractiveness, whose future success is unlikely; the soaring cypress corresponds to a technology with limited potential but high attractiveness and possible success; the rooted dwarf mountain pine corresponds to a technology with limited attractiveness but with a large potential and possible future success; the wide-stretching oak refers to the best possible situation guaranteeing success, in which the technology has large potential and high attractiveness. The meteorological matrix of environment influence presents graphic results of assessing the influence of external factors on specific technology groups which were divided into negative difficulties and chances which positively influence the analysed technologies, determined based on expert opinions in response to several dozen questions included in an electronic survey. The technology values were illustrated as frosty winter corresponding to the worst possible situation when success is very hard or impossible to obtain; hot summer when the success of the technology under given conditions is possible but charged with risk; rainy autumn corresponding to a neutral situation; and sunny spring which is the best possible variant, in which the success of a given technology under such good conditions is guaranteed. The test results presented in graphic form using a dendrological matrix of technology value and meteorological matrix of environment influence should be placed on a matrix of strategies for technologies, consisting of sixteen fields corresponding to specific variants resulting from the set of combinations of four types of technologies and four types of environment. To facilitate the transfer of specific numeric values from the dendrological and meteorological matrices to the matrix of strategies for technologies, mathematical relations were formulated which enable the rescaling and objectivising of test results with the use of the notions of the relative value of technology V_n and the relative value of environment influence E_n. For specific technologies/groups of technologies the strategic development tracks are specified which constitute a forecast of their development in the years 2015, 2020, 2025 and 2030 in three variants: optimistic, pessimistic and most probable, and they are presented against a matrix of strategies for technologies. The results of carried out experimental-comparative research constitute source data for technology roadmapping for the purpose of a detailed forecasting of each technology's development starting from today, as well as in 10 and 20 years. The vertical axis of the technology roadmap contains seven main layers which include answers to the following questions: When? Why? What? How? Where? Who? How many?, which are additionally divided into sub-layers with a higher degree of detail. The main layers of the technology roadmap were organised hierarchically so that the upper parts contain the most general layers specifying the premises, reasons and causes of realised actions which influence, through „push", the layers placed under them containing technical-organisational details impacting, through „pull", the layers placed above. On the other hand, the middle part of the technology roadmap pertains to the essence of the analysed problem by characterizing the product and technology used for its manufacturing. The technology roadmap presents relations between its specific layers and sub-layers, with a division into: cause-and-effect relations, capital relations, time correlations and two-way flows of data and/or resources, visualised using differentiated arrows. Such created technology roadmaps function as a tool of comparative analysis, facilitating the selection of the best technology in terms of the specified selected criterion, and when supplemented by operation sheets with precise technological details – they enable the implementation of a selected technology in industrial practice.

A very important element of the worked out methodology is the inclusion of detailed materials science research whose description, because of objective reasons standing from a countless number and diversity of possible variants, is impossible to realise in only one chapter. One should underline the significance of materials science research of the surface layer structures created using various surfacing methods, research of mechanical and tribological properties, as well as research of utility functions under exploitation or similar conditions. The results of these research in relation to technologies selected through the previously described technology valuating methods constitute an important premise for working out and experimentally verifying the evaluations made using methods of working out matrices of strategies for these technologies and they are necessary for creating a technology roadmap and an operation sheet. A synergic impact of the materials science and foresight researching methods guarantees the accuracy and compatibility of evaluations made according to the worked out methodology. The unity of place and time is not necessary for these research realisation and does not in any way influence the correctness of the concluding process carried out as part of the technology foresight; however, in sure abandoning of these research has negative influence for foresight realisation correctness.

The worked out tools have broader significance and enable the forecasting of the development of various technologies pertaining not only to surface engineering, in different time intervals with the consideration of various factors which characterize the analysed technologies. It is possible to use them at the initial, general stage of the realised technology foresight when opinions of few key experts are used, as well as at the final stages of formulating results of foresight research for evaluating the results of survey studies carried out among a large group of trade experts. If it is anticipated to carry out studies using the Delphi method, the described method may also be useful. In accordance with the worked out e-foresight methodology, the performance of survey studies is expected through an original developed method which uses modern IT tools for obtaining the adopted aims: virtual organisation, internet platform and neuron networks. Attention must be paid also to an important practical feature of the proposed approach which enables the application of the worked out methodology with regard to each detailed technology of manufacturing a specific final product, as well as to different sizes of technology groups if their division is possible in terms of a clearly defined objectively verified criterion. As part of a broad scope of the currently realised individual studies, the working out of many groups of surfacing technologies was anticipated, using the mentioned newly-developed tools, which will certainly be the subject of further publications in which the methods described in this work will be applied.

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5 Assessment of strategic development perspectives of laser treatment of casting magnesium alloys

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Abstract

Purpose: *The purpose of this chapter is to assess the strategic perspectives of laser treatment of casting magnesium alloys such as MCMgAl12Zn1 MCMgAl9Zn1, MCMgAl6Zn1, MCMgAl3Zn1 using carbide TiC, WC, VC, SiC and* Al_2O_3 *oxide powders. The type of powder was taken as a criterion for the technology groups distinguishing in that way five groups of technologies for further research studies.*

Design/methodology/approach: *In the framework of foresight-materials science research: the dendrological matrix of technology value, the meteorological matrix of environment influence and the matrix of strategies for technology with the strategic development tracks were made, X-ray microanalysis, qualitative X-ray analysis, hardness tests and roughness measurements the research were carried out under the scanning electron microscope and the light microscopy, as well as technology roadmaps were prepared.*

Findings: *The outcarried research pointed out very high potential and attractiveness of the given technologies in the background environment and the promising improvement of mechanical properties of examined materials.*

Research limitations/implications: *Described materials science and foresight research concerning the cladding and remelting of carbides and oxides in the surface of casting magnesium alloys are a part of a wider research project aiming to define, examine and characterise innovative technology of surface engineering of engineering materials.*

Practical implications: *The presented results of experimental materials science research prove a significant positive effect of laser treatment with the use of carbides and oxides on the* *structure and properties of casting magnesium alloys that is why it is legitimate that they are included in the set of innovative technologies qualified for use in an industrial practice including small and medium enterprises.*

Originality/value: *The value of this chapter is to determine the value of laser treatment technology of casting magnesium alloys in the background environment with a recommended procedure strategies, the strategic development tracks and technology roadmaps including the influence of this treatment on the quality, microstructure and properties of surface layers obtained by cladding and remelting casting magnesium alloys.*

Keywords: *Manufacturing and processing; Laser surface treatment; Magnesium alloys; Foresight; Technology Roadmapping*

This chapter has been also published as:

A.D. Dobrzańska-Danikiewicz, T. Tański, S. Malara, J. Domagała-Dubiel, Assessment of strategic development perspectives of laser treatment of casting magnesium alloys, Archives of Materials Science and Engineering 45/1 (2010) 5-39.

1. Introduction

One of the tasks under the policy guidelines for science, technology and innovation for decades to come is a knowledge-based economy, which thrives on creating treated as a production and distribution and the practical use of knowledge and information. The basis of economic development are, therefore, production, distribution and implementation and knowledge which is the product of a major contribution to sustainable development. Effects of innovation and thus the competitiveness of manufacturers of products in international markets are of course dependent on the adopted strategy of development of technology [1]. It is necessary to choose a strategy of integration of various advanced fields of science and technology and to achieve synergies in the development of new technologies. Materials processing technologies and new materials are a key range of research and development. which are essential for industry and other areas of application of these technologies. Generalisation of the results of a European Foresight on various new materials and different technologies, materials processing [2, 3] is the expectation to produce materials with properties of the products with appropriately shaped structure and ordered by the users. Preparation of materials that meet needs of manufacturers of commercial products at the right time and place, called materials on demand, is a priority of new material technologies and manufacturing processes [4, 5]. Continuing desire of designers is the will to develop and produce the ideal material that would account for both the maximum possible resistance to wear in the operating conditions and high ductility. By their very nature such a combination of properties is impossible to obtain. To one of the most widely studied concept in the world of customisation (called tailoring) properties of various elements to the operational requirements. as one of the possibilities if only partial implementation of this postulate, it must be the development of graded materials, including the gradient of appropriately shaped surface properties of the surface layers of engineering materials [4-38]. Gradient properties of these materials are achieved by changing the location of structure, phase composition, chemical composition or arrangement of atoms. As a result of the proper selection of the element material together with the processes determining its structure and properties and a kind and technology of the surface layer, ensuring the required utility properties, the best combination of properties of the core and the surface layer of a produced element is possible. The concept of the gradient structure and materials properties can also deal with the complement of losses in the long-term exploited elements of machines and equipment, and the formation of the structure and surface of re-produced constructional elements being fundamentals of re-manufacturing. Defining the leading technology and strategic research directions for methods of forming the structure and properties of engineering materials surface is a condition for the outworking of own development strategies for many small and medium-sized enterprises and the improvement of their competitiveness, as a result of the application and the development of advanced technologies of surface treatment, as an essential part of manufacturing technologies and determines more flexible adaptation of production to market needs. Surface treatment technologies of engineering materials are most often used in many sectors of industry, and the branch of surface treatment and surface coating is one of the most dynamically developing sectors of economy [3, 4]. In order to direct the development of the most advantageous technological solutions of forming structure and properties of the surface layers of products and their components made from engineering materials, from the viewpoint of improving the competitiveness of enterprises and for the development of their proinnovative activities, for the intensification of the transfer of knowledge to economy, and the improvement of utility properties, production durability and reliability, research by foresight methods on the prospects for the development of these technologies in relation to different groups of engineering materials are necessary [5, 6, 39-48]. The scale of the own research planned in this area has become the reason of outworking of the development of methodology and information technology, which would assign, improve and modernise the outcarried foresight research. Thus the idea of e-foresight [40], with reference to known and commonly used terms [49-51]: e-management, e-business, e-commerce, e-banking, e-logistics, e-services, e-government, e-education always meaning certain activities with the use of computer networks, particularly the Internet was born. The main aim of those research is to identify priority directions of innovative technologies and strategic research on methods of forming the structure and surface properties of engineering materials, whose development will be crucial over the next 20 years.

Among various processes of surface treatment and manufacturing of gradient materials, the specific role is played by methods of laser surface treatment, described in the literature in the relation to selected engineering materials [35-38, 52-71], and also developed in the own works in the Department of Materials Processing Technology, Management and Computer Science of the Institute of Engineering Materials and Biomaterials, the Silesian University of Technology [72-88]. Advantages which characterise laser surface treatment processes, namely: short process time, flexibility, and operational precision cause that this method takes advantages over other methods used in surface engineering. The main objective of laser remelting of materials surface layers is the formation of structures and related properties. With the creation of chemically homogeneous, fine crystalline surface layer without changing the chemical composition, the increase of resistance mainly wear one and thermal fatigue is achieved. Even better results in the increase of utility properties can be obtained by alloying material surface layer by hard phases of carbides, oxides or nitrides. Mentioned technologies are more often applied to magnesium alloys [89, 90], what among others is the subject of the own work [91-94]. During the last decade in the world the rapid increase of the use of magnesium and its alloys almost in every field of contemporary industry is observed [89-105]. This is due to numerous properties of magnesium, which allow for its use both as a structural element and an addition to other metal alloys. It is 35% lighter than aluminium (2.7 g/cm^3) and more than four times lighter than steel (7.86 g/cm^3). Magnesium alloys in spite of low density (1.7 g/cm^3) also have other advantages such as good ductility, better than in aluminium, noise and vibration damping, very good castability, big dimensional and shape stability, low shrinkage, low density combined with high strength compared to lighter weight. They can also be recycled, which makes recycled alloys having high quality and properties which are very similar to those of original casting alloy and allow to use these materials, instead of newly manufactured magnesium alloys for the construction of minor importance [29, 80, 94-105]. Lower weight and high strength enable to produce components made of this material by casting, plastic deformation, mechanical working or welding. Magnesium alloys including aluminium, manganese, rare earth metals, thorium, zinc and zirconium present the increased strength ratio on weight, making them important materials in applications where weight is important and necessary to reduce the forces of inertia [97]. The need of the application of magnesium alloys is associated mainly with the development of the automotive and aerospace industries.

Advantageous properties of magnesium and its alloys with the advantages of laser surface treatment were the basis of making a series of interdisciplinary foresight and materials science research to determine the value, attractiveness and potential of laser treatment technology of casting magnesium alloys at the background of micro- and microenvironment together with the outworking of the recommended strategy, strategic development tracks and roadmaps of analysed technologies, taking into consideration the impact of laser treatment on quality, structure and properties of the surface layers of casting magnesium alloys.

This chapter is a practical application of foresight and materials science research methodology [40, 41] to laser surface treatment of Mg-Al-Zn selected alloys basing on the evaluation by a small group of key experts with the intention of repeating this described foresight research with the participation of a wide group of trade experts and on the basis of these evaluations worked out by them in the framework of questionnaire surveys. Casting magnesium alloys: MCMgAl12Zn1, MCMgAl9Zn1, MCMgAl6Zn1, MCMgAl3Zn1 undertook the experimental research such as laser cladding and remelting with TiC, WC, VC, SiC carbide and Al_2O_3 oxide powders.

Such research as: X-ray microanalysis, qualitative X-ray analysis, hardness tests and roughness measurements were made under the scanning electron microscope and the light microscope. Materials science and foresight research are a part of a wider actions aiming to isolate, examine, characterize and define strategic developmental perspectives of priorities and innovative technologies of surface engineering of engineering materials, qualified for the application in industrial practice, including small and medium-sized enterprises.

The consideration of this issue at a higher level of generality leads to generate a set of priority innovative technologies of surface engineering, contributing to the statistical increase in the quality of applied technology in industrial enterprises, stimulating sustainable development and strengthening the knowledge-based economy.

2. Implemented research methodology

The outcarried research are interdisciplinary and applied methodology of research deals mainly with technological foresight being an element of the field of knowledge called organisation and management, and surface engineering being a part of the broader understood materials science. At certain stages of research also methods deriving from artificial intelligence, statistics, information technology, machine building and operation, strategic and operational management were used.

According to the taken methodology performed research include in turn [41]: selecting groups of technology for experimental and comparative research, collecting experts' opinions, carrying out multi-criteria analysis and putting its results to a dendrological and meteorological matrix, determining strategy for technology preceded by graduating and making objective results of research with the use of the formulated mathematical relationships, identifying strategic development tracks and performing a series of specialised materials science research using professional diagnostic and measurement equipment and making roadmaps of technology. According to the taken methodology of foresight and materials science research first of all among analysed technologies it is necessary to extract a few as far as possible homogeneous groups, in order to undergo the scheduled research having experimental and comparative character.

To determine the objective values of given selected technologies or their groups (as in the case of research described in this chapter) a dendrological matrix of value technology is used, and to determine the strength of positive and negative influence of the environment on a given technology a meteorological matrix of environment influence is used. The methodological construction of those both matrices refers to portfolio methods, commonly known in sciences about management, and first of all to BCG matrix [106] owing its incredible popularity because of its simple associations and intuitive reasoning, which serves as an inspiration when creating the methodological assumptions of meteorological and dendrological matrices [41]. To evaluate various groups of technology paying a special attention to their value and influence of the environment a 10-point universal scale of relative states, where 1 is the minimum level 2 – very low, 3 – low, 4 – quite low, 5 – intermediate, 6 – moderate, 7 – quite high, 8 – high, 9 – very high, and the highest value of 10 is the level of excellence was adopted.

A dendrological matrix of technology value presents graphically the results of the evaluation of given groups of technology taking into consideration their potential which is a real objective value and attractiveness of technology that illustrates how technology is subjectively perceived among its potential users. The potential of a given group of technology applied to the horizontal scale and attractiveness of technology applied to the vertical scale of a dendrological matrix is a result of multi-criteria analysis using the results of the experts' evaluation. Depending on the value of the potential and the level of attractiveness of which were identified in the framework of the experts' evaluation given technology can be placed in one of the following quarter of the matrix:

- A sparing aspen which is technology with a limited potential and limited attractiveness in the range, which a future success is unlikely;
- A quaking cypress which corresponds with technologies with a limited potential, but highly attractive, what causes that a success of technology is possible;
- A rooted dwarf mountain pine which is technology with limited attractiveness, but a high potential, so that its future success is possible;
- A wide-stretching oak which corresponds to the best possible situation in which the analysed technology has both a great potential and great attractiveness, which is a guarantee of a future success.

A meteorological matrix of environment influence presents graphically the results of the influence evaluation of external factors on different groups of technologies, which were divided into difficulties and opportunities that influence negatively and positively the analysed technologies. Experts' questionnaire poll on external factors affecting technology took place on the basis of a questionnaire consisting of dozens of questions about micro-and macro environment in strictly defined proportions. Difficulties arising from the environment were put on the horizontal scale of a meteorological matrix, and on the vertical scale of this matrix opportunities that is positive environmental influence factors were put. Depending on the level of the influence of positive and negative factors of the environment on the analysed technology, defined in the framework of the experts' evaluation in a 10-point scale the level of technology is placed in one of the following quarter of the matrix:

- Freezing winter corresponding to the worst possible situation in which the environment brings a large number of problems and few opportunities, which means that success in a given environment is difficult or impossible to achieve;
- Hot summer corresponding to a situation in which the environment brings a lot of opportunities, which, however, are accompanied by many difficulties, meaning that the success of technology in the given circumstances is possible, but is a subject to the risk;
- Rainy autumn corresponding to the neutral position, in which for given technology traps do not wait, but also the environment does not give too many opportunities;
- Sunny spring being the best option denoting friendly environment with lots of opportunities and a little number of difficulties, which means that the success of given technology is guaranteed.

At the next stage of research their results presented in a graphical form using a dendrological matrix of technology value and a meteorological matrix of environment influence were put on a **matrix of strategy for technology** consisting of sixteen boxes corresponding to each set of versions resulting from the combination of the types of technology and the types of environments. A matrix of strategy for technology presents graphically the place of technology taking into consideration its value and environment influence and indicate the conduct strategy which should be taken with respect to a given technology, taking into account the previously analysed factors. To allow for transferring specific numerical values of dendrological and meteorological matrices measuring [2x2] to a matrix of strategy for technology measuring [4x4] the terms: the relative value of technology V_n and the relative value of environment influence $E_{n.}$ and mathematical dependence allowing to graduate and make objective research results were introduced [41].

The next stage of research comes down to **the strategic development tracks** for different technologies / groups of technologies, which forecast their development successively in: 2015, 2020, 2025 and 2030 in three versions: optimistic, pessimistic and most likely ones, followed by their visualisation against a background of a matrix of strategy for technology.

In order to precise the value of the potential and attractiveness of laser treatment of casting magnesium alloys **a series of metallographic research** using specialised diagnostic and measurement equipment were carried out. The investigations were carried out on test pieces of MCMgAl12Zn1, MCMgAl9Zn, MCMgAl6Zn1, MCMgAl3Zn magnesium alloys in as-cast and after heat treatment state. The chemical compositions of the investigated materials are given in Table 1. A casting cycle of alloys was carried out in an induction crucible furnace using a protective salt bath Flux 12 equipped with two ceramic filters at the remelting temperature of 750±10ºC, suitable for the manufactured material. In order to maintain metallurgical purity of the remelting metal, a refining with a neutral gas with the industrial name of Emgesalem Flux 12 was carried out. To improve the quality of a metal surface a protective layer Alkon M62 was applied. The material was cast in dies with betonite binder because of its excellent sorption properties and shaped into plates of 250x150x25. The casting alloys were heated in an electrical vacuum furnace Classic 0816 Vak in a protective argon atmosphere. The heat treatment involved the solution heat treatment (warming material in temperature 375°C the 3 hours, warmed in the temperature to 430°C, held for 10 hours) and cooled in air and then aged at temperature of 190°C, holding for 15 hours and cooling in air (Table 2). Next, MCMgAl12Zn1, MCMgAl9Zn, MCMgAl6Zn1 MCMgAl3Zn1 magnesium alloys were used as substrate materials to laser surface treatment using a high power diode laser. Plates were polished with 1200-grit SiC paper prior to laser surface treatment to obtain smooth surface and then cleaned with alcohol and dried. Five types of powders were used in a present study for alloying process, namely titanium, tungsten, vanadium and silicon carbides as well as aluminium oxide (Table 3). Laser treatment was made using the Rofin DL020 HPDL high power diode laser in the argon shield gas cover to protect the molten metal pool from oxidation with the technique of the continuous powder supply to the remelted pool area, by feeding the granulate using the TecFlo fluidisation feeder equipped with the powder flow digital controller (Fig. 1a). Powder feeder was connected with the transport gas cylinder and powder feed nozzle (Fig. 1b). A gas feed rate was 5 l/min.

Table 1. *Chemical composition of examined alloy*

The mass concentration of main elements, %							
Material type	Al	Zn	Mn	Si	Fe	Mg	Rest
MCMgAl12Zn1	12.1	0.62	0.17	0.047	0.013	86.96	0.0985
MCMgAl9Zn	9.09	0.77	0.21	0.037	0.011	89.79	0.0915
MCMgAl6Zn1	5.92	0.49	0.15	0.037	0.007	93.33	0.0613
MCMgAl3Zn	2.96	0.23	0.09	0.029	0.006	96.65	0.0361

Table 2. *Parameters of heat treatment of examined alloy*

Sing the state of heat treatment	Conditions of solution heat treatment		
	Temperature, °C	Time of heating, h	Way coolings
0	As-cast		
	Solution treatment		
1	430	10	Water
	Aging treatment		
2	190	15	Air

Table 3. *Properties of powders used to alloying process*

Property	WC	TiC	VC	SiC	Al_2O_3
Density, kg/m^3	15.69	4.25	5.36	3.44	3.97
Hardness, HV	3400	1550	2850	1600	2300
Remelting temperature, °C	2870	3140	2830	1900	2047
Average of size grain, µm	0.7-0.9	<1.0		<10	1-5
	>5	>6.4	>1.8	<75	80

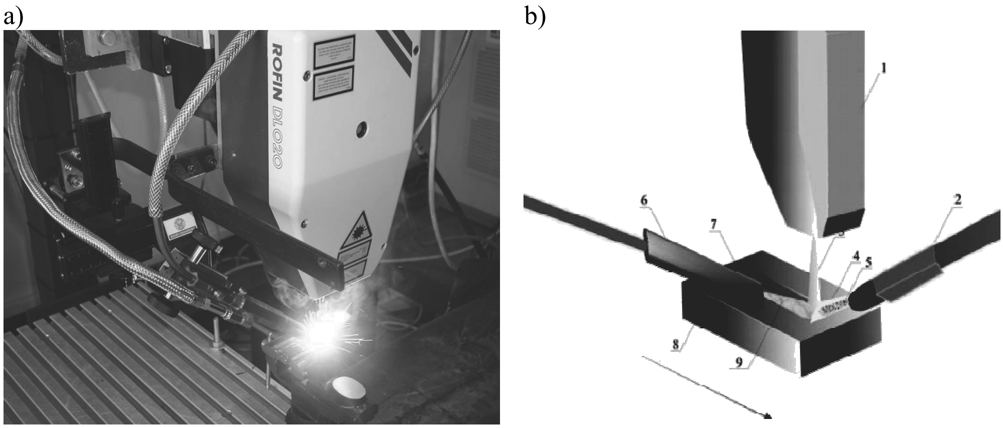

Figure 1. *a) Laser HPDL Rofin DL 020 in working process, b) laser treatment scheme for casting magnesium alloys: 1 − laser head, 2 − transport gas cylinder and powder feed nozzle, 3 − beam laser, 4 − gas, 5 − powder, 6 − powder feed nozzle, 7 − remelting zone, 8 − base material, 9 − protective gas*

The laser treatment of casting magnesium alloys was conducted by remelting Mg-Al-Zn substrate and feeding of carbides or oxides particles performed by a high power laser diode HPDL Rofin DL 020 under argon shielding gas. The parameters are presented in Table 4. Argon was used during laser remelting to prevent oxidation of the coating and the substrate. Prior to approach laser treatment, powders were desiccated in the furnace in temperature of 100°C. Experiments were made with the following process parameters: laser power 1.2-2.0 kW;

alloying feed rate 0.5-1.0 m/min; powder feed rate: 6-9 g/min. After initial experiments laser power in the range 1.2-2.0 kW was assumed for the investigations, with alloying feed rates of 0.25; 0.50; 0.75; 1.00 m/min. The examinations revealed that the optimum geometry of a single laser path was obtained for alloying with the feed rate of 0.75 m/min. However, for laser treatment with powder injection of Al_2O_3 powder the optimum feed rate was 0.50 m/min and 0.25 m/min, respectively. This distinction probably is the result of different laser radiation absorption for each powder and each magnesium alloy. The optimisation of process parameters was made for the sake of mixture quality, distribution uniformity of alloying particles in the remelting zone and substrate geometry after laser treatment.

Table 4. *HPDL Rofin DL 020 parameters*

Laser wave length, nm	808-940
Focus length of the laser beam, mm	82
Power density range of the laser beam in the focus plane, kW/cm^2	0.8-36.5
Dimensions of the laser beam focus, mm	1.8 x 6.8

The metallographic examinations were made on casting magnesium alloys specimens mounted in thermohardenable resins. The observations of the investigated casting materials were made on the light microscope LEICA MEF4A as well as on the electron scanning microscope ZEISS Supra 35. Phase composition and crystallographic structure were determined by the X-ray diffraction method using the X'Pert device with a cobalt lamp, with 40 kV voltage. The measurement was performed in angle range of 2Θ: 20-130º. Hardness tests were made using Zwick ZHR 4150 TK hardness tester in the HRF scale. Roughness measurements of surface layers of laser cladded casting alloys were performed on Taylor Hobson Precision Surtronic 3+. Measuring device is characterised by measuring resolution 0.2 μm and measuring range to 150 μm. Measurements were made on distance 0.8 mm. The X-ray qualitative microanalysis and the analysis of a surface distribution of casting elements in the examined casting magnesium alloys specimens were made on transverse micro-sections on the ZEISS Supra 35 scanning microscope with the Oxford EDS WDS dispersive radiation spectrometer at the accelerating voltage of 15 kV.

In order to verify the correctness of the experimental values of hardness after laser cladding of Mg-Al-Zn, casting magnesium alloys model used a designed neural network, constructed on the basis of experimental data: the kind of used powder, the concentration of aluminium in the alloy, the laser power and speed of alloying – as the input variable – and HRF-hardness as the output variable, were used. The data set was divided into three subsets: learning

(48 cases), validation (23 cases) and test (24 cases) ones. The fundamentals of the assessment of the network quality were the three characteristics of regression: average absolute error, the quotient of standard deviations, and Pearson's correlation coefficient. The quotient of the standard deviation is a gauge of the model quality used to solve regression problems. It is determined by dividing the standard deviation of prediction error and standard deviation of the output variable. A smaller value indicates a better gauge of the quality of prediction, because the smaller it is, the larger the variance explained by the model is. As a result of design and optimisation of selected one-way network MLP (multilayer perception) with 4 neurons in input layer – corresponding to the input variable: the nature of the powder (nominal variable), the concentration of aluminium in the alloy, the laser power and speed of alloying (numerical variables) and one numerical output variable (hardness HRF) (Fig. 2) were selected.

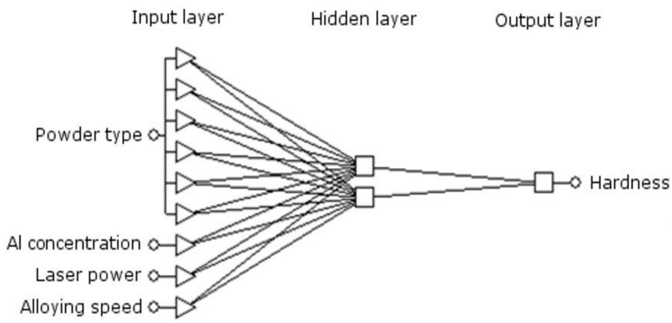

Figure 2. *Scheme of MLP neural network calculates hardness*

For a nominal input variables conversion technique of one of Zn was used, while for numerical input variables and output variable the technique of conversion of variable minimax was used. The number of layers of the network was identified as three layers with two neurons in the hidden layer. The activation function in the input and output layers was defined as a linear with saturation, and in the hidden layer as the logistics, but for all the layers PSP linear functions were used. Networks were taught by methods of back propagation of errors (50 epochs learners) and conjugate gradients (62 students ages). Table 5 presents the error values, the quotient of standard deviation and correlation coefficients of designed neural networks, which were the basis for evaluating the characteristics of the network. Figure 3 shows a chart comparing the HRF hardness values provided by the network and the experimentally measured and of the trend line for a set of test was determined. On the basis of achieved indicators to assess the quality of the neural network i.e., Pearson's correlation coefficients for a set of test between the calculated and actual values of output: 0.90 in the training set, 0.90 in the validation set and 0.89 in the test set, and the quotient of standard deviations for the training and test sets: <0.47 one can be inferred about the accuracy in predicting the value of the output network (HRF hardness).

Table 5. *Regression statistics of neural network calculating hardness value in data sets*

Indicators of quality assessment models	Data set		
	Training	Validating	Testing
Error, HRF	5.35	6.49	5.90
Standard deviation	0.43	0.44	0.46
Pearson's correlation coefficient	0.90	0.90	0.89

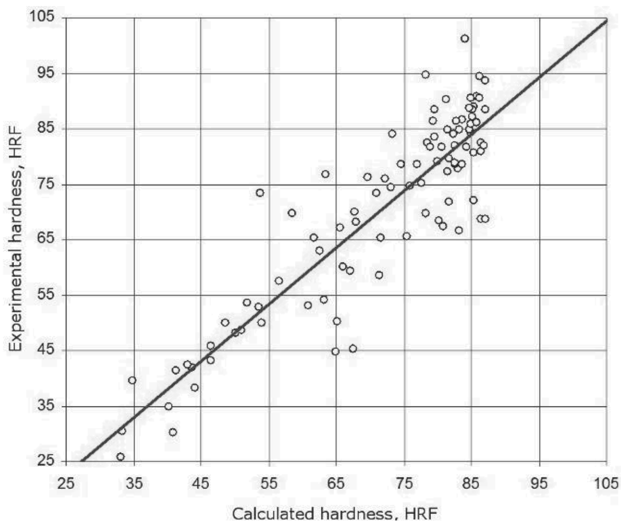

Figure 3. *Real hardness value and calculating value comparison for all test data sets*

Results of made experimental and comparative research are source data to create **technology roadmaps**. The arrangement of a technology roadmap worked out for realised research corresponds to the first quarter of the Cartesian coordinate system. On the horizontal axis there are the forward-looking time intervals, and a time horizon of the whole research put on the roadmap equals 20. On the vertical axis of a technology roadmap seven main layers, concerning the defined ranges sequentially relating to the questions: When? Why? What?

How? Where? Who? How much? are placed. Major layers of a technology roadmap are organised hierarchically starting from the top, most general ones determining causes and reasons for realised actions, through the middle ones characterising a product and ending with technology at lower layers precising organisational and technical details. The middle layers of a technology roadmap are a subject to two types of influence – suction from the upper layers and pushing from the bottom layers. On the roadmap using different arrows the links between its various layers and sublayers, with the division into: cause and effect connections, capital connections, time correlation and two-time correlation of data flows and / or resources are presented. Technology roadmaps are a very convenient tool for comparative analysis, making easier to select the best technology in the respect of selected criteria, and complemented with technological cards containing details of technological specifications allowing for the implementation of technology in industrial practice. An important feature of technology roadmaps is their flexibility, so that if necessary they can complement and extend for additional sublayers adjusting them to user's expectations.

3. Research results of technology values and their development tracks

The results of research described in this chapter include, firstly, the evaluation of the potential and attractiveness of analysed technologies against the background of micro- and macroenvironment carried out on the basis of opinions of key experts expressed in a 10-point universal scale of relative states and the recommended strategy for dealing with a particular technology, together with strategic development tracks resulting from that evaluation. Then the results of materials science experiments examining the effects of laser cladding and remelting with the use of carbide and oxide powders on structure and properties of surface layers of casting magnesium alloys were presented. Supplementing these research is the experimental verification of the developed technology carried out to determine quality of manufactured surface layers, made using high power diode lasers on a finished item. It includes the following metallographic research: light and scanning microscopy, X-ray phase analysis and qualitative analysis, analysis of surface distribution of alloyed elements and examination of mechanical properties, including hardness, microhardness and roughness. On the basis of experimental and comparative research results technology roadmaps, showing a clear uniform format of various kinds of internal and external factors directly and indirectly characterising given technologies taking into consideration ways of interactions, interconnections, and changes of individual factors in time, were created.

Taking as a criterion of the division, a type of powder deposited to the substrate, in order to carry out comparative and experimental works, five homogeneous groups were isolated from the analysed technologies in turn:

(A) Mg-Al-Zn casting magnesium alloys which underwent laser treatment by TiC titanium carbide;
(B) Mg-Al-Zn casting magnesium alloys which underwent laser treatment by WC tungsten carbide;
(C) Mg-Al-Zn casting magnesium alloys which underwent laser treatment by VC vanadium carbide;
(D) Mg-Al-Zn casting magnesium alloys which underwent laser treatment by SiC silicon carbide;
(E) Mg-Al-Zn casting magnesium alloys which underwent laser treatment by Al_2O_3 aluminium oxide.

Each group of technology was evaluated by experts using the 10-point universal scale of relative states paying attention to economic, humanistic, natural, systematic attractiveness and creative, application, quality, developmental, technical potential. Using the multi-criteria analysis a weighted average of the considered criteria (attractiveness and potential) was calculated, and the result obtained for given groups of technology was put on a dendrological matrix of technology value (Fig. 4). The conducted analysis showed that all groups were classified to the most promising quarter called the wide-stretching oak, covering technologies with both great potential and attractiveness. The best result A (9.65, 9.75) was reached by casting magnesium alloys laser treated by titanium carbide, and the worst one used for laser treatment were silicon carbides D (7.55, 8.45).

The evaluation of positive and negative environmental influences of technology on various groups was performed using the meteorological matrix of environment influence. Results of multi-criteria analysis acquired in the questionnaire process of experts evaluation was applied to this matrix, as shown in Fig. 5. The questionnaire used to research includes a few dozen or so questions about the influence of micro- and microenvironment on technologies in specific proportions. Results of those research show that in all groups of technology which were examined environment is extremely favourable, brings a lot of opportunities, and little difficulty. Hence, all evaluated groups of technology were in the quarter of the sunny spring, very well predicting their development. Again the highest grade was achieved by rating a group of technology identified as A (4.04, 7.36), and the lowest group of technologies was marked as E (3.77, 6.02).

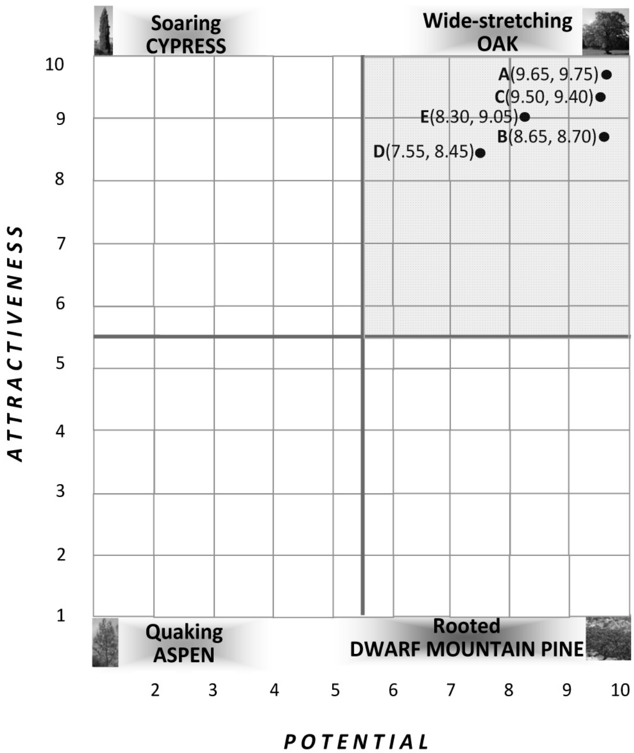

Figure 4. *The dendrological matrix of technology value for the laser cladding and remelting of casting magnesium alloys using TiC (A), WC (B), VC (C), SiC (D) carbide and Al_2O_3 oxide (E) powders*

Results presented in a graphical form using a dendrological matrix of technology value and a meteorological matrix of environment influence were put on a matrix of strategy for technology in the next stage of research work (Fig. 6). This matrix is a graphical representation of individual groups of technology of casting magnesium alloys laser treatment, with carbides and aluminium oxide taking into consideration their values and the environment influence indicating the appropriate strategy for conduct. In order to transfer specific numerical values from a dendrological matrix and a meteorological matrix to a matrix of strategy for technology having other size, mathematical dependence allowing to graduate and make objective research results were used [41]. For all analysed very promising groups of technology, it is recommended to use the strategy of an oak in the spring, relying on developing, strengthening and implementing attractive technology with a great potential in industrial practice with reference to a spectacular success.

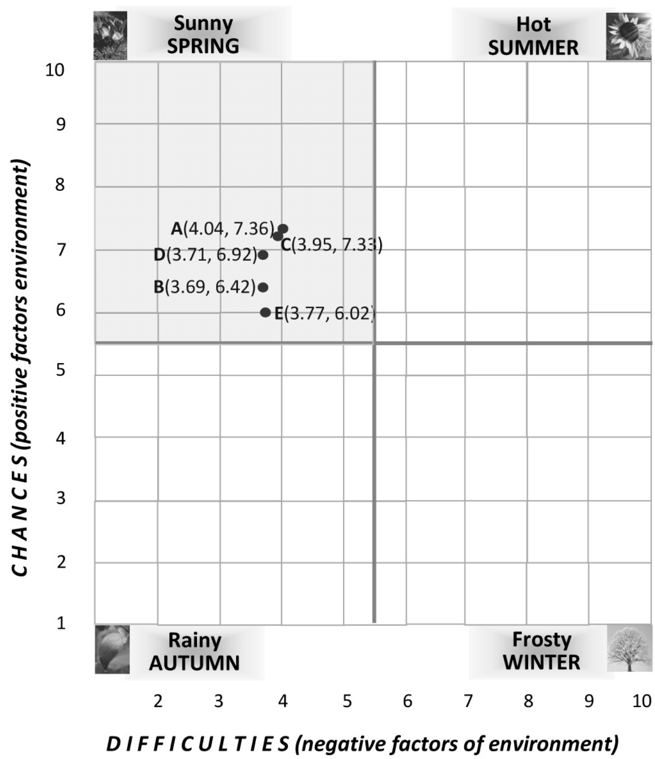

Figure 5. *The meteorological matrix of environment influence for the laser cladding and remelting of casting magnesium alloys using TiC (A), WC (B), VC (C), SiC (D) carbide and* Al_2O_3 *oxide (E) powders*

The next stage of research comes down to determine the strategic development tracks for specific technology / groups of technologies on the basis of experts' opinions which forecast their development successively in: 2015, 2020, 2025 and 2030 in three variants: optimistic, pessimistic and most likely ones, followed by their visualisation at the background of the matrix of strategies for technology. A representative example of a graphical matrix of strategies for technology with the marked strategic development tracks in three versions made for laser treatment of casting magnesium alloys by TiC titanium carbides is presented in Fig. 7.

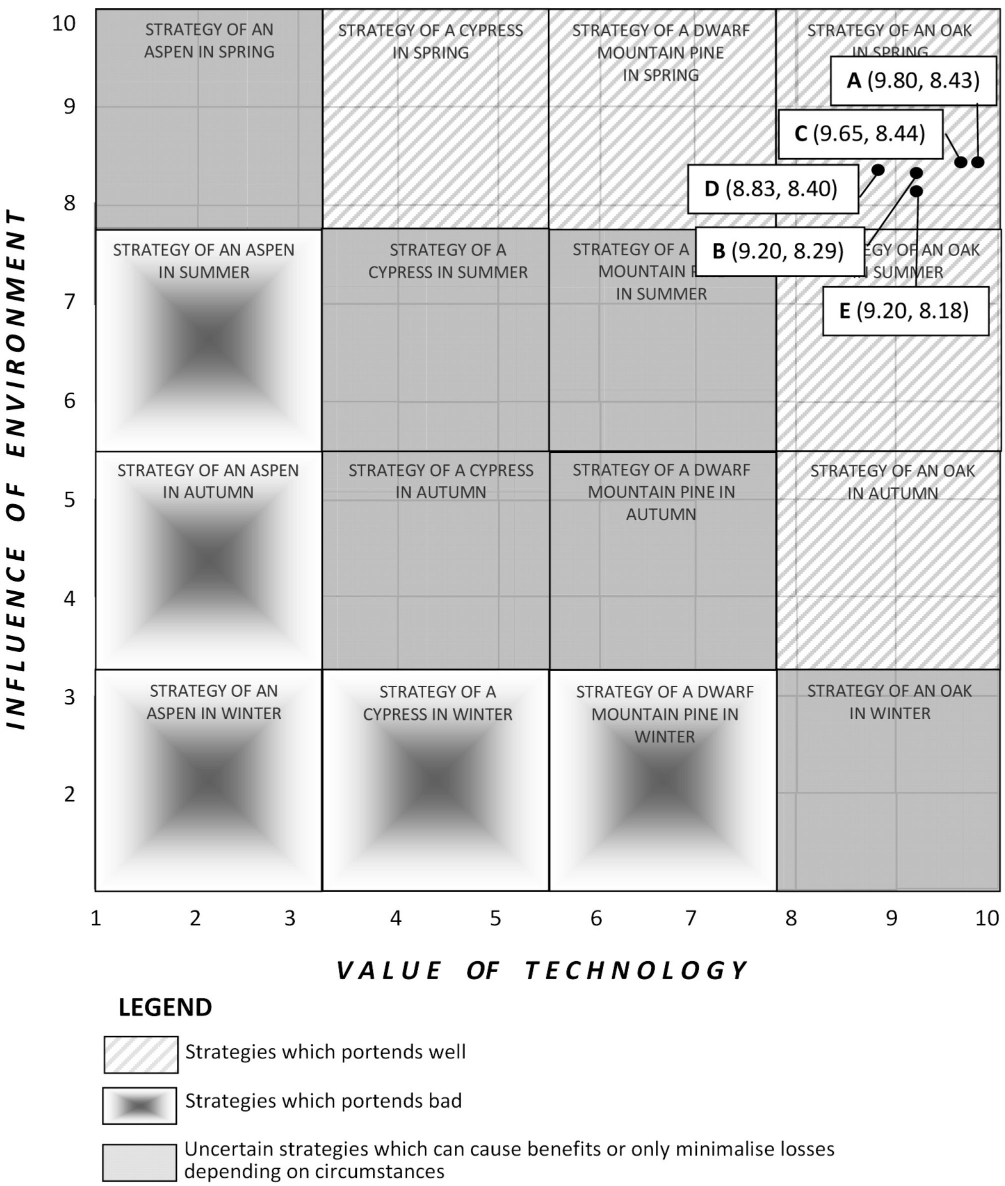

Figure 6. *The matrix of strategies for technology called the laser cladding and remelting of casting magnesium alloys using TiC (A), WC (B), VC (C), SiC (D) carbide and Al_2O_3 oxide (E) powders*

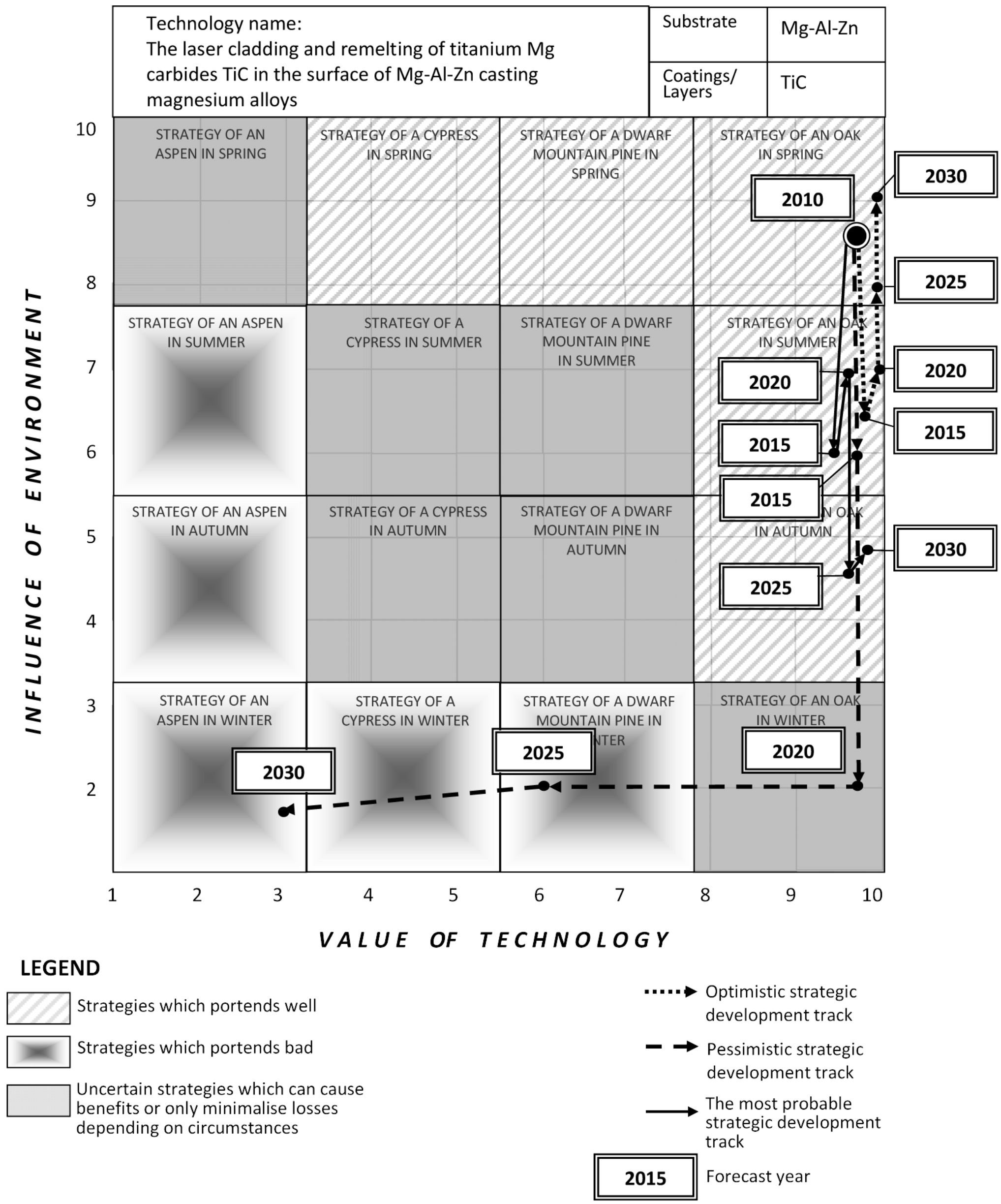

Figure 7. *The strategic development tracks for demonstration technology called the laser cladding and remelting of casting magnesium alloys using TiC (A) carbide powders*

The most probable strategic development track of this group of technology assumes a change of environment from friendly spring into risky summer maintaining a high potential and attractiveness characteristic for a wide-stretching oak. In the next years the environment will become more stable passing into the phase of autumn. In this case the success of attractive and stable technology on the predictable market while seeking for new markets, new groups of potential customers and new products possible for manufacturing by given technology is foreseen. An optimistic strategic development track for laser treatment of casting magnesium alloys by TiC titanium carbides assumes that despite of the transitional appearance (2015-2020) of numerous difficulties in the environment, opportunities which appear parallelly will be used and in the coming years they will determine the development of this group of technology

Table 6. *Strategic development tracks of laser treatment of Mg-Al-Zn casting magnesium alloys using carbide and oxide powders. Types of strategic development tracks: (O) − optimistic, (P) − pessimistic; (MP) − the most probable*

No.	Technology name	Steady state 2010	Type of strategic development tracks	Years			
				2015	2020	2025	2030
1.	The laser cladding / remelting of TiC titanium carbides in the surface of Mg-Al-Zn casting magnesium alloys	Strategy of an oak in spring A (9.8, 8.4)	(O)	(9.8, 6.5)	(9.9, 7.0)	(9.9, 8.0)	(9.9, 9.0)
			(P)	(9.8, 6.0)	(9.8, 2.0)	(6.0, 2.0)	(3.0, 1.8)
			(MP)	(9.7, 6.0)	(9.8, 7.0)	(9.8, 4.5)	(9.9, 4.8)
2.	The laser cladding / remelting of WC tungsten carbides in the surface of Mg-Al-Zn casting magnesium alloys	Strategy of an oak in spring B (9.2, 8.3)	(O)	(9.2, 5.6)	(9.3, 6.2)	(9.4, 7.0)	(9.4, 8.0)
			(P)	(9.2, 5.3)	(9.2, 1.6)	(5.7, 1.6)	(3.0, 1.4)
			(MP)	(9.2, 5.6)	(9.2, 6.0)	(9.3, 3.9)	(9.3, 4.2)
3.	The laser cladding / remelting of VC vanadium carbides in the surface of Mg-Al-Zn casting magnesium alloys	Strategy of an oak in spring C (9.7, 8.4)	(O)	(9.7, 6.2)	(9.8, 6.5)	(9.8, 7.5)	(9.8, 8.5)
			(P)	(9.7, 5.7)	(9.7, 1.8)	(5.9, 1.8)	(3.0, 1.5)
			(MP)	(9.6, 5.7)	(9.7, 6.5)	(9.7, 4.0)	(9.8, 4.3)
4.	The laser cladding / remelting of SiC silicon carbides in the surface of Mg-Al-Zn casting magnesium alloys	Strategy of an oak in spring D (8.8, 8.4)	(O)	(8.8, 5.6)	(8.8, 6.0)	(8.9, 7.0)	(9.0, 8.2)
			(P)	(8.8, 5.7)	(8.7, 1.7)	(5.9, 1.7)	(3.0, 1.4)
			(MP)	(8.8, 5.6)	(8.8, 5.4)	(8.8, 4.0)	(8.9, 4.3)
5.	The laser cladding / remelting of Al_2O_3 aluminium oxide in the surface of Mg-Al-Zn casting magnesium alloys	Strategy of an oak in spring E (9.2, 8.2)	(O)	(9.2, 5.6)	(9.4, 6.0)	(9.4, 7.1)	(9.4, 8.1)
			(P)	(9.2, 5.2)	(9.2, 1.5)	(5.6, 1.5)	(3.0, 1.4)
			(MP)	(9.2, 5.6)	(9.3, 6.0)	(9.3, 4.0)	(9.3, 4.1)

providing its return to friendly area of sunny spring, which combined with continuing high attractiveness and potential of technology will ensure a spectacular success. A pessimistic version expressed by a third pointed strategic development track of a group of technology assumes that the crisis in the world will become stronger due to an unfavourably growing political and economic situation, what will cause the appearance of an increasing number of problems in the environment (2015), and fewer opportunities what will cause the necessity to function in unfavourable conditions of cold winter in 2020. The economic situation will be unfriendly, resulting in the decrease of interest of the potential users in the analysed group of technology. Using a huge potential being an objective high value of technologies in 2025 the analysed group of technology being a rooted dwarf will overcome difficulties and become weaker systematically, so that in 2030, it will move to the area of a trembling aspen in the winter, for which it is recommended to disappear from the market. The numerical values resulting from all research carried out for five analysed groups of technology are presented in Table 6. Due to the relatively small differences between the analysed groups of technology at the macro scale, the path marked out for them adopt a similar direction of strategic development, showing minor discrepancies.

4. Research results of the impact of technological conditions on properties of examined alloys

The outcarried experimental works bowling down to the laser cladding of carbide and oxide powders in the surface of MCMgAl12Zn1, MCMgAl9Zn1, MCMgAl6Zn1, MCMgAl3Zn1 casting magnesium alloys the influence of parameters process like power laser and using powders on shape and surface topography were shown. A view of the casting magnesium alloys face of weld after laser treatment with carbides and aluminium oxide were shown in Figures 8-11. The front view after laser treatment with TiC and WC powders was regular and flat surface (Figs. 8, 9). In case, when VC was used, the front view was characterised by flat surface, however the surface layer was discontinuity (Fig. 10). The magnesium alloys after laser treatment with SiC particles were characterised by convexity of remelting zone over base surface. Surface layer of casting magnesium alloys after laser treatment with Al_2O_3 was characterised by small hollow in central area of bead face for laser power 2.0 kW (Fig. 11). Investigations reveal, that laser power increase by constant beam scanning rate had an influence on the size of the area, where occurred structural changes of the surface layer of the Mg-Al-Zn alloys.

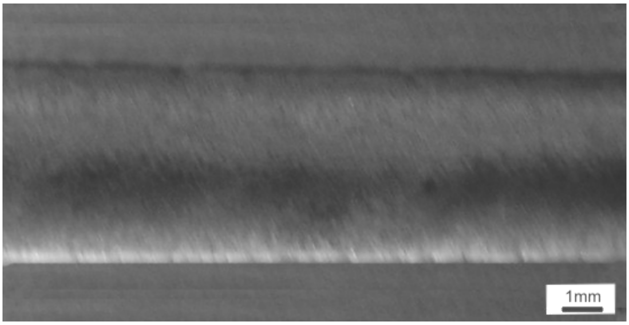

Figure 8. *View of the MCMgAl12Zn1 casting magnesium alloy face of weld after laser treatment with TiC, scan rate: 0.75 m/min, laser power: 1.2 kW*

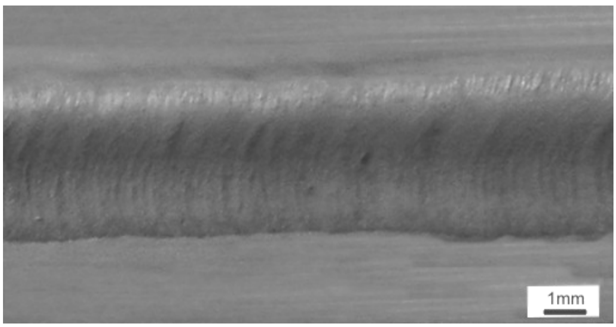

Figure 9. *View of the MCMgAl3Zn1 casting magnesium alloy face of weld after laser treatment with WC, scan rate: 0.75 m/min, laser power: 1.2 kW*

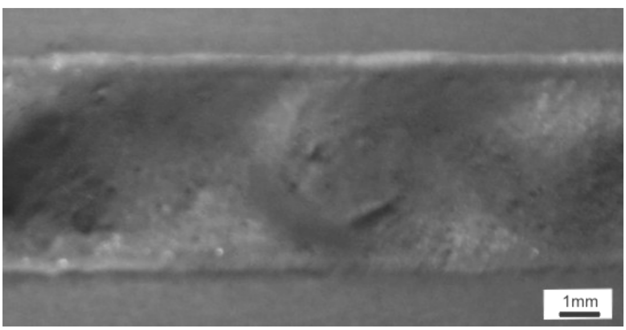

Figure 10. *View of the MCMgAl12Zn1 casting magnesium alloy face of weld after laser treatment with VC, scan rate: 0.75 m/min, laser power: 2.0 kW*

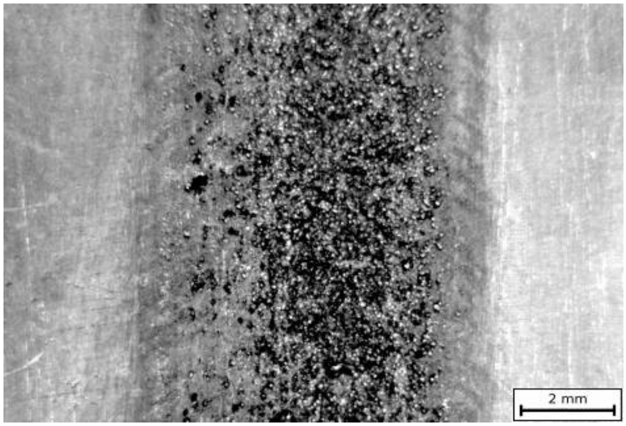

Figure 11. *View of the MCMgAl12Zn1 casting magnesium alloy face of weld after laser treatment with Al_2O_3, scan rate: 0.5 m/min, laser power: 2.0 kW*

On the basis of roughness measurements of the surface of casting magnesium alloys after laser treatment with the titanium, tungsten, vanadium, silicon and aluminium oxide (Fig. 11) it was stated that apart from the applied ceramic powder, the roughness of the surface layers abstained by laser remelting of the Mg-Al-Zn alloys with a power in the range of 1.2-2.0 kW increases and reach a value in the range of R_a = 6.4-42.5 µm.

For each type of substrate (independent of the aluminium content) the highest roughness had the samples after laser treatment by a scanning rate of 0.5 m/min with laser power of 2.0 kW. By a stable scanning rate and a not changed powder feeding, together with an increase of the laser power the surface roughness decreased. Among the investigated Mg-Al-Zn casting magnesium alloys the lowest roughness respectively 4.0 and 5.6 µm have the MCMgAl9Zn1 and MCMgAl12Zn1 materials after treatment with VC powder, by applied laser power of 2.0 kW (Fig. 12).

Maximal measured surface roughness of R_a = 42.5 µm occurs in case of the surface layer of the MCMgAl9Zn1 alloy after laser treatment with SiC powder with laser power of 1.2 kW (Fig. 12). The investigated material after treatment with titanium carbide powder are characterised by roughness in the range of 6.4-13.9 µm (Fig. 12). In case of vanadium carbide powder it was found out that the highest roughness value, by a stable scanning rate of 0.75 m/min, for each type of alloy, had the samples after treatment with 1.2 kW laser power, whereas the highest roughness value had the MCMgAl3Zn1 alloy by applied laser power of 1.2 kW (25.43 µm) (Fig. 12). An increase of the irregularity of the surface after laser treatment was related to the fluctuation of the alloyed material caused by changes of the remelting material tensions as well the laser beam energy absorbed by the alloyed material.

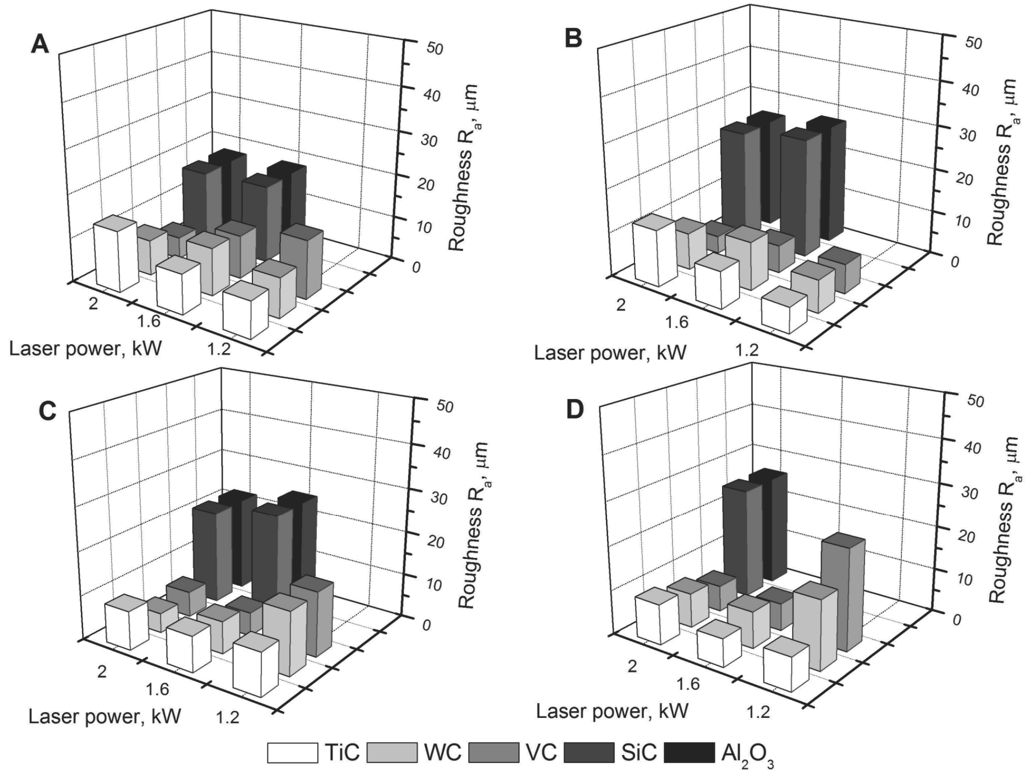

Figure 12. *Laser power and Al mass concentration influence on roughness of alloyed surface layer: A – MCMgAl3Zn1; B – MCMgAl6Zn1; C – MCMgAl9Zn1; D – MCMgAl12Zn1*

In Figures 13 and 14 the zone placement on the cross section of the remelting laser face of the Mg-Al-Zn casting magnesium alloys is presented. On the basis of the performed metallographic investigations it was found out that in each of the surface layer after surface laser treatment of the MCMgAl12Zn1 and MCMgAl9Zn1 casting magnesium alloys a remelting zone (RZ) as well a heat affected zone (HAZ) occurred. These zones, depending on the laser power as well the ceramic powder used had different thickness and shape. In case of the TiC, WC and VC powder for the MCMgAl6Zn1 alloy a very small HAZ, which increased together with the applied laser power was found. In case of alloying of the powder into the MCMgAl3Zn1 substrate only the remelting zone as well the boundary between the remelting zone and the material substrate were present. On the basis of the performed research it was possible to state that the change of the laser power by a constant alloying rate clearly influences the thickness of both zones in the surface layer. The applied laser power influenced also the shape and the convexity of the remelting zone (Figs. 13, 14), which reached over the surface of the untreated material.

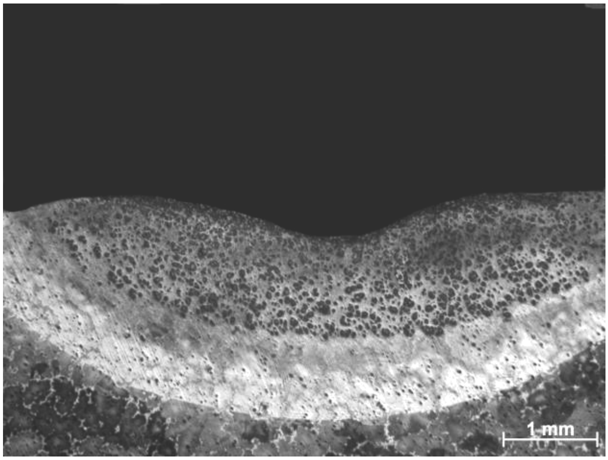

Figure 13. *Surface layer of MCMgAl12Zn1 alloy after laser treatment with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

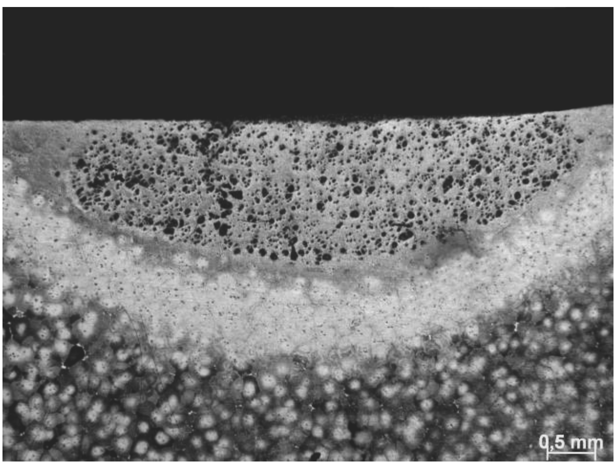

Figure 14. *Surface layer of MCMgAl9Zn1 alloy after laser treatment with WC particles, scan rate: 0.75 m/min, laser power: 2.0 kW*

Detailed results of measuring the thickness of the melted zone and heat affected zone on the pictures taken with light microscopy and confirmed by tests in a scanning electron microscope are shown in Figure 15. On the basis of tests, it was found out that the thickness of the analysed coatings, assessed by computer image analysis, was located in a wide range of measurement and was a function of four variables, namely: laser beam power, alloying speed, type of alloying material and the substrate. Proportional influence of laser power to the thickness of each zone-melted zone and heat affected zone (surface layer) was observed. The greatest thickness of the surface layer was observed after alloying of SiC powder to melt MCMgAl12Zn1 (Fig. 15), alloyed with laser power 2.0 kW, respectively 3590 μm. For the other used powders the largest values obtained for the alloy surface layer MCMgAl9Zn1 and MCMgAl12Zn1 (Fig. 15) were in the range 2340-2470 μm. Casting magnesium alloys with

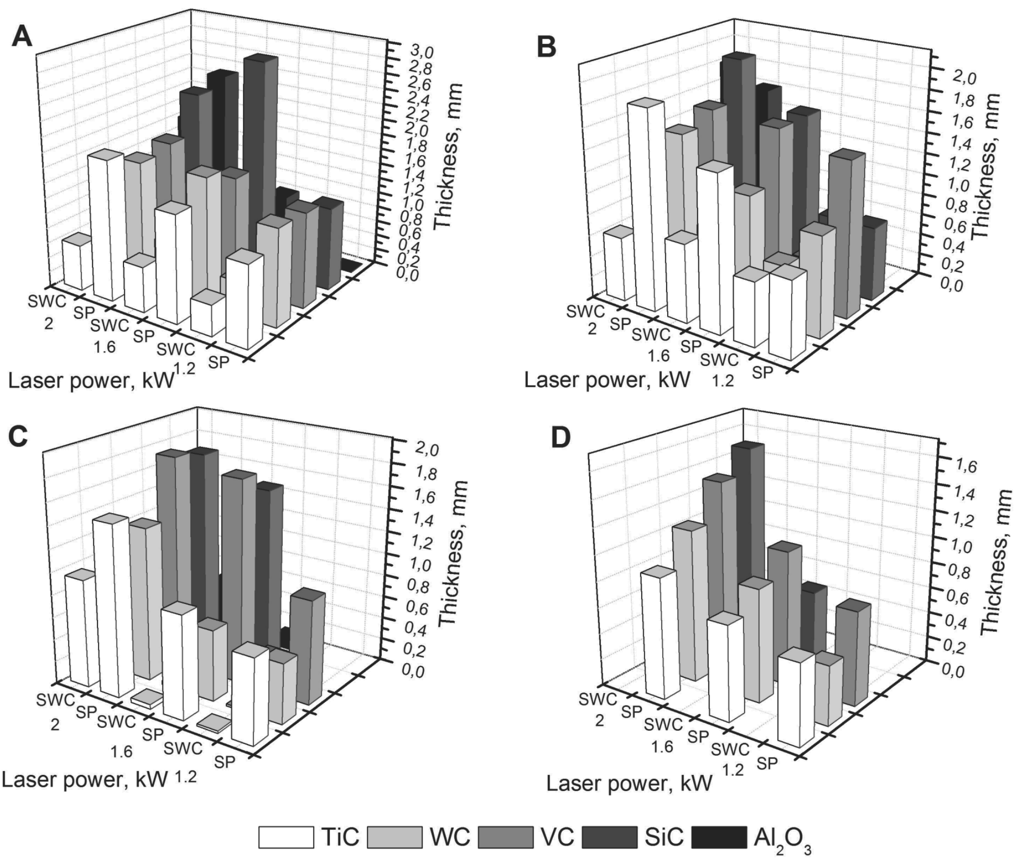

Figure 15. *Effect of laser power and the substrate material on the thickness of the SP melted zone, SWC heat affected zone and the WW surface layer of casting alloys after laser remelting: A – MCMgAl3Zn1, B – MCMgAl6Zn, C – MCMgAl9Zn1, D – MCMgAl12Zn1*

aluminium concentrations of 3% (MCMgAl3Zn1) were characterised by the smallest thickness of the surface layer of laser-melted samples. The zone of melted samples from MCMgAl3Zn1, alloyed with carbides of tungsten, titanium, vanadium, for the laser power of 1.2 kW (Fig. 14) was in the range 450-720 µm.

It was noticed that the width of the formed surface layer changes together with the laser power, speed remelting and depending on the type of substrate, of which the most important was the laser power, mainly because the increase of absorbed energy took place together with power increase, while decreases with the increase of remelting speed. The greatest width of penetration for different powders was achieved for alloys MCMgAl12Zn1 and MCMgAl9Zn1, the lowest ones for the alloy MCMgAl3Zn1. The greatest width of the remelting was observed for the alloy after remelting of MCMgAl12Zn NbC powder with a laser power of 2.0 kW – 8320 µm (which is now the largest one), while the lowest one for the MCMgAl3Zn1 alloy after remelting WC powder with a laser power of 1.2 kW – 3540 µm (Fig. 16).

Results of the metallographic investigations showed that the structure of the solidified material after laser treatment was characterised by the occurrence of areas with different morphology connected to the crystallisation of the magnesium alloys (Figs. 17-22). As a result of the laser treatment a structure which was free of defects and with a clear grain refinement was achieved. The structure of the laser modified layer contains mainly dispersive particles of the applied TiC, WC, VC, SiC carbide or Al_2O_3 oxide powder placed in the matrix of the Mg-Al-Zn alloy. Morphology of the treated area after laser treatment was mainly compound of dendrites with plate shaped $Mg_{17}Al_{12}$ eutectic and Mg present in the interdendritic space, where the main growing axes were directed according to the heat transport directions. This can be interpreted with an occurrence of a non-normal eutectic with a small amount of α-Mg in the eutectic solution (Figs. 17-22). Moreover the composite structure of the area after laser treatment results from the hypoeutectic alloy change to a hyper-eutectic one, depending from the alloyed elements distribution and the change of the process condition parameters of the laser treated surface.

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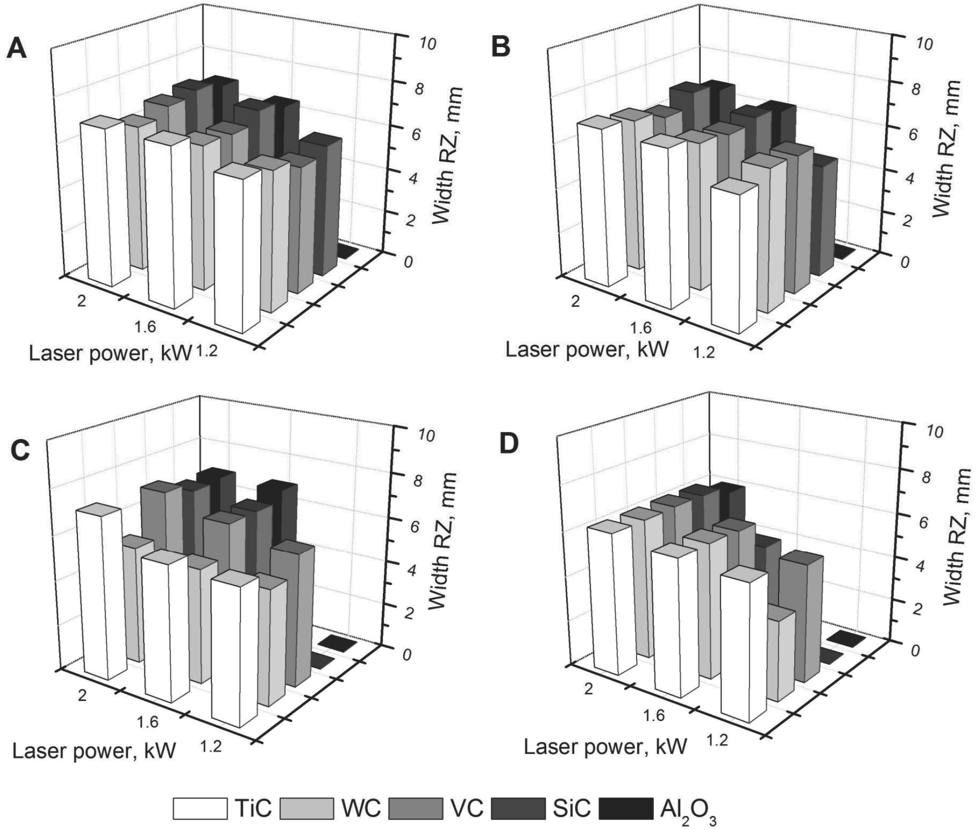


Figure 16. *Dependence of the remelting width from laser power aluminium mass concentration casting magnesium alloys after laser cladding: A – MCMgAl3Zn1, B – MCMgAl6Zn, C – MCMgAl9Zn1, D – MCMgAl12Zn1*

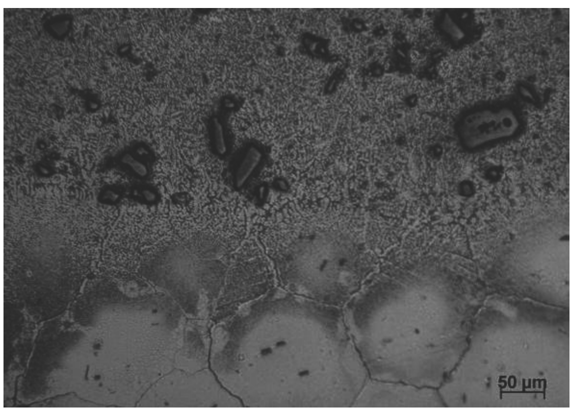


Figure 17. *Remelting path edge of the MCMgAl6Zn1 alloy surface layer after laser treatment with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

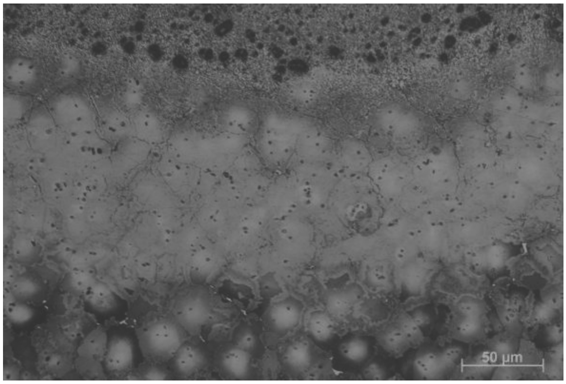


Figure 18. *Structure of the interface between the laser-melted zone, heat affected zone and the substrate of the MCMgAl12Zn1 alloy after laser treatment with WC particles, scan rate: 0.75 m/min, laser power: 2.0 kW*

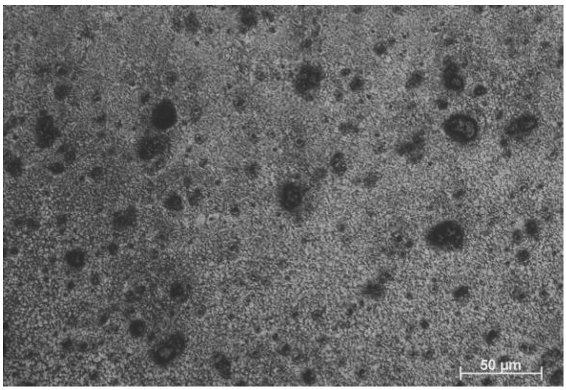


Figure 19. *Central zone of the MCMgAl12Zn1 alloy surface layer after laser treatment with WC particles, scan rate: 0.75 m/min, laser power: 2.0 kW*

Figure 20. *Remelting path edge of the MCMgAl9Zn1 alloy surface layer after laser treatment with WC particles, scan rate: 0.75 m/min, laser power: 2.0 kW*

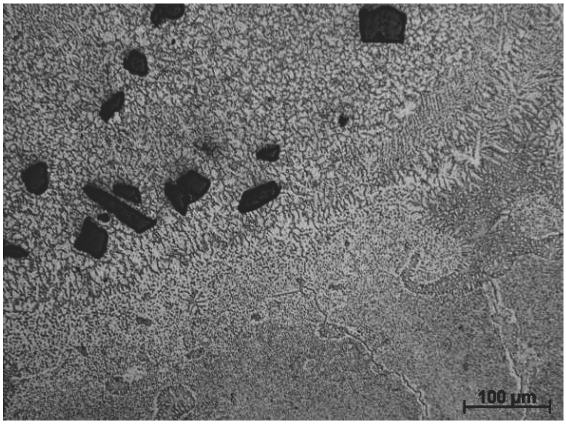


Figure 21. *Border zone of the MCMgAl12Zn1 alloy surface layer after laser treatment with SiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

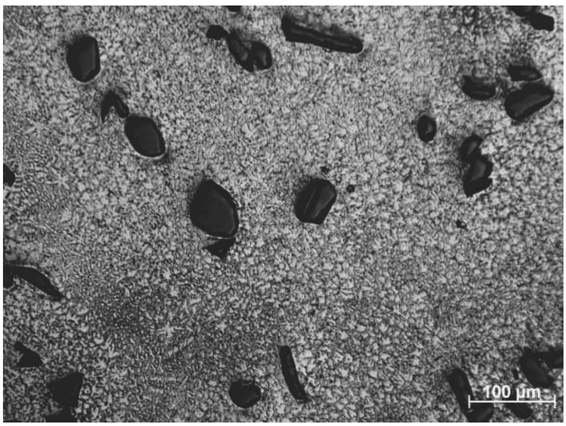


Figure 22. *Central zone of the MCMgAl6Zn1 alloy surface layer after laser treatment with SiC particles, scan rate: 0.75 m/min, laser power: 2.0 kW*

Investigations carried out on the scanning electron microscope confirmed the occurrence of the zones in the surface layer of the investigated casting magnesium alloys (Figs. 23-28). In the remelted zone a dendritic structure built according to the heat transport directions and the alloyed powder particles of the carbides or aluminium oxide were present. The morphology after surface laser treatment, including the amount and distribution of the carbide particles, depended on the applied laser parameters. On the basis of the metallographic investigations of the uniform distribution of MCMgAl3Zn1, MCMgAl6Zn1, MCMgAl9Zn1, MCMgAl12Zn1 alloys – on the whole remelting zone – of the used carbide particles TiC, WC and Al_2O_3

(Figs. 22-27) was found. In case of cladding of the SiC particles with laser power of 1.2 kW and same cases 1.6 kW, the carbides were mainly located on the top of the surface layer (Fig. 26). For power of 2.0 and 1.6 kW in samples of the MCMgAl12Zn1 and MCMgAl9Zn1 material, caused by a strong movement of the liquid metal in the remelting area, the SiC particles were distributed over the whole area of the remelting zone. After treatment with vanadium carbide in the surface of the casting magnesium alloys there only a sporadical occurrence of the carbide in the remelting area was observed (Fig. 28).

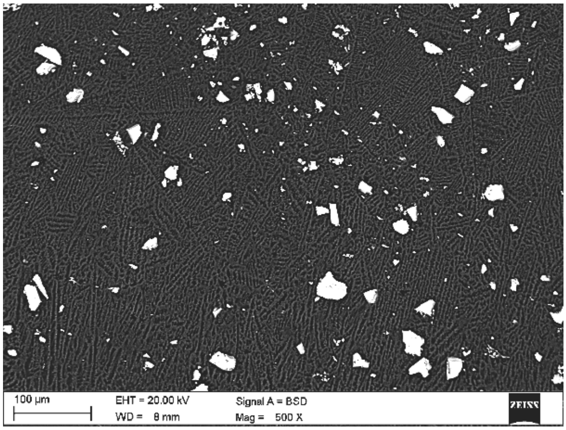


Figure 23. *Scanning electron microscope micrograph of laser modified surface of MCMgAl3Zn1 alloy with TiC particles of the central modified zone, scan rate: 0.75 m/min, laser power: 1.2 kW*

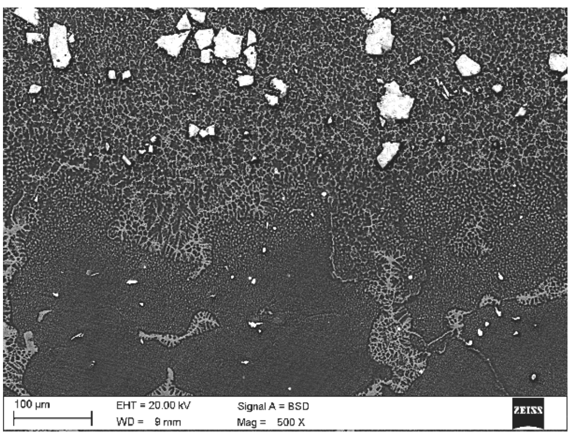


Figure 24. *Scanning electron microscope micrograph of laser modified surface of the MCMgAl9Zn1 alloy with TiC particles at the interface between the modified zone and the substrate, scan rate: 1 m/min, laser power: 1.6 kW*

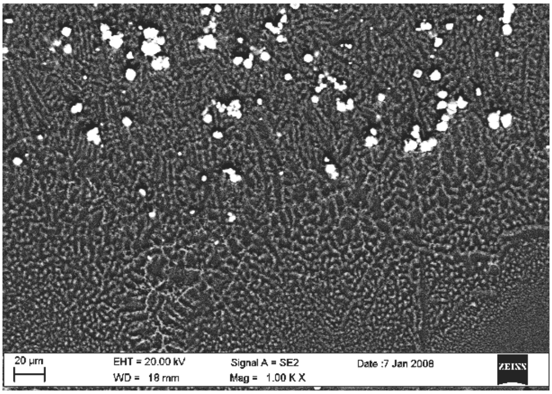


Figure 25. *Scanning electron microscope micrograph of laser modified surface of the MCMgAl6Zn1 alloy with WC particles at the interface between the modified zone and the substrate, scan rate: 0.5 m/min, laser power: 1.6 kW*

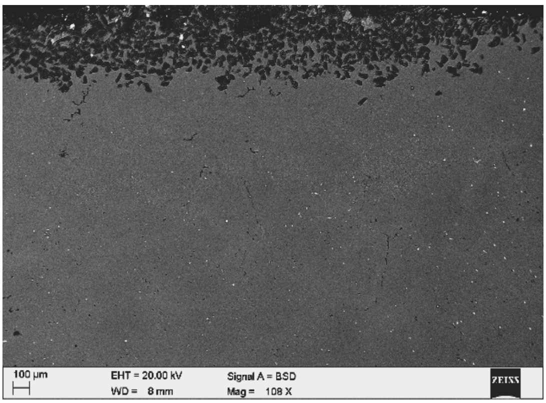


Figure 26. *Scanning electron microscope micrograph of laser modified surface layer of the MCMgAl6Zn1 alloy with SiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

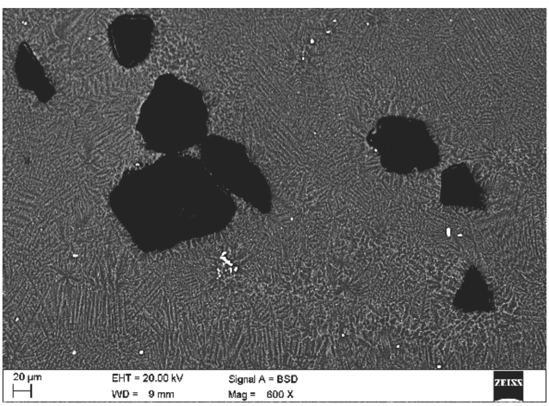


Figure 27. *Scanning electron microscope micrograph of laser modified surface of MCMgAl12Zn1 alloy with Al_2O_3 particles of the central modified zone, scan rate: 0.5 m/min, laser power: 1.6 kW*

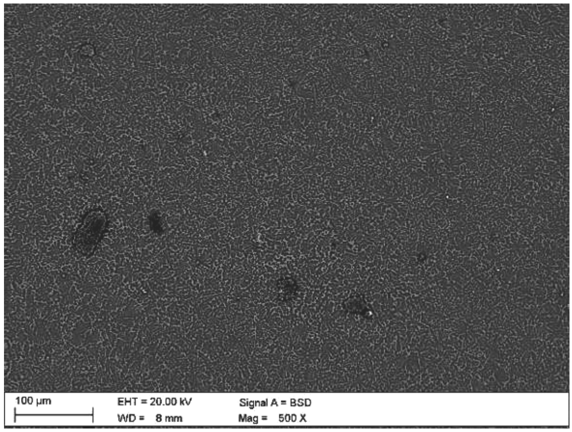


Figure 28. *Scanning electron microscope micrograph of laser modified surface of MCMgAl12Zn1 alloy with VC particles of the central modified zone, scan rate: 0.75 m/min, laser power: 1.6 kW*

X-ray diffraction diagrams of Mg-Al-Zn casting magnesium alloys after laser treatment with WC, TiC, VC, SiC carbides and Al_2O_3 oxide confirm the occurrence of α-Mg phase, γ-$Mg_{17}Al_{12}$ phase, as well of picks coming from the powders using for cladding (Figs. 29-33).

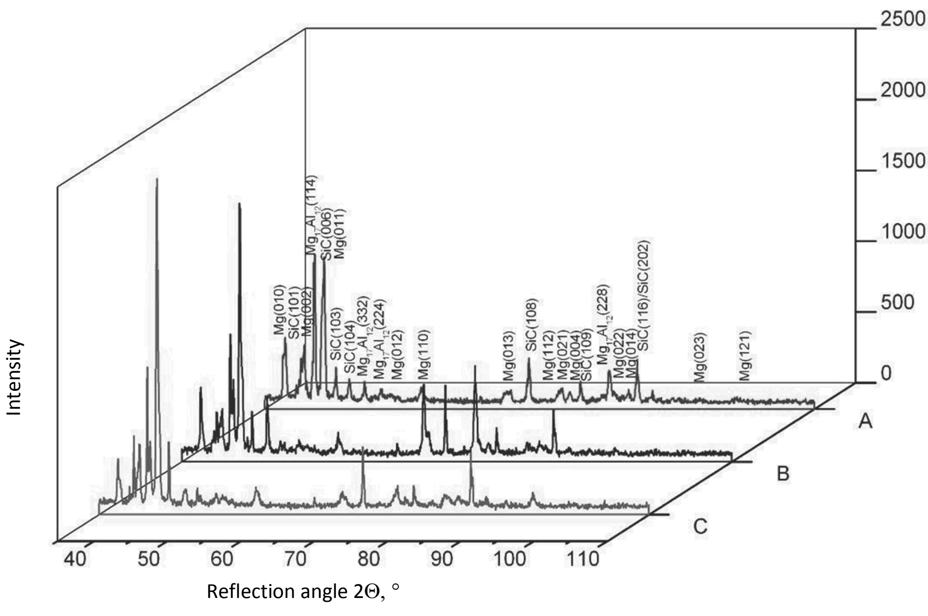


Figure 29. *X-ray diffraction pattern of MCMgAl12Zn1 casting magnesium alloys after laser treatment by silicon carbide powder, scan rate: 0.75 m/min, laser power: A – 1.2 kW, B – 1.6 kW, C – 2.0 kW*

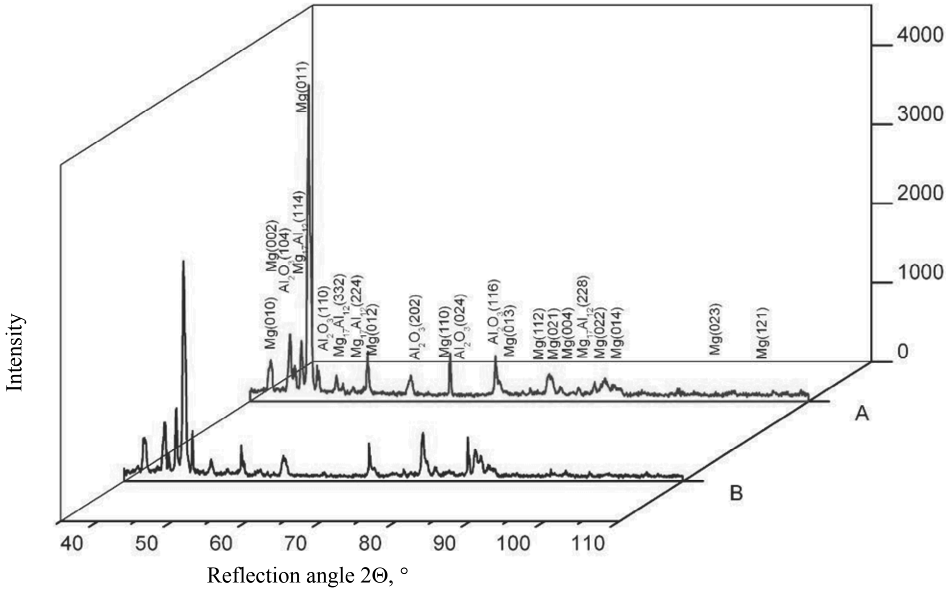


Figure 30. *X-ray diffraction pattern of MCMgAl12Zn1 casting magnesium alloys after laser treatment by aluminium oxide powder, scan rate: 0.75 m/min, laser power: A – 2.0 kW, B – 1.6 kW*

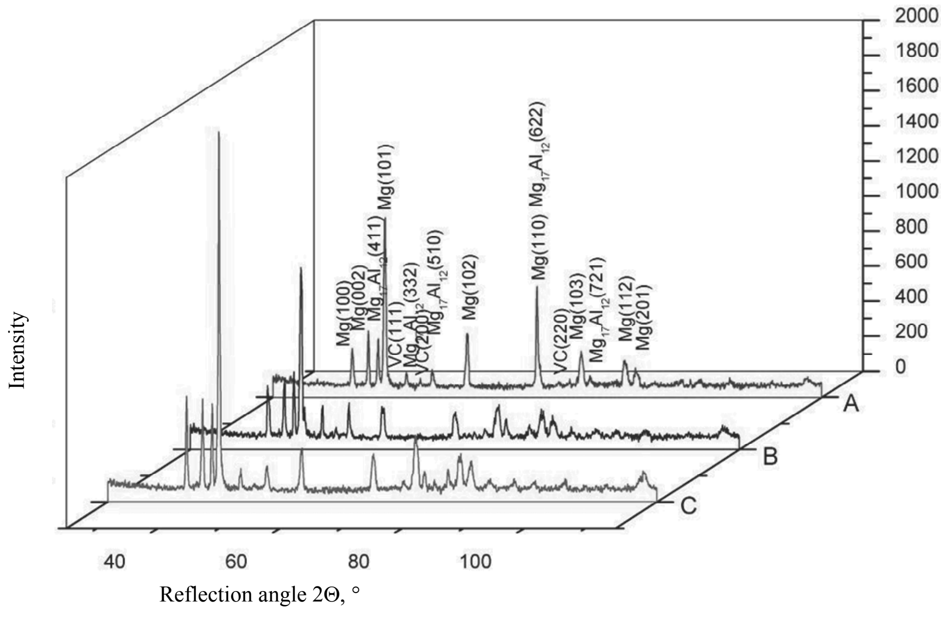


Figure 31. *X-ray diffraction pattern of MCMgAl12Zn1 casting magnesium alloys after laser treatment by vanadium carbide powder, scan rate: 0.75 m/min, laser power: A – 1.2 kW, B – 1.6 kW, C – 2.0 kW*

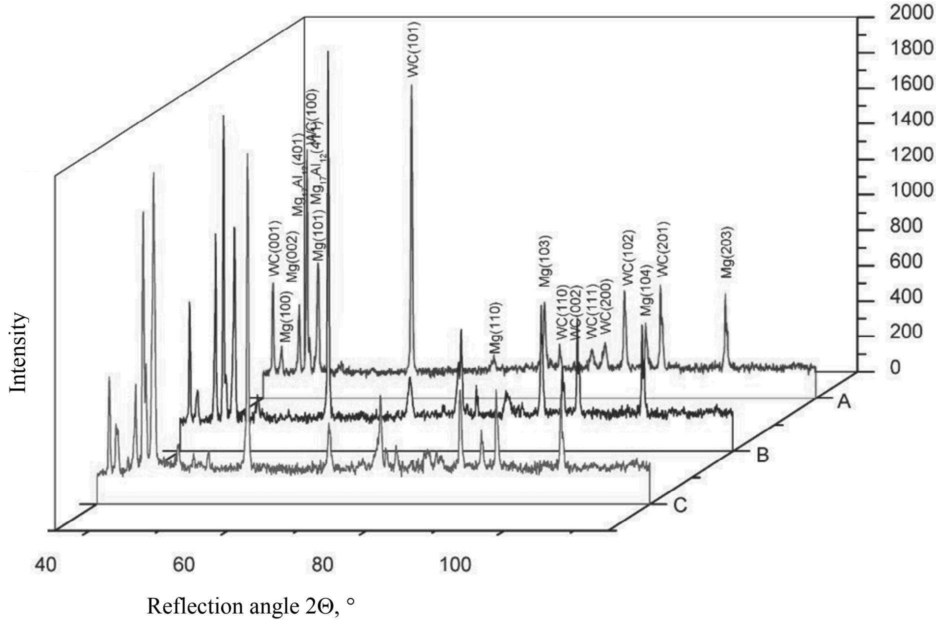


Figure 32. *X-ray diffraction pattern of MCMgAl12Zn1 casting magnesium alloys after laser treatment by tungsten carbide powder, scan rate: 0.7 m/min, laser power: A – 1.2 kW, B – 1.6 kW, C – 2.0 kW*

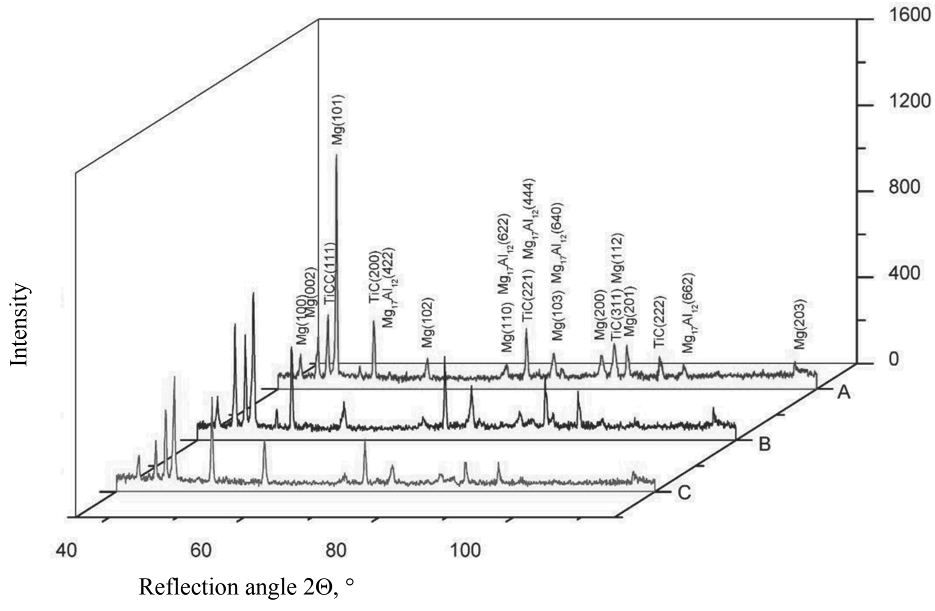


Figure 33. *X-ray diffraction pattern of MCMgAl12Zn1 casting magnesium alloys after laser treatment by titanium carbide powder, scan rate: 0.75 m/min, laser power: A – 1.2 kW, B – 1.6 kW, C – 2.0 kW*

The results of measurements of hardness of Mg-Al-Zn casting magnesium alloys after laser cladding and remelting of WC powder, TiC, VC, SiC and Al_2O_3 (Fig. 34) showed that in most cases, for MCMgAl6Zn1 and MCMgAl3Zn1 alloys laser treatment of surface layer the hardness increase, while for alloys MCMgAl12Zn1, MCMgAl9Zn1 hardness did not increase, on the contrary, at certain used parameters of treatment became slightly worse. The largest increase in hardness by 56 HRF in relation to the hardness of the usual heat treatment was obtained for the MCMgAl3Zn1 magnesium alloy after treatment TiC powder with a laser power of 1.2 kW and the alloying speed 1.0 m/min. For the highest hardness of the MCMgAl6Zn1 alloy (93.4 HRF) after laser treatment was measured at the surface layer of the TiC powder alloying with 1.2 kW laser power and alloying speed 0.75 m/min (Fig. 34). However, the greatest decrease in hardness of the surface layer was observed for alloys and MCMgAl12Zn1 MCMgAl9Zn1 after alloying SiC powder with a laser power of 1.2 kW and the alloying speed 0.75 m/min. The study indicates that the hardness increase occurred with decreasing laser power and increase of the concentration of aluminium in the alloy, as well as the descending alloying speed. In the case of fusing powdered tungsten carbide and vanadium, the largest increase in hardness occurred for alloys: MCMgAl6Zn1 and MCMgAl3Zn1. In the case of alloys: MCMgAl12Zn1 and MCMgAl9Zn1 hardness remained at a similar level as in the case of material after the conventional heat treatment.

Furthermore, on the basis of the outworked neural network model diagrams of the impact of laser power, concentration of aluminium, and also the type of powder on the hardness of the analysed casting magnesium alloys after laser treatment of the surface layer (Figs. 35-39) were made. The diagrams in most cases concern the remelting speed of 0.75 m/min, corresponding to the optimum geometry of the path of the laser. The obtained results clearly show that MCMgAl12Zn1 casting magnesium alloys alloyed by TiC and WC powders with a laser power of 2.0 kW and a speed of 0.75 m/min. are characterised by the highest hardness.

The outcarried works were included also experimental verification of the outworked technology. The study on the ready item was conducted on an experimental MCMgAl6Zn1 alloy whose surface was improved by the use of laser techniques thanks to the use of laser melting techniques of titanium carbide particles with overlapping paths of remelting using laser power 1.6 kW and alloying speed 0.75 m/min. In order to determine the quality of the outworked surface layers, made by the use of high power diode lasers, on the ready item metallographic studies were conducted, including: light microscopy (Figs. 40, 41), scanning and X-ray phase analysis and qualitative analysis of the surface distribution of alloying elements and the research of mechanical properties, including hardness, microhardness and surface roughness.

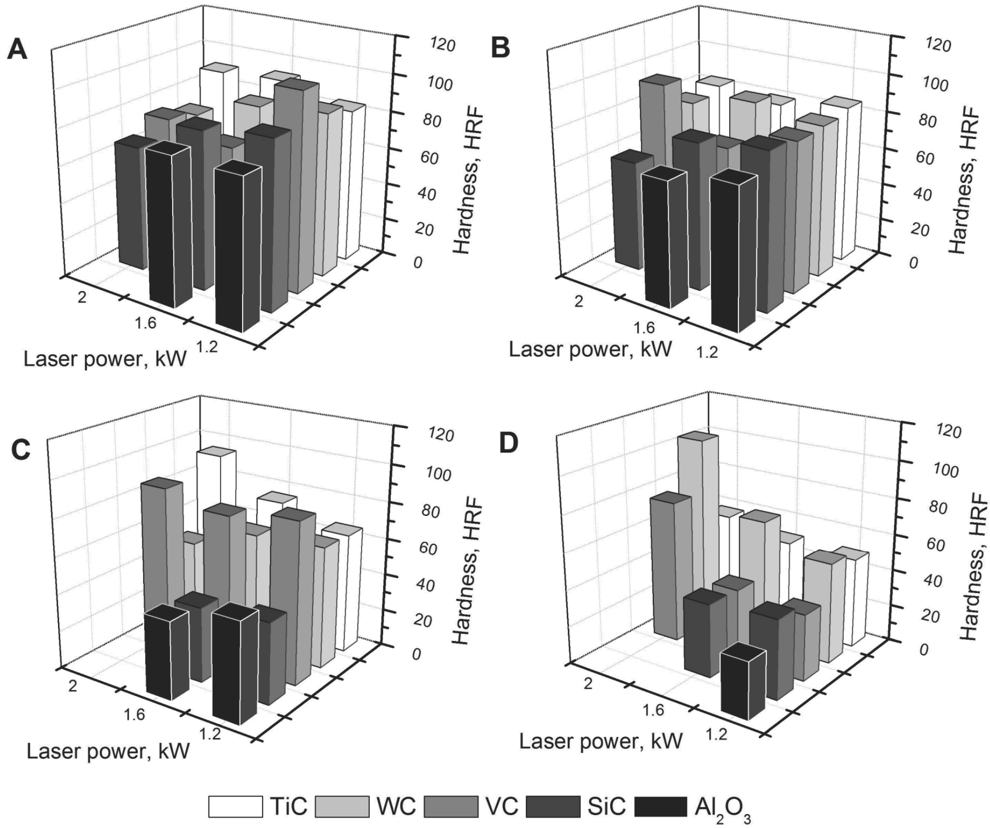


Figure 34. *Change in the average hardness of the surface layer of casting magnesium alloys after laser treatment: A – MCMgAl3Zn1, B – MCMgAl6Zn, C – MCMgAl9Zn1, D – MCMgAl12Zn1*

The results of microhardness in the cross-section of laser paths in a function of distance from the surface of samples of the casting magnesium alloy reinforced with TiC particles (Fig. 42) indicated that in the middle of the run and the overlap run the microhardness of alloyed surface increases in comparison to material substrate for about 20 HV 0.05. The measurements taken in the middle of the run (2 mm from the surface) were characterised by a mild change in hardness from about 1970 HV 0.05 at the surface to about 55 HV 0.05 into the material (Fig. 40). In case of change of microhardness measurements on the overlap run one can observe more visible differences of about 70 HV 0.05 at the surface to about 60 HV 0.05 at a distance of 1 mm from the surface of the material (Fig. 40). This is probably due the fact thicker remelting zone in the central part compared to the thickness of the zone of the overlap run. As a result of research of hardness of magnesium alloy laser treatment by titanium carbide hardness of the surface layer equal to 69.6 ± 2 HRF, increased on average by about 20% of the hardness of the material subjected only to treatment of precipitation hardening was measured. Roughness R_a of the surface layer obtained by remelting of magnesium alloy by the laser beam power of 1.6 kW was 6.02 ± 1.1 μm.

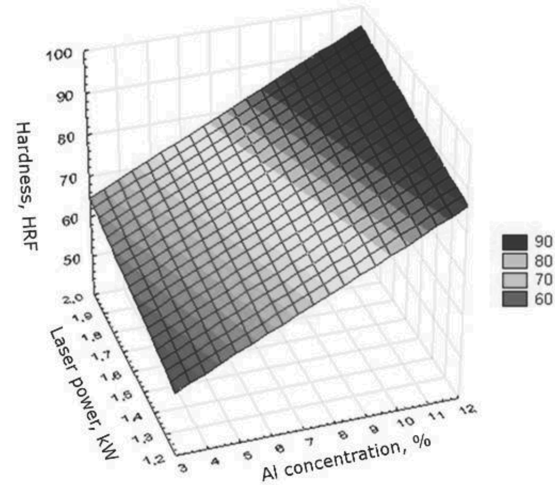


Figure 35. *Simulation of the laser power and aluminium concentration (wt. %) influence on hardness of casting magnesium alloys after laser treatment with TiC particles, scan rate 0.75 m/min*

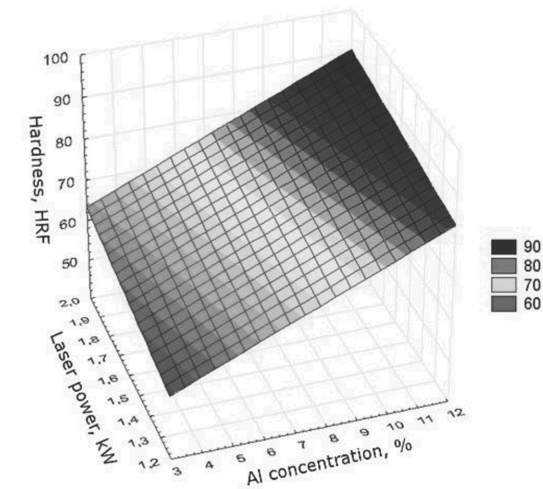


Figure 36. *Simulation of the laser power and aluminium concentration (wt. %) influence on hardness of casting magnesium alloys after laser treatment with VC particles, scan rate 0.75 m/min*

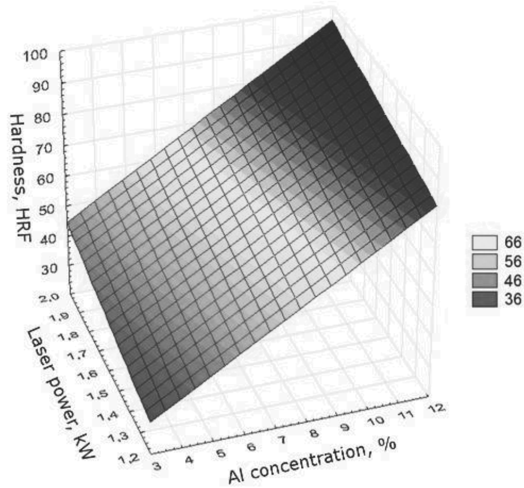


Figure 37. *Simulation of the laser power and aluminium concentration (wt. %) influence on hardness of casting magnesium alloys after laser treatment with WC particles, scan rate 0.75 m/min*

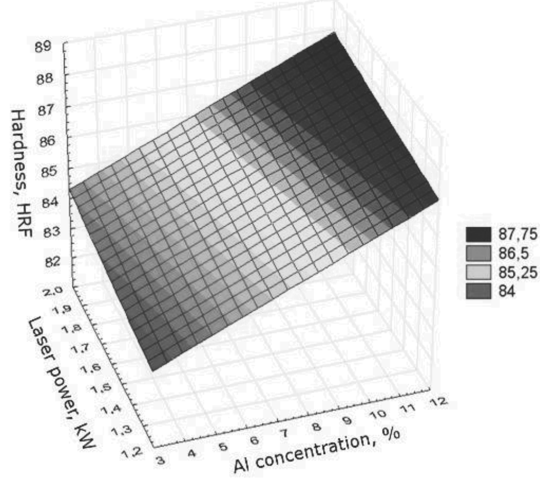


Figure 38. *Simulation of the laser power and aluminium concentration (wt. %) influence on hardness of casting magnesium alloys after laser treatment with SiC particles, scan rate 0.75 m/min*

In Figure 43 as a result of observation in the light microscope the boundary of remelting of the surface layer and the central zone was presented. Remelting zone is characterised by defect-free structure with a clear fragmentation of grains, which consists mainly of TiC carbide particles dispersed in the MCMgAl6Zn1 alloy matrix. Results of the analysis of phase composition in Figure 44 confirm the presence in the structure α-Mg and γ-$Mg_{17}Al_{12}$ phase and also in the structure of the surface layer of the used powder which alloys TiC, what was also proved by research of the surface distribution of elements (Fig. 45).

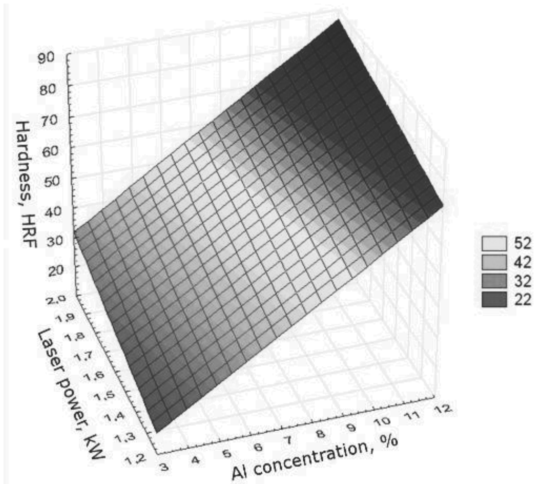


Figure 39. *Simulation of the laser power and aluminium concentration (wt. %) influence on hardness of casting magnesium alloys after laser treatment with Al_2O_3 particles, scan rate 0.5 m/min*

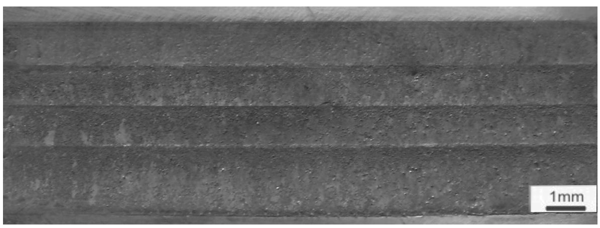


Figure 40. *View of the MCMgAl6Zn1 casting magnesium alloy face of weld after laser treatment with TiC, scan rate: 0.75 m/min, laser power: 1.6 kW*

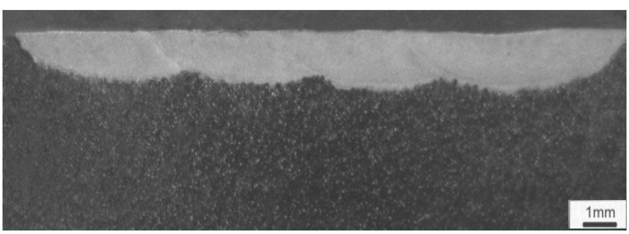


Figure 41. *Surface layer of MCMgAl6Zn1 alloy after laser treatment with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

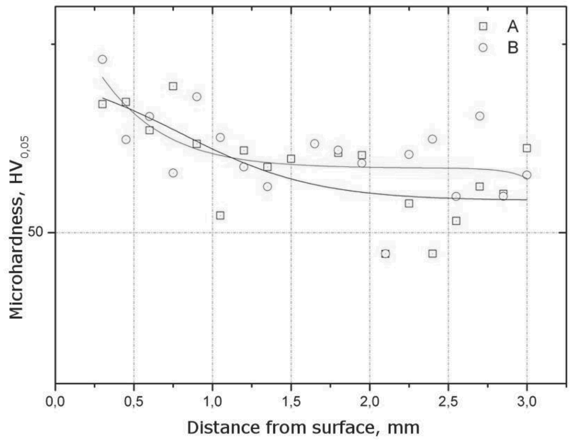
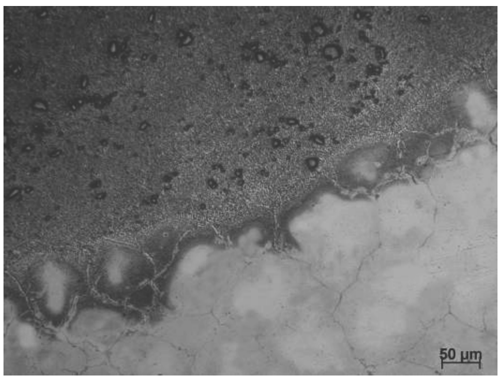


Figure 42. *Cross-section microhardness profile from the MCMgAl6Zn1 substrate with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW: A – measurements in the middle run, B – measurements in overlap run*

a)



b)

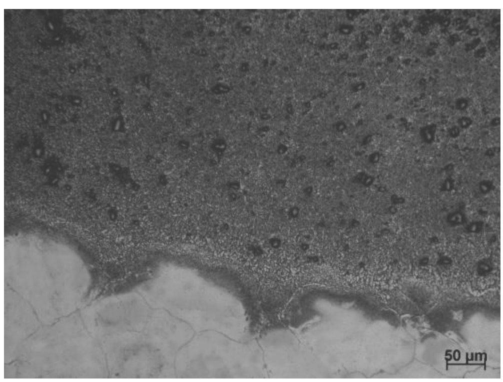


Figure 43. *Boundary between alloyed zone and heat affected zone of MCMgAl6Zn1 alloy after laser treatment with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

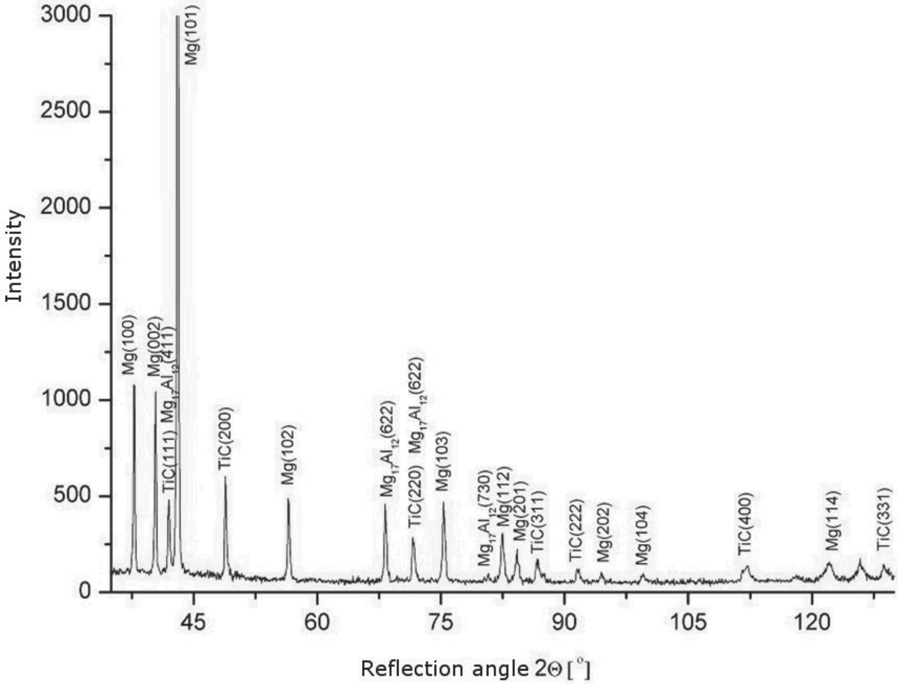


Figure 44. *X-ray diffraction pattern of the MCMgAl6Zn1 casting magnesium alloy after laser treatment with TiC particles, scan rate: 0.75 m/min, laser power: 1.6 kW*

5. Technology roadmapping results of examined laser treated alloys

On the basis of achieved results of experimental and comparative research a series of roadmaps of the analysed groups of technology were created. A representative roadmap prepared for laser cladding of TiC titanium carbide particles in the surface of Mg-Al-Zn casting magnesium alloys are shown in Table 7. The horizontal axis of a roadmap corresponds to time intervals, and the vertical axis to seven main layers answering the questions in turn: When? Why? What? How? Where? Who? How much? The upper layers were divided into more sublayers arranged hierarchically Detailed starting from the upper ones being the most general one Determining Premises, Causes and Reasons of the Realised actions through the middle ones characterizing a product and technology in lower layers of the technology precising an organisational and technical details at the end. The summary worked out on the basis of data contained in given roadmaps prepared for all the analysed groups of technology is presented in Table 8.

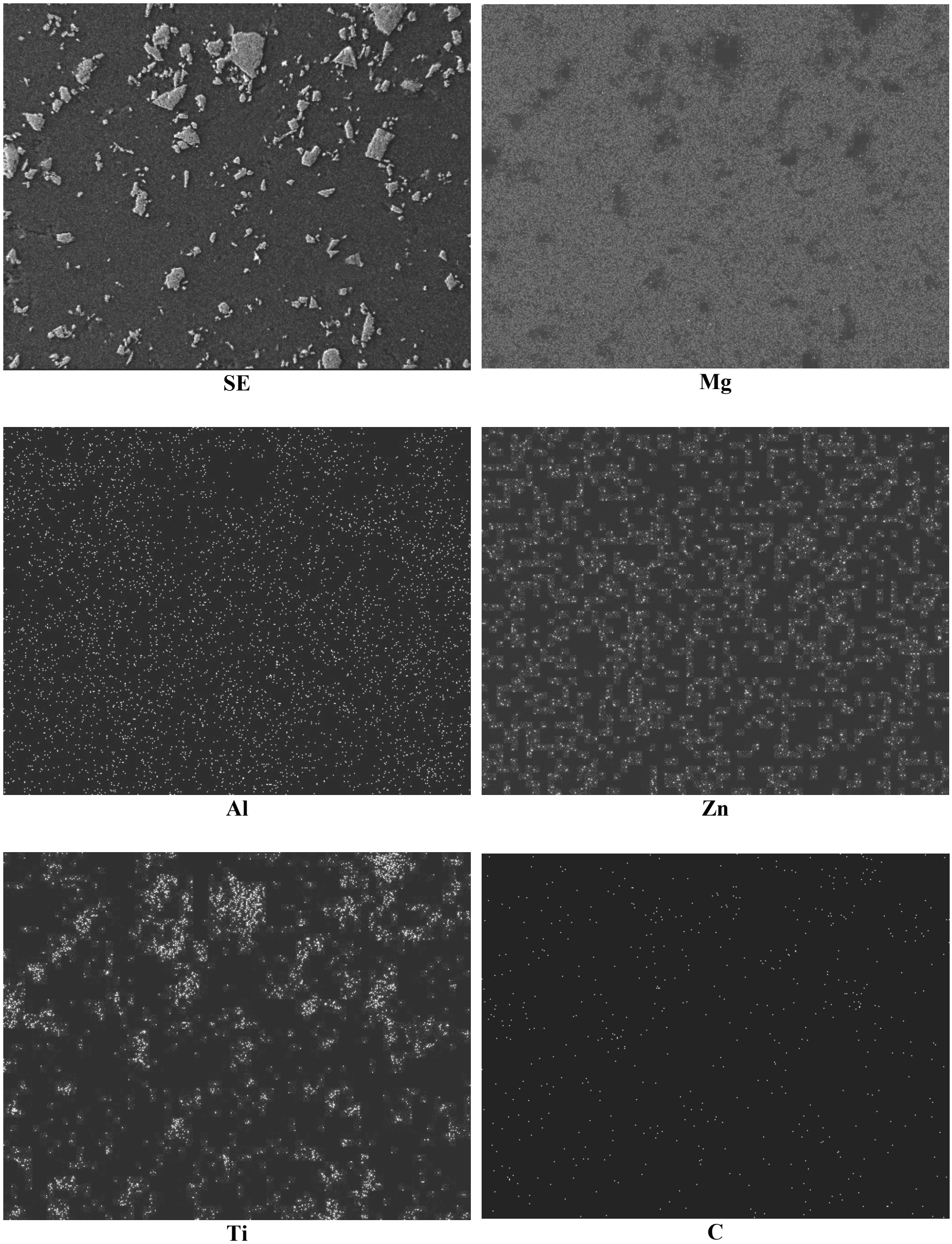


Figure 45. *X-ray mapping of the microstructure MCMgAl6Zn1 cladding layer and the distribution of Mg, Al, Zn, Ti, C, scan rate: 0.75 m/min, laser power: 1.6 kW*

Table 7. *Demonstrating technology roadmapping for laser cladding of TiC carbide particles in the substrate of Mg-Al-Zn casting magnesium alloys*

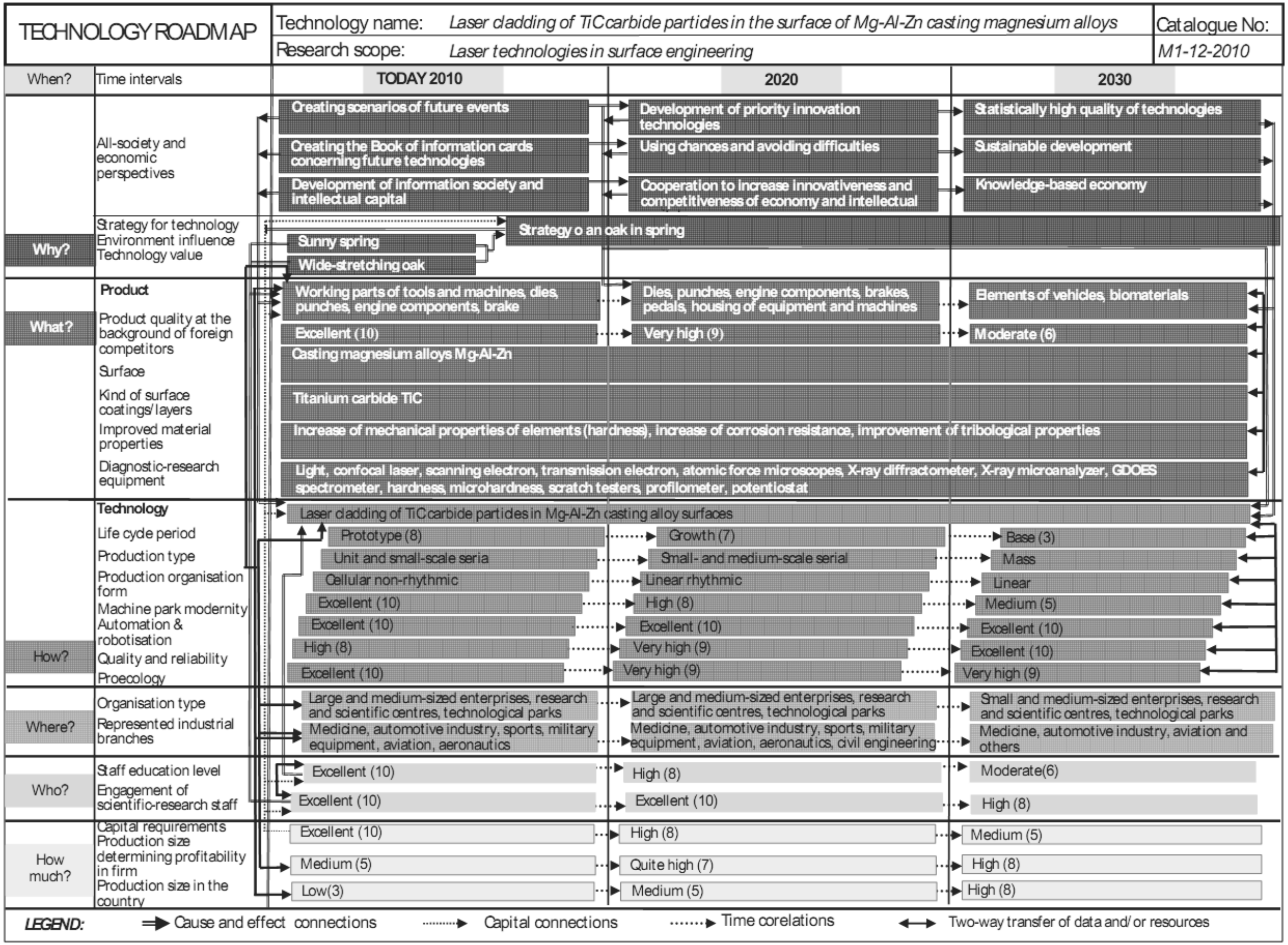


Table 8. *Selected main source data used for preparation of roadmaps for laser treatment of Mg-Al-Zn casting magnesium alloys using: (A) TiC titanium carbide, (B) WC tungsten carbide, (C) VC vanadium carbide, (D) SiC silicon carbide and (E) Al_2O_3 aluminium oxide*

No.	Analysed factor		Time interval	Analysed technology				
				A	B	C	D	E
1.	All-society and economic perspectives	Trend 1	2010	Creating scenario of future events				
			2020	Development of priority innovation technologies				
			2030	Statistically high quality of technologies				
		Trend 2	2010	Creating the Book of information cards concerning future technologies				
			2020	Using chances and avoiding difficulties				
			2030	Sustainable development				
		Trend 3	2010	Development of information society and intellectual capital				
			2020	Cooperation to increase innovativeness and competitiveness of economy and intellectual capital				
			2030	Knowledge-based economy				
2.	Strategy for technology		2010	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in spring
			2020	Strategy of an oak in summer	Strategy of an oak in summer	Strategy of an oak in summer	Strategy of an oak in autumn	Strategy of an oak in summer
			2030	Strategy of an oak in autumn	Strategy of an oak in autumn	Strategy of an oak in autumn	Strategy of an oak in autumn	Strategy of an oak in autumn
3.	Environment influence		2010	Sunny spring				
4.	Technology value		2010	Wide-stretching oak				
5.	Product		2010	Working parts of tools and machines, dies, punches, engine components, brake	Civil aviation, gearboxes, gear transmission components, high-speed elements	Elements for automotive industry, which are friction, corrosive and/ or erosive worn	dies, punches, engine components, gearboxes, gear transmission components	Elements corrosive worn, dies, punches, engine components
			2020	Dies, punches, engine components, brakes, pedals, housing of equipment and machines	MMCs composites, civil aviation, elements of motorbikes and bicycles	Elements with quasi-gradient structure, MMCs composites	MMCs composites, engine components, gearboxes, gear transmission components	MMCs composites, engine components, gearboxes, gear transmission components

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
		2030	Elements of vehicles, biomaterials	Elements of vehicles, future applications	Elements of vehicles, biomaterials, future applications	Elements of vehicles, future applications	Elements of vehicles, sport equipment, future applications
6.	Product quality at the background of foreign competitors	2010	Excellent (10)	Very high (9)	High (8)	Very high (9)	Moderate (6)
		2020	Very high (9)	Very high (9)	Very high (9)	Very high (9)	High (8)
		2030	Moderate (6)	Excellent (10)	Excellent (10)	Excellent (10)	High (8)
7.	Improved material properties	2010	Better mechanical properties of elements (hardness), better anticorrosive properties, better tribological properties				
		2020					
		2030					
8.	Diagnostic-research equipment	2010	Light, confocal laser, scanning electron, transmission electron, atomic force microscopes, X-ray diffractometer, X-ray microanalyzer, GDOES spectrometer, hardness, microhardness, scratch testers, profilometer, potentiostat				
		2020					
		2030					
9.	Life cycle period	2010	Prototype (8)	Prototype (8)	Prototype (8)	Prototype (8)	Prototype (8)
		2020	Growth (7)	Growth (7)	Growth (7)	Growth (7)	Growth (7)
		2030	Base (3)	Mature(5)	Mature(5)	Mature(5)	Base (3)
10.	Production type	2010	Unit and small-scale serial	Unit and small-scale serial	Unit and small-scale serial	Unit and small- scale serial	Unit and small-scale serial
		2020	Small- and medium-scale serial	Small- and medium-scale serial	Small- and medium-scale serial	Small- and medium-scale serial	Small- and medium-scale serial
		2030	Mass	Unit and small-scale serial	Unit and small-scale serial	Mass	Unit and small-scale serial
11.	Production organisation form	2010	Cellular non-rhythmic	Cellular	Cellular	Cellular	Cellular
		2020	Linear rhythmic	Cellular rhythmic	Cellular rhythmic	Cellular rhythmic	Cellular rhythmic
		2030	Linear	Rhythmic	Cellular rhythmic	Linear	Cellular rhythmic
12.	Machine park modernity	2010	Excellent (10)	Excellent (10)	Excellent (10)	High (8)	High (8)
		2020	High (8)	High (8)	High (8)	High (8)	High (8)
		2030	Medium (5)	Medium (5)	Medium (5)	Medium (5)	Moderate (6)
13.	Automation and robotisation	2010	Excellent (10)	High (8)	High (8)	High (8)	Quite high (7)
		2020	Excellent (10)	Excellent (10)	Excellent (10)	Very high (9)	High (8)
		2030	Excellent (10)	Excellent (10)	Excellent (10)	Excellent (10)	Moderate (6)

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
14.	Quality and reliability	2010	High (8)	Quite high (7)	High (8)	Moderate (6)	High (8)
		2020	Very high (9)	High (8)	High (8)	High (8)	High (8)
		2030	Excellent (10)	High (8)	High (8)	Very high (9)	Very high (9)
15.	Proecology	2010	Excellent (10)	High (8)	Very high (9)	High (8)	Quite high (7)
		2020	Very high (9)	High (8)	Very high (9)	Very high (9)	Quite high (7)
		2030	Very high (9)	High (8)	Very high (9)	Very high (9)	Very high (9)
16.	Organisation type	2010	Large and medium-sized enterprises, research and scientific centres, technological parks				
		2020	Large and medium-sized enterprises, research and scientific centres, technological parks				
		2030	Small and medium-sized enterprises, research and scientific centres, technological parks				
17.	Represented industrial branches	2010	Medicine, automotive industry, sports, military equipment, aviation, aeronautics	Medicine, automotive industry, sports, military equipment, aviation, aeronautics, civil engineering	Medicine, automotive industry, military equipment, aviation, aeronautics	Automotive industry, military equipment, aviation, aeronautics	Automotive industry, aviation, civil engineering, sport
		2020	Medicine, automotive industry, sports, military equipment, aviation, aeronautics, civil engineering	Medicine, automotive industry, sports, military equipment, aviation, aeronautics	Medicine, automotive industry, military equipment, aviation	Automotive industry, sport, military equipment, aviation, aeronautics	Automotive industry, sport, aviation
		2030	Medicine, automotive industry, aviation and others	Medicine, automotive industry, aviation and others	Medicine, automotive industry and others	Automotive industry, sport, aviation and others	Automotive industry, sport, aviation and others
18.	Staff education level	2010	Excellent (10)	Very high (9)	Very high (9)	Very high (9)	Very high (9)
		2020	High (8)	Medium (5)	High (8)	Medium (5)	Medium (5)
		2030	Moderate (6)	Medium (5)	Moderate (6)	Medium (5)	Medium (5)

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
19.	Engagement of scientific-research staff	2010	Excellent (10)	Quite high (7)	Very high (9)	Excellent (10)	Quite high (7)
		2020	Excellent (10)	Quite high (7)	Excellent (10)	Very high (9)	Quite high (7)
		2030	High (8)	Quite high (7)	Moderate (6)	Very high (9)	Quite high (7)
20.	Capital requirements	2010	Excellent (10)	Very high (9)	Very high (9)	Very high (9)	Quite high (7)
		2020	High (8)	Very high (9)	High (8)	Quite high (7)	Quite high (7)
		2030	Medium (5)	Quite low (4)	Moderate (6)	Medium (5)	Low (3)
21.	Production size determining profitability in firm	2010	Medium (5)	Moderate (6)	Medium (5)	Moderate (6)	Medium (5)
		2020	Quite high (7)	Quite high (7)	Quite high (7)	Quite high (7)	Quite high (7)
		2030	High (8)	High (8)	Very high (9)	High (8)	High (8)
22.	Production size in the country	2010	Low (3)	Very low (2)	Very low (2)	Low (3)	Very low (2)
		2020	Medium (5)	Medium (5)	Quite low (4)	Moderate (6)	Medium (5)
		2030	High (8)	Quite high (7)	Moderate (6)	Very high (9)	Quite high (7)

6. Conclusions

Materials science and foresight research described in this chapter are a part of own wider actions aiming to isolate, investigate, characterise and define strategic perspectives of developmental priority innovative technologies of surface engineering of engineering materials in the process of technological e-foresight. In this chapter, the scope of research and analysis was limited only to laser surface treatment of selected Mg-Al-Zn casting magnesium alloys. The chapter presents the results of experimental and comparative interdisciplinary research located mainly in the area of materials surface engineering and technological foresight, and to a lesser extent, artificial intelligence, statistics, information technology, machine building and operation and strategic and operational management. The main goal of outcarried research was to determine the value of laser treatment technology of casting magnesium alloys in the background environment with recommended procedure strategies, the strategic development tracks including the influence of this treatment on quality, microstructure and properties of surface layers obtained by cladding and remelting casting magnesium alloys. The final result closing the whole experimental and competitive works was the creation of a series of roadmaps of the analysed groups of technologies. The outworking and the use of this tool allowed to present various factors characterising directly and indirectly examined groups of technologies taking into consideration forecasts and perspectives of their development in different time intervals of taken time horizon in a uniform transparent format. As it was shown in the discussed example, roadmaps of technology are very convenient and practical tool for comparative analysis, making easier to choose technologies in terms of selected criteria, and complemented by technological cards including precise technical details which allow to implement technology in industrial practice. A very important advantage of a roadmap of technology is its flexibility, which allows to complement and expand with additional sublayers, depending on emerging needs.

Analyzing the achieved results it is clear that it will be possible to use tested Mg-Al-Zn alloys and technologies of their treatment, and alternative surface layers to ensure the best possible properties of a "quasi-gradient" on the cross-section of products, in industrial practice, especially in aerospace and automotive industries where low weight products, increased abrasion resistance, high mechanical properties as well as repair parts of ready-made components are required. The requirement to reduce the weight of car components as a result of legislation limiting emission created renewed interest in magnesium [102]. Recent research and development studies of magnesium and magnesium alloys have focused on weight reduction, energy saving and limiting environmental impact. Together with mass decreasing the driving parameters improve, this is connected mainly to the dynamic behaviour of the vehicles. A need for reduction of the transportation vehicle mass is very important, because more and more transportation vehicles is equipped with additive accessories (like airbags, safety belts, raising and lowering system for car windows, etc.) which increases mass and have influence not only on safety, but also on usable attractiveness of these vehicles [57, 89, 93, 94, 98, 99, 101-103, 107, 108]. Volkswagen was the first to apply magnesium in the automotive industry on its Beetle model, which used 22 kg magnesium in each car of this model [102]. General Motors in their big cars (Savana & Express) use 26.3 kg of casting magnesium alloys, and in smaller cars (Safari, Astro) – 16.5 kg, Ford F – 150 – 14.5 kg, VW Passat and Audi A4 and A6 from 13.6 to 14.5 kg, Alfa Romeo – 9.3 kg. The engine weight of Audi V8 Quattro model was reduced 5 kg comparing to other Audi eight-cylinder by using magnesium components. The instrument panel for the GMH-van vehicle is made of magnesium alloy which weights 12 kg as opposed to the 18 kg in steel [98, 101, 102]. Magnesium substitution in the automotive industry has steadily increased over the last decade, in parts such as valve covers, instrument panels, steering wheels, steering wheel armatures, cylinder block heads, transmission cases, clutch housings, lower crank cases, intake manifolds, brake and gas pedals and seat bodies [93, 98, 99, 101, 102]. A steering wheel is the component that has received the greatest worldwide acceptance which made of Mg alloy. Assuming 20 kg of magnesium alloy components will be used instead of alternatives on 25% of the 40 million cars produced, one calculates that the car manufacturing industry requires 200,000 tons of magnesium alloys [102]. The strategy regarding the use of Mg in vehicles to achieve a "second Mg age" is based on the applications for cast components; here further development of Mg properties (e.g. improved creep resistance) will be helpful. Mg components in body, sheet and extrusion applications will follow in niche and premium vehicles supported by new Mg alloys with improved ductility and energy absorption. Significant research is still needed on magnesium processing, alloy development, joining, surface treatment, corrosion resistance, and mechanical properties improvement [93, 98, 99, 101, 102]. Different coating methods are used to increase the properties of surface layer of magnesium alloys (corrosion resistance, lubricity, high frictional-resistance, non-wetting properties, hardness, decorative and etc.), e.g. galvanic coating, anodisation, coating using the PVD method, laser cladding/ remelting.

The presented results of materials science research show a beneficial improvement of the mechanical properties of material. Laser cladding and remelting of all mentioned carbide and oxide powders affect the fragmentation of the structure throughout the range of laser power and the diversity of grain size in different zones of the surface layer of the examined alloys. The surface layers are divided into two zones: remelting (SP) and the heat affected ones (HAZ), whose characteristic size (layer thickness) is dependent on the applied laser power and used alloying material. The thickness of the achieved surface layer of casting magnesium alloys increases with the increase of laser power from 1.2 to 2.0 kW and ranges from tenths of a mm to about 4 mm (in the MCMgAl12Zn1 alloy alloyed with SiC). The structure of the material solidifying after laser remelting is characterised by a diverse morphology and consists of a dispersive particles of used carbide: TiC, WC, VC, SiC or aluminium oxide Al_2O_3, with the dendrites of lamellar eutectic and $Mg_{17}Al_{12}$ and Mg in intereutectic areas, whose main axes are oriented along the lines of heat abstraction and the precipitates containing Mg and Si, as well as phases of high concentrations of Mn and Al. In addition, the morphology of the composite structure of alloyed area was achieved due to the change of the alloy from hypoeutectic into hypereutectic, depending on the arrangement of alloyed elements and change of the process parameters of laser surface treatment. The applied laser processing parameters also affect the surface roughness of casting magnesium alloys after laser treatment. With invariable speed of scanning and intensity of powder feed together with the increase of laser power surface roughness is reduced. Measured surface roughness is in the range from 4.0 μm to 42.5 μm. In Mg-Al-Zn casting magnesium alloys which underwent cladding and remelting by carbides and oxides, resulting in fragmentation of the grains and the presence of hard particles of used powders, maximum hardness of about 103 HRF was obtained for the MCMgAl12Zn1 alloy alloyed with titanium carbide with a laser power of 1.2 kW and the alloying speed of 0.5 m/min. One may think that such manufactured materials may be used in mass production of items of equipment and vehicle construction in the automotive and aviation industry.

Generally, this chapter, which is a practical application of methodology of foresight and materials science research to a given technology having practical industrial meaning, indicates that it is intentional, reasonable and useful to use the methodology presented theoretically in previous works [40, 41] to analyze the value, strategic development tracks and the outworking of technology roadmaps of forming structure and surface properties of engineering materials. This methodology will therefore be used for other technologies and in relation to other engineering materials in their further own research and presented in subsequent publications. It is intentional also to use not only the evaluations made by key experts, but also to analyse the results of surveys carried out by a broad group of industry experts in which the possibilities of e-foresight, theoretically worked out in the own work will be fully applied [41].

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6 Foresight methods application for evaluating laser treatment of hot-work steels

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Abstract

Purpose: *The purpose of this chapter is to evaluate the strategic growth perspectives of laser treatment of X40CrMoV5-1 and 32CrMoV12-28 hot-work alloy tool steels using NbC, TaC, TiC, VC and WC carbide powders. The criterion assumed for dividing the technologies into groups was the powder type; thus, five groups were selected to realised research.*

Design/methodology/approach: *As a part of the foresight-materials science research, a dendrological matrix of technology value, a meteorological matrix of environment influence, and a matrix of strategies for technologies were elaborated, the strategic development tracks were determined, and materials science experiments were conducted using a scanning electron microscope, an optical microscope, a transmission electron microscope, a microhardness tester, a scratch tester, an X-ray diffractometer, an electron microprobe X-ray analyzer and a device for testing of heat fatigue and abrasive resistance. Also, technology roadmaps were prepared.*

Findings: *The research conducted demonstrated huge potential and attractiveness of the analysed technologies, compared to others, and the promising properties improvement of the tested surface layers, as a result of laser surface treatment.*

Research limitations/implications: *Research concerning laser treatment of hot-work alloy tool steels constitute a part of a larger research project aimed at identifying, researching, and characterizing the priority innovative technologies in the field of materials surface engineering.*

Practical implications *The presented results of experimental materials science research prove the significant positive impact of laser treatment on the structure and the properties of hot-work alloy tool steels, which justifies including them in the set of priority innovative technologies recommended for use in small and medium enterprises and in other business entities.*

Originality/value: *The value of this chapter lies in the fact that it determines the value of laser treatment of hot-work alloy tool steels compared to other technologies and identifies the recommended strategic development tracks and technology roadmaps for them, taking into account the impact of such treatment on hardness, abrasion resistance, and coarseness of the tested surface layers.*

Keywords: *Manufacturing and Processing; Laser Surface Treatment; Hot-work steels; Foresight; Technology Roadmapping*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, E. Jonda, K. Labisz, Foresight methods application for evaluating laser treatment of hot-work steels, Journal of Achievements in Materials and Manufacturing Engineering 43/2 (2010) 750-773.

1. Introduction

The increasing role of advanced technologies in our daily lives is one of the key trends that determine the way the contemporary world works. The Internet is more and more often used to perform various processes that require exchange of information, goods or capital, which makes these processes simpler, more efficient, and faster. Terms such as e-management, e-business, e-commerce, e-banking, e-logistics, e-services, e-administration and e-education have become common in the last few years. Another new term can be added: e-foresight [1-3]. It can be defined as the process of foresight research intended to identify the priority innovative technologies and strategic development tracks in a given research area, involving the use of the Internet. On the one hand, e-foresight focuses on supporting the work of foresight research teams. On the other hand, it gives freedom to the trade experts participating in survey research who can work in more convenient time and place, which leads to faster, more efficient, and more effective obtaining of intermediate and final research results. The e-foresight process, defined as computer aided scientific forecasting and shaping of the future is aimed to achieve the following goals: development of knowledge-based economy, statistical improvement of the technology quality applied in domestic enterprises, and continuation of sustainable development.

The conducted domestic research [4] have demonstrated that Poland still is not very successful in developing a knowledge-based economy. The following indices were analysed: the Knowledge Economy Index (KEI), the Summary Innovation Index (SII), the inventiveness index, the percentage of the GDP spent on research and development, the share of high- and medium-technology products in the industrial production, and the share of high- and medium-technology products in total exports. The carried out analyses have demonstrated the presence of a technology gap between Poland's economy and the economies of developed countries which, with regards to knowledge-based economy, are lead by Finland. It was also demonstrated that in the economy of Poland, the sectors which are considered to be the so-call high-technology sectors and which constitute a part of knowledge-based economy are underdeveloped; also, it was found that the research and development sector is underinvested as the amounts spent on it by enterprises are several times smaller than in the leading countries and as research and development works are poorly used in practice. Considering the above, it is very important to strengthen the links between the science and the business, especially with respect to the statistical majority of companies present in the market, namely the small and medium-sized enterprises. They require guidance with regards to actions that will guarantee their success in the market. One of the necessary conditions for their success is to apply innovative, prospective technologies, among which are many technologies in the field of surface engineering. Materials surface engineering, which includes surface treatment and surface coating, is one of the most dynamically growing sectors of the economy in many technologically advanced countries. For example, according to source data for the year 2008, this particular sector constituted 8-10% of the German economy. Thus, one can assume that a similar situation should occur soon in the rapidly growing economy of Poland. The broadly defined surface treatment and surface coating are implemented in nearly all production sectors of the industry, which demonstrates good future prospects and can potentially significantly contribute to Poland’s economic growth. What must also be emphasised is the importance of ecology and the need to both prevent new pollution and eliminate the consequences of the present degradation of the natural environment. Care for the natural environment goes hand in hand with the notion of sustainable development, defined as [5] a process of integrating systemic, economic, and social activities while maintaining a balance in the nature and preserving the basic natural processes, with the interest of the future generations in mind.

A particularly important role among surface treatment processes and production of gradient materials is played by laser surface treatment methods. Such methods have been described both in international and domestic literature, in reference to selected engineering materials [6-18], and in the works of the Division of Materials Processing Technologies, Management, and Computer Techniques in Materials Science of the Institute of Engineering Materials and Biomaterials of the Silesian University of Technology in Gliwice [19-36]. Laser treatment of surface layers of materials is aimed mostly to form their structure and properties, which takes place in the process of creating a chemically uniform, fine-crystalline surface layer, without changing the chemical composition of the material. Laser surface treatment contributes mostly to increasing the abrasion resistance and heat fatigue of the treated materials. It is also possible to improve the functional properties of materials by alloying the top layer of material with particles of hard phases of carbides, oxides, or nitrides. The advantages of laser surface treatment over other surface engineering methods are the short duration of the process, the flexibility and the precision of the process operations that can be performed on different types of materials, from hard-machinable, through soft, to brittle materials, with efficiency and accuracy which are often superior to those for the methods used so far. The ability to precisely adjust the process parameters, such as the speed of scanning of the surface with the laser beam, the power of the beam, the type and thickness of the alloying material, and the gas shield, makes it possible to obtain an alloy layer with the properties required for the particular application.

Hot-work alloy tool steels are still a widely used group of tool materials and are particularly interesting due to their low price and very good functional properties. The processes which are traditionally used in order to improve their characteristics are heat treatment, heat and chemical treatment, and heat and mechanical treatment, e.g. nitrogen hardening, carburizing, and boronizing. Laser treatment of the top layers of hot-work alloy tool steels appears to be an attractive alternative which allows for improving their functional properties, especially their hardness, abrasion resistance, and coarseness. Thanks to the benefits it provides, especially the high density of the laser radiation power, which allows for precise heating and controlled cooling of a small volume of the material, laser cladding and/or alloying enjoy a growing interest in many research centres world-wide [6-13].

The continuous importance of hot-work alloy tool steels to the industry and the advantages of laser surface treatment were the basis for the series of interdisciplinary foresight-materials science research aimed to determine the value, i.e. the attractiveness and the potential of laser treatment of hot-work alloy tool steel on the background of micro- and macroenvironment. The works also involved elaboration of the recommended strategies, strategic development tracks, and technology roadmaps for the analysed technologies, taking into account the impact of laser treatment on the quality, the structure, and the properties of surface layers of hot-work steels. The experimental materials science research covered the X40CrMoV5-1 and the 32CrMoV12-28 hot-work alloy tool steels which were laser remelted and/or alloyed using the NbC, TaC, TiC, WC, and VC carbide powders. The purpose of the research was to determine the impact of the alloying parameters on the refinement of the structure and the mechanical properties of the top layer, in particular on its hardness, abrasion resistance, and coarseness. The research was performed using the following diagnostics and measurement equipment: a scanning electron microscope, an optical microscope, a transmission electron microscope, a microhardness tester, a scratch tester, an X-ray diffractometer, an electron microprobe X-ray analyser and a device for testing of heat fatigue and abrasive resistance. The foresight-materials science research described herein constitute a part of broader own activities [2-3, 37-43] initiated to identify a set of priority innovative surface engineering technologies to be applied in practice in the industry, and to determine the strategic development directions in this field of science. The activities are intended to contribute to the achievement of the assumed objectives of the e-foresight [1], namely growth of knowledge-based economy, statistical improvement of the technology quality, and strengthening of the concept of sustainable development.

2. Applied research methods

The carried out interdisciplinary research using outworked methodology pertain mainly to technology foresight [44] and to surface engineering included in materials science. At certain stages of the conducted studies, also methods were used which come from artificial intelligence, statistics, IT technology, construction and exploitation of machines, as well as strategic [45], operational [46] and quality [47] management. The conducted research, according to the adopted methodology, include: selecting technology groups for experimental-comparative research, collecting expert opinions, carrying out a multi-criteria analysis and marking its results on the dendrological and meteorological matrix, determining strategies for technologies preceded by rescaling and objectivising test results using simple software, setting strategic development tracks for technologies, carrying out a series of specialist materials science experiments in experienced team [19-40] using a specialist diagnostic-measuring apparatus and the creation of technology roadmaps. According to the applied methodology of foresight-materials science research, several homogenous groups should be singled out from all analysed technologies in order to subject them to planned experimental-comparative nature research. To determine the objective values of given selected technologies or their groups a dendrological matrix of technology value is used. However, to determine the strength of positive and negative influence of the environment on a given technology a meteorological matrix of environment influence is used. The methodological construction of those both matrices refers to portfolio methods, and first of all to BCG matrix [48]. A ten-point universal scale of relative states was adopted for the purpose of evaluating technology groups with regard to their values and environmental influence. According to that scale the smallest value 1 corresponds to a minimum level, and the highest value 10 is the level of perfection.

The dendrological matrix of technology value [2] presents graphic results of evaluating specific technology groups, with special attention paid to the potential constituting the real objective value of a given technology and to the attractiveness reflecting how a given technology is subjectively perceived among its potential users. The potential of a given technology group expressed through a ten-point universal scale of relative states, marked on the horizontal scale of the dendrological matrix is the result of a multi-criteria analysis carried out based on an expert opinions. On the vertical scale of the dendrological matrix the level of attractiveness was marked of a given technology group which is the mean weighed expert opinions based on detailed criteria. Depending on the type of potential and level of attractiveness determined as part of the expert opinions, a given technology may be placed in one of the quarters of the matrix. In Table 1 the quarters distinguished in the dendrological matrix of technology value are presented.

Table 1. *The quarters of the dendrological matrix of technology value [38]*

Factors		Potential	
		Low	High
Attractiveness	High	A **quaking cypress** which is technology with a limited potential, but highly attractive, what causes that a success of technology is possible	A **wide-stretching oak** which corresponds to the best possible situation in which the analysed technology has both a huge potential and huge attractiveness, which is a guarantee of a future success
	Low	A **sparing aspen** which is technology with a limited potential and limited attractiveness in the range, which a future success is unlikely	A **rooted dwarf mountain pine** which is technology with limited attractiveness, but a high potential, so that its future success is possible

The meteorological matrix of environment influence [2] presents graphic results of evaluating the impact of external factors on specific technology group which had been divided into difficulties with a negative impact and chances which positively influence the analysed technologies. The testing of expert opinions on the subject of positive and negative factors which influence specific technologies was carried out based on a survey pertaining to the micro- and macroenvironment. External difficulties expressed with the use of a ten-point universal scale of relative states (from 1 to 10), which are the result of a multi-criteria analysis conducted based on the expert opinions, have been placed on the horizontal scale of the meteorological matrix. On the other hand, chances, i.e. positive environment factors being a mean weighed expert opinions based on detailed criteria, were placed on the analysed vertical scale. Depending on the level of influence of positive and negative environment factors on the analysed technology, determined as part of the expert opinions on a ten-point scale, it is placed in one of the matrix quarters. In Table 2 the quarters distinguished in the meteorological matrix of environment influence are presented.

Table 2. *The quarters of the meteorological matrix of environment influence [38]*

Factors		Difficulties	
		A small number	A large number
Chances	A large number	**Sunny spring** being the best option denoting friendly environment with lots of opportunities and a little number of difficulties, which means that the success of given technology is guaranteed	**Hot summer** corresponding to a situation in which the environment brings a lot of opportunities, which, however, are accompanied by many difficulties, meaning that the success of technology in the given circumstances is possible, but is a subject to the risk
	A small number	**Rainy autumn** corresponding to the neutral position, in which for given technology traps do not wait, but also the environment does not give too many opportunities	**Frosty winter** corresponding to the worst possible situation in which the environment brings a large number of problems and few opportunities, which means that success in a given environment is difficult or impossible to achieve

A **matrix of strategies for technologies** includes the research results transformed from a dendrological matrix of technology value, as well as a meteorological matrix of environment influence. A matrix of strategies for technologies consists of sixteen fields corresponding to each set of versions resulting from the combination of the types of technology and the types of environments. To facilitate the transfer of specific numeric values from the dendrological matrix [2x2] and the meteorological matrix [2x2] to the matrix of strategies for technologies with the dimensions of [4x4], mathematical relations and simple software were formulated [2] which enable the rescaling and objectivising of test results.

The **strategic development tracks** for analysed technologies/ technology group forecast given technology development successively in each five years during future twenty years being the time horizon of carried out research. The strategic development tracks in three versions: optimistic, pessimistic and most possible ones, were prepared. Also, they were visualised against a background of a matrix of strategies for technologies.

Table 3.*Chemical composition of the tested hot-work alloy tool steel*

Steel grade	Mass concentration of elements, %								
	C	Mn	Si	P	S	Cr	W	Mo	V
X40CrMoV5-1	0.41	0.44	1.09	0.015	0.010	5.40	0.01	1.41	0.95
32CrMoV12-28	0.308	0.37	0.25	0.020	0.002	2.95	–	2.70	0.535

A **series of materials science research** using specialised diagnostic and measurement equipment were carried out in order to precise the value of the potential and attractiveness of laser treatment of hot-work alloy tool steels. Tests were undertaken on the samples made of hot-work alloy tool steel: X40CrMoV5-1 and 32CrMoV12-28 with the chemical composition as provided in Table 3. The material for tests was poured into an ingot of approx. 250 kg after being melted in an electric vacuum furnace at the pressure of approx. 1 Pa and then was subjected to preliminary forging into rods with the diameter of 76 mm and 3 m long. The rods were next soft annealed to ensure good workability and uniform carbides distribution in the matrix. Samples were made by machining and the samples next underwent standard heat treatment (selected acc. to product sheets) including quenching and double tempering. X40CrMoV5-1 steel was austenitised in a vacuum furnace at 1020°C with annealing lasting 30 min. Two 30 min. isothermal intervals were made while heating to the austenitising temperature, the first one at 640°C and the other at 840°C. The samples were tempered twice after quenching, each time for 2 h at 560°C, and then at 510ºC. 32CrMoV12-28 steel was austenitised at 1040ºC, with annealing for 30 min. Two isothermal intervals were made while heating to the austenitising temperature, the first one at 585ºC, and then 850ºC. Double tempering for 2 hours was made after quenching at 550ºC, and then 510^{o} C. The samples after heat treatment were sand blasted and worked mechanically with a magnetic grinder. Special heed was paid to preventing from creating microcracks that could have disqualified a sample for further testing. Approx. 0.05 mm thick powder coatings were applied onto to the degreased surfaces of the samples in form of paste containing, respectively, tungsten carbide, niobium carbide NbC, vanadium carbide VC, titanium carbide TiC or tantalum carbide TaC bonded with inorganic binder in form of silicate water glass with the composition of Na_4SiO_4+$Na_2Si_2O_5$. The selected properties of powders are shown in Table 4, and the data in Figures 1-4 shows the topography of the selected powders used for the laser alloying of the tested steels. The laser cladding or laser alloying of the following hot-work alloy tool steel grades: X40CrMoV5-1 and 32CrMoV12-28 was carried out using a SINAR DL 020 High-Power Diode Laser (HPDL). Its technical specifications are provided in Table 5. The laser cladding or alloying processes were performed at a constant speed of 0.5 m/min with the process progress at such speed being stable, changing the laser beam power within 1.2-2.3 kW. A protected atmosphere was used to secure the melting area from the access of air and shielding gas (argon) through a ϕ 12 mm round nozzle was supplied for the purpose at a rate of 20 l/min. It was found based on the tests, including structural and hardness tests and a qualitative X-ray phase analysis, that it is advantageous to introduce the following ceramic powders to improve the functional properties of the surface layer: vanadium carbide, wolfram carbide, titanium carbide, tantalum carbide and niobium carbide; however, if oxide and nitride powders are introduced to the surface layer of hot-work steel, this does not improve the tested properties or such powders are not introduced into the melted steel. For this reason it was not verified positively if it is reasonable to use oxide and nitride oxides. The greatest improvement in the properties of the steel surface layer was achieved using titanium carbide and vanadium carbide powders for laser alloying. The steel alloyed with the above powders imparts high hardness and low roughness of the laser-treated surface. Further tests were undertaken to select the powders ensuring the best properties to the steel surface layer after laser alloying, in particular resistance to abrasive wear for the metal-ceramic material configuration. The smallest mass loss in the metal - ceramic material configuration for the tested ceramic powders is seen for the steel alloyed with titanium carbide and vanadium carbide powders. The mass loss for the steel alloyed with wolfram carbide, niobium carbide and tantalum carbide powder is comparable to this for remelted steel. The abrasive wear resistance tests for the metal-metal configuration imitating tool work in industrial conditions (abrasion of matrices or forging tools) confirm the results of the previous tests. In such configuration, the steel surface layer produced as a result of alloying with vanadium carbide or titanium carbide exhibits greater abrasive resistance.

Table 4. *Selected properties of ceramic powders*

Coating Type	Hardness HV, GPa	Melting Point, °C	Density, g/cm^3	Thermal expansion coefficient α, $10^{-6}\cdot K^{-1}$
WC	2400	2730-2870	15.77	23.8
NbC	1800	3480-3610	7.6	7.6
VC	2600	2650-2830	5.81	7.5
TiC	3200	3065-3180	4.94	8.3
TaC	1600	3780-3985	14.5	7.8

Table 5. *Technical data of HPDL ROFIN DL 020 diode laser*

Parameter	Value
Length of laser radiation waves, nm	808±5
Output power of laser beam (constant radiation), W	2300
Power range, W	100-2300
Focal length of laser beam, mm	82 / 32
Focal point dimensions of laser beam, mm	1.8 x 6.8 / 1.8 x 3.8
Power density range in laser beam focal plane, kW/cm^2	0.8-36.5

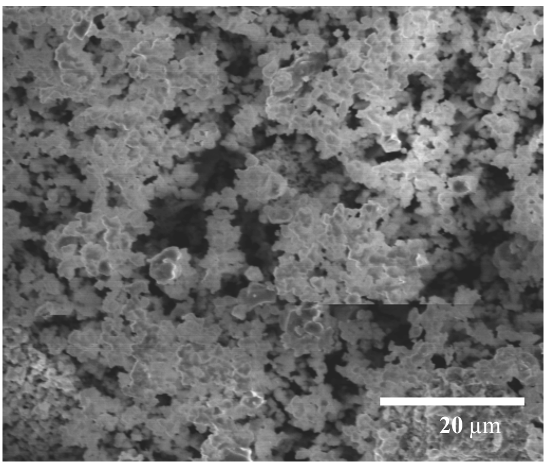

Figure 1. *Topography of tantalum carbide powder (TaC) used for the tests (SEM)*

Roughness was measured with a Surtronic 3+ contact profilometer by Taylor-Hobson. The samples' surface was cleaned with acetone, and then an average arithmetic roughness profile deviation R_a was measured. The structure of the tested steels was observed with a Leica MEF4A light microscope in a light, dark and polarised field with the magnification of: 25-1000x and with a DSM-940 electron scanning microscope by Opton with the accelerating voltage of 20 kV, using a detector of secondary electrons and backscattered electrons.

The structures were photographed with a Leica - Qwin computer-aided image analysis system. A qualitative X-ray microanalysis and a surface and linear distribution analysis of alloy elements in the samples of the tested steels in the quenched condition was performed with a DSM-940 scanning microscope by Opton fitted with a LINK ISIS energy dispersive spectrometer (EDS) by Oxford at the accelerating voltage of 20 kV. The direct structure tests in a light field and in a dark field and diffraction tests enabling a phase analysis of the selected micro-area were carried out in a JEM 3010UHR transmission electron microscope by JEOL, at the accelerating voltage of 300 kV.

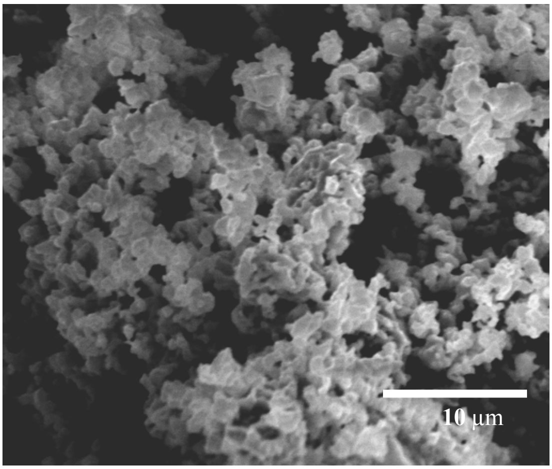

Figure 2. *Topography of niobium carbide powder (NbC) used for the tests (SEM)*

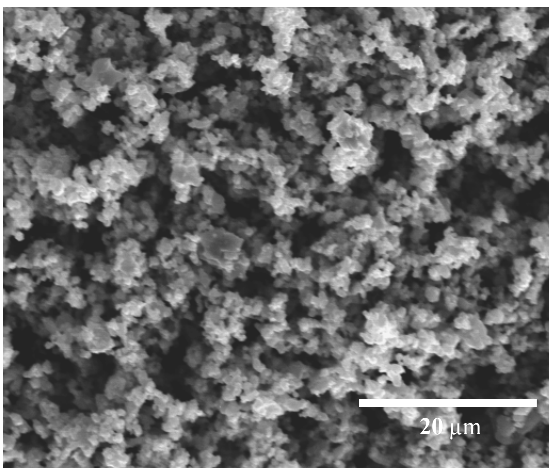

Figure 3. *Topography of vanadium carbide powder (VC) used for the tests (SEM)*

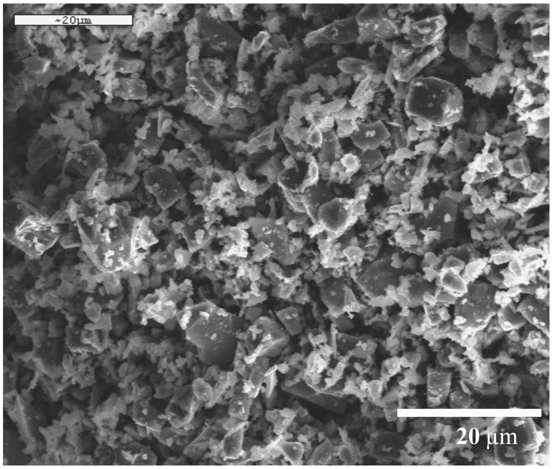

Figure 4. *Topography of titanium carbide powder (TiC) used for the tests (SEM)*

Hardness measurements were made on the surface layers of the samples cleaned mechanically using a Zwick ZHR 4150TK hardness tester with an electronic sensor fitted allowing to read hardness directly with Rockwell's method. The tests were made for all the laser-cladded or alloyed samples and also for the sample made of the tested steels subjected to conventional heat treatment. The wear resistance of the surface layers achieved from laser cladding or alloying using the following carbide powders: WC, NbC, TaC, VC and TaC was determined with an abrasability test acc. to the American standard – ASTM G65-04. The data presented as a quotient of the mass loss of the laser-remelted or alloyed sample with carbide powder in respect of the mass of the sample heat-treated conventionally. It was concluded based on the tests results that a relationship exists for the tested steels between the laser power used for alloying and the resistance to abrasive wear. The abrasive wear resistance tests with the metal – ceramic material method were carried out at a stand designed at the Welding Department of the Silesian University of Technology, Gliwice, in accordance with ASTM G65 standard. The mass loss according to laser power used for cladding or alloying and according to the alloying material was measured with Wa 33 PRL T A13/1 laboratory balance with the accuracy of up to 0.0001 g. The samples were weighed before and after the abrasion test.

A set of the **technology roadmaps** [2, 3, 40] on the basis of source data received during carried out experimental-comparative research were prepared. The layout of the technology roadmap created for the purpose of the realised research corresponds to the first quarter of the

Cartesian coordinate system. Three time intervals were placed on the horizontal axis, pertaining to: the situation as of today, in ten years' and in twenty years' time. The time horizon of all the research placed on the technology roadmap equals 20 years and is adequate to the dynamics of changes occurring in the surface engineering area. On the vertical axis of the technology roadmap seven main layers were placed corresponding to a specific question pertaining to the analysed scope. Each of the main layers has been additionally divided into more detailed sub-layers. In addition, the technology roadmap presents relations between its specific layers and sub-layers, with a division into: cause-and-effect relations, capital relations, time correlations and two-way flows of data and/or resources, visualised using different types of arrows. The technology roadmap is a universal tool which enables presenting, in a unified and clear format, different types of internal and external factors directly and indirectly characterizing a given technology, taking into account the ways of influence, interdependencies and the change of specific factors over time. When needed, the technology roadmap may be supplemented and expanded by additional sub-layers, adapting it, e.g. to the specificity of the carried out scientific-research studies, the requirements of a given industrial field or the enterprise size.

Using the adopted set of interdisciplinary methods, a research cycle was performed; the results of the research are presented in the present chapter. The most important among them are those related to evaluation of the potential and the attractiveness of the technology group in question on the background of their environment, which was performed based on the opinions of key experts, expressed in a ten-point universal scale of relative states. The next step was to formulate the recommended strategy for a given technology and the forecast strategic development tracks (sub-chapter 3). The results of the series of materials science experiments intended to determine the impact of the selected laser treatment parameters on the structure and the characteristics of the hot-work alloy tool steels in question which were laser-melted and/or alloyed are presented in sub-chapter 4 of the chapter. In particular, the sub-chapter describes the results of the metallographic structure tests involving scanning electron microscopy, hardness and micro-hardness tests, and micro-analysis of the chemical composition. Sub-chapter 5 of the chapter presents the roadmaps for the technology group in question, prepared based on the results of the interdisciplinary experimental and comparative research which was performed. The technology roadmaps show, in a uniform and clear format, the internal and external factors which directly characterize the individual technology group, taking into account the interactions, the mutual relations, and the change of the individual factors in time.

3. Evaluated value and development directions of analysed technologies

Taking as a criterion of the division, a type of powder deposited to the substrate, in order to carry out comparative and experimental works, five homogeneous groups were isolated from the analysed technologies in turn:

(A) hot-work alloy tool steels which underwent laser treatment by NbC niobium carbide;
(B) hot-work alloy tool steels which underwent laser treatment by TaC tantalum carbide;
(C) hot-work alloy tool steels which underwent laser treatment by TiC titanium carbide;
(D) hot-work alloy tool steels which underwent laser treatment by VC vanadium carbide;
(E) hot-work alloy tool steels which underwent laser treatment by WC tungsten carbide.

The individual technology group were evaluated by key experts with regards to their attractiveness and potential, using a ten-point universal scale of relative states. Using a multi-criterion analysis, the weighted average was calculated of the detailed criteria selected within the attractiveness and the potential, and the results obtained for the individual technology group were charted on the dendrological matrix of technology value (Fig. 5). As a result of the analysis, all technology group were classified in the most promising quarter of the matrix, which covered technologies with a great potential and high attractiveness, called a patulous oak tree. The best result with regards to the internal potential, which demonstrates the objective value of the technology, was achieved by the C (8.05, 7.75) technology group, which includes laser treatment of hot-work alloy tool steel using TiC titanium carbide powder. The technology group which was found to be the most attractive was laser treatment of hot-work alloy tool steels using VC vanadium carbide powder, designated as D (6.95, 8.20), which should lead to the greatest interest in this technology group among potential buyers and users.

The meteorological matrix of environmental influence has been used to evaluate the positive and negative impact of the environment on the individual technology group. The results of the multi-criterion analysis performed on the opinions of experts who filled out a survey form comprising several dozen questions, were charted on the meteorological matrix (Fig. 6). The research indicates that the most advantageous environmental conditions, corresponding to early spring, are associated with the following technology group: C (5.10, 5.72) – laser treatment of hot-work alloy tool steels using TiC titanium carbide powder, and

D (5.37, 5.81) – laser treatment using VC vanadium carbide powder. Hot summer, which brings both numerous opportunities and numerous difficulties, is the environment for the A (6.68, 5.73) technology group involving laser treatment of hot-work alloy tool steel using NbC niobium carbide powder and the B (6.10, 6.31) technology group involving laser treatment using TaC tantalum carbide powder. Almost in the very centre of the matrix, yet in the field of rainy autumn with few opportunities and difficulties, was the E (5.42, 5.49) technology group involving laser treatment of hot-work alloy tool steel using WC tungsten carbide powder.

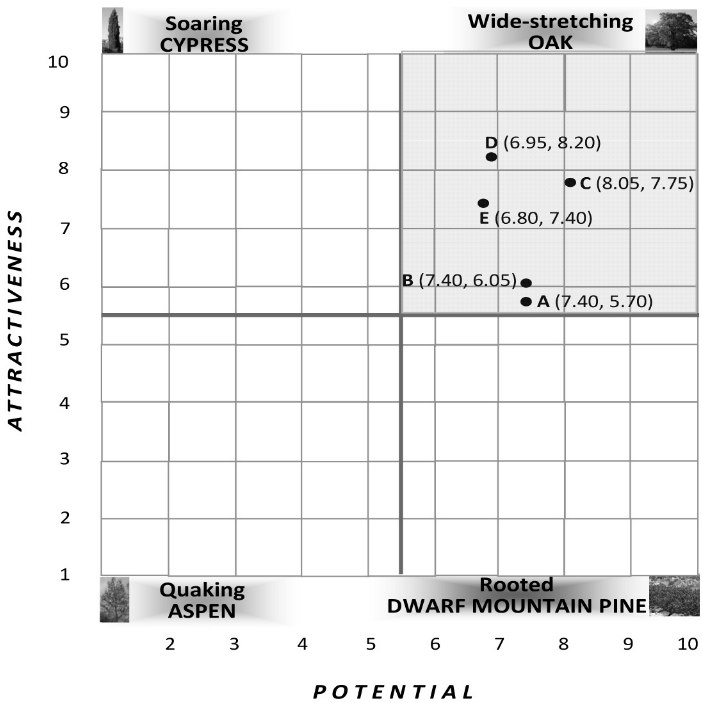

Figure 5. *The dendrological matrix of technology value for the laser treatment of hot-work alloy tool steels using NbC (A), TaC (B), TiC (C), VC (D) and WC (E) carbide powders*

At the next stage of research, the results of the research, presented in a graphic form with a dendrological matrix of technology value and the meteorological matrix of environmental influence, were charted on the matrix of strategies for technologies (Fig. 7). The matrix graphically depicts the place of the different groups of hot-work alloy tool steel laser treatment technologies, taking into account their value, which is the product of their potential and attractiveness, and the strength of the environmental influence, and indicates the appropriate strategy.

The transfer of specific numerical values from the dendrological matrix and the meteorological matrix into the matrix of strategies for technologies with different dimensions was performed using mathematical relations and simple computer software based on such relations, which allowed for scaling and objectivizing the research results [2].

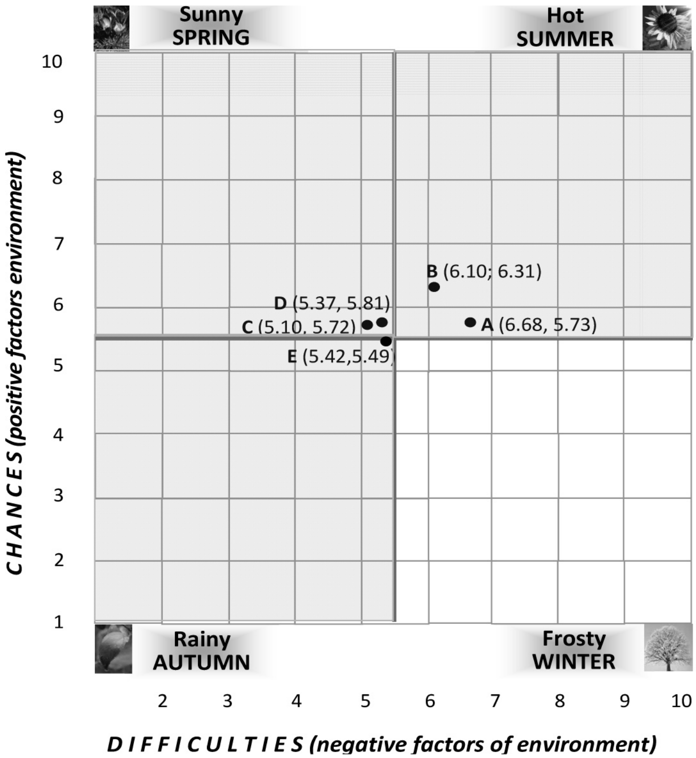

Figure 6. *The meteorological matrix of environment influence for the laser treatment of hot-work alloy tool steels using NbC (A), TaC (B), TiC (C), VC (D) and WC (E) carbide powders*

The C (8.79, 7.86) technology group, involving laser treatment of hot-work alloy tool steel using TiC titanium carbide powder and the D (8.61, 7.80) technology group, involving laser treatment using VC vanadium carbide powder are recommended to use the strategy of an oak in spring. The strategy consists in developing, strengthening and implementing an attractive technology with large potential in industrial practical applications in order to achieve spectacular success. As for the A (8.13, 6.15) technology group, involving laser treatment of hot-work alloy tool steels using NbC niobium carbide powder and the B (8.22, 6.44) technology group,

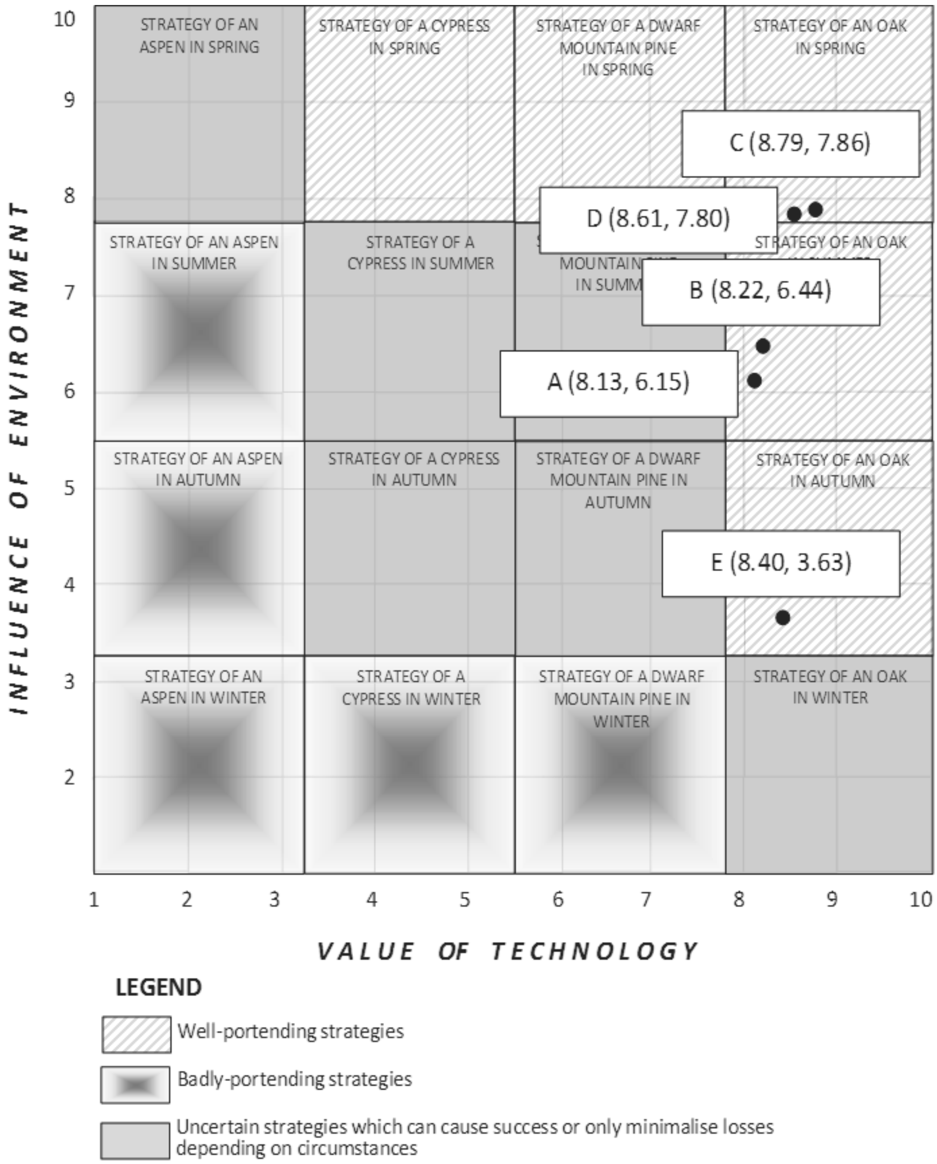

Figure 7. *The matrix of strategies for technology called the laser treatment of hot-work alloy tool steels using NbC (A), TaC (B), TiC (C), VC (D) and WC (E) carbide powders*

involving laser treatment using TaC tantalum carbide powder the strategy of an oak in summer was recommended. The strategy assumes taking advantage of the attractiveness and the potential of the technologies in a risky environment and avoiding possible difficulties, while making the product suitable to meet the requirements of the customers, based on a thorough marketing research. The E (8.40, 3.63) technology group involving laser treatment of hot-work alloy tool steels using WC tungsten carbide requires the oak in the autumn strategy. This strategy involves achieving success with an attractive and stable technology in a reliable market while searching for new markets customer groups and products that can be made using this technology.

The next stage of the research consists in defining, based on experts' opinions, the strategic development tracks for the individual technologies/technology group, which constitute a forecast of their development in the years 2015, 2020, 2025 and 2030, and according to three scenarios: optimistic, pessimistic, and the most likely, and in their display on the background of the matrix of strategies for technologies.

The case selected to be presented in this chapter is the E technology group, involving laser treatment of hot-work alloy tool steels using WC tungsten carbide powder. It is shown in Figure 8, together with the anticipated strategic development tracks for this group according to three scenarios (optimistic, pessimistic, and the most probable), on the background of the matrix of strategies for technologies. The most probable strategic development track for the group of analysed technologies assumes that the neutral environmental conditions, with small number of both opportunities and risks, will be maintained, which will lead to a fairly slow strengthening of the potential and the attractiveness of the technology, which already have a high value. According to this forecast, the (B) technology group will remain in the oak in the autumn field until 2030. The optimistic development track for this technology group assumes that the environmental conditions will improve, which will result in a shift of this technology group in 2025 into the matrix field corresponding to the best possible strategic situation, that of an oak in the spring, where the technology group will stay until the end of the forecast period. The pessimistic scenario, depicted as the third strategic development track, for the technology group in question, assumes that the global crisis will become more severe and the present neutral environment will become more problematic. This will result in a shift of the technology group into the oak in the summer field in 2015. If the circumstances are not advantageous, later (in 2025), the technology group involving laser treatment of hot-work alloy tool steels using WC tungsten carbide powders may shift into the oak in the winter field, due to the higher competitiveness in the sector and the need to intensify the search of new markets, customers, and products that can be made using this technology. The numerical values which result from the research performed for the five analysed technology group, which correspond to the different types of applied powders, are shown in Table 6. Because the different technology group are located in the central part of the meteorological matrix, in many cases despite the fairly small differences in the presented numerical values, it is recommended to use different approaches towards the different fields of the matrices used as an evaluation tool.

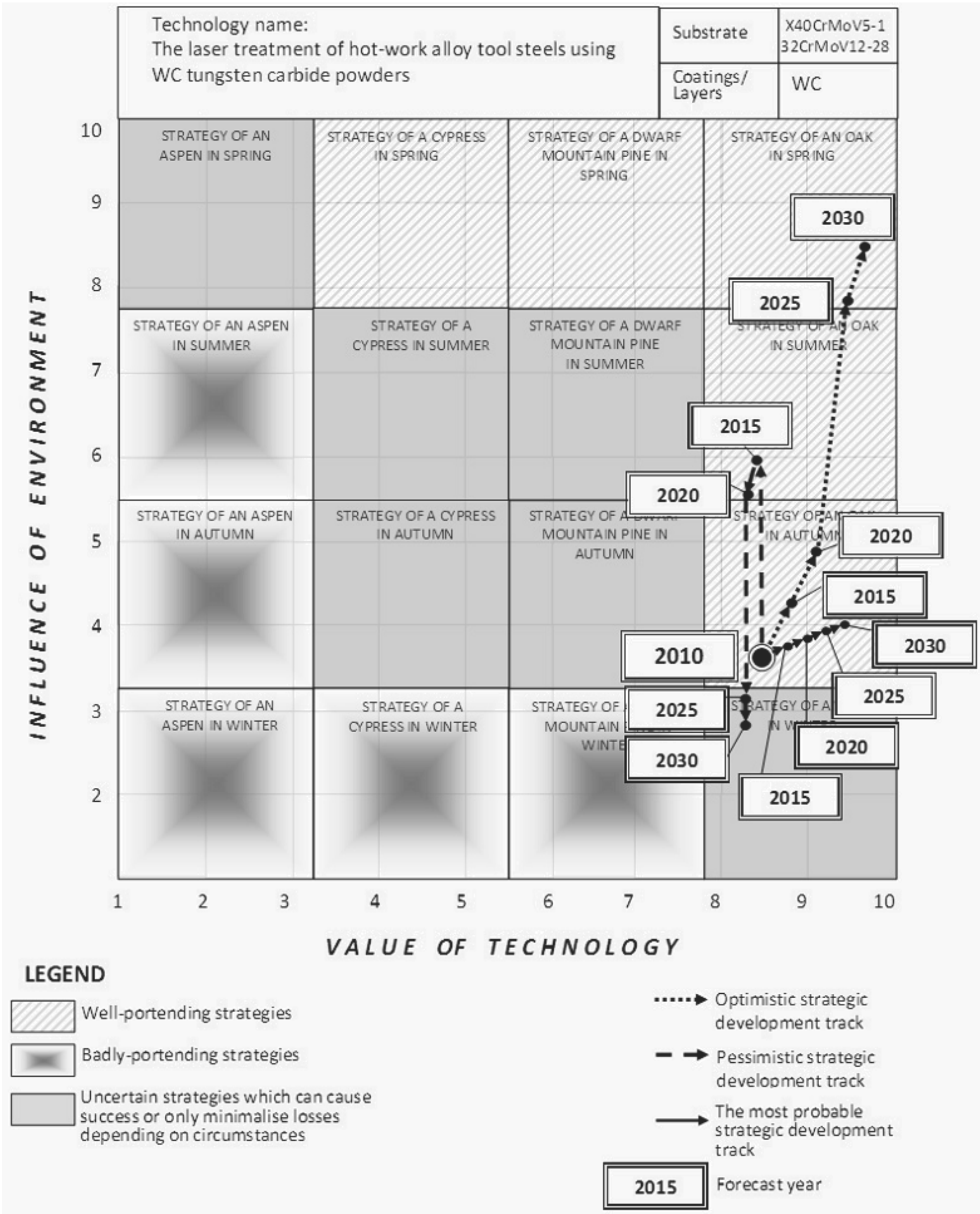

Figure 8. *The strategic development tracks for the (E) demonstration technology called the laser treatment of hot-work alloy tool steels using WC tungsten carbide powders*

Table 6. *Strategic development tracks of laser treatment of hot-work alloy tool steels using carbide powders. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic; (MP) – the most probable*

No.	Technology name	Steady state 2010	Type of strategic development tracks	Years			
				2015	2020	2025	2030
1.	The laser treatment of NbC niobium carbide powders in the surface of hot-work alloy tool steels	Strategy of an oak in summer A (8.1, 6.2)	(O)	(8.3, 6.5)	(8.5, 7.0)	(8.7, 8.0)	(8.8, 8.2)
			(P)	(8.1, 5.7)	(8.1, 3.0)	(8.0, 2.8)	(8.0, 2.6)
			(MP)	(8.2, 5.8)	(8.3, 3.5)	(8.5, 4.2)	(8.6, 4.8)
2.	The laser treatment of TaC tantalum carbide powders in the surface of hot-work alloy tool steels	Strategy of an oak in summer B (8.2, 6.4)	(O)	(8.4, 6.8)	(8.6, 7.4)	(8.8, 8.2)	(9.0, 8.4)
			(P)	(8.3, 5.8)	(8.3, 3.1)	(8.2, 2.9)	(8.2, 2.7)
			(MP)	(8.3, 5.9)	(8.4, 3.7)	(8.6, 4.4)	(8.8, 4.9)
3.	The laser treatment of TiC titanium carbide powders in the surface of hot-work alloy tool steels	Strategy of an oak in spring C (8.8, 7.9)	(O)	(9.0, 8.2)	(9.2 8.4)	(9.4, 8.6)	(9.7, 8.8)
			(P)	(8.8, 7.2)	(8.8, 6.2)	(8.8, 5.9)	(8.8, 3.2)
			(MP)	(8.9, 5.7)	(9.0, 6.3)	(9.1, 8.1)	(9.2, 8.5)
4.	The laser treatment of VC vanadium niobium carbide powders in the surface of hot-work alloy tool steels	Strategy of an oak in spring D (8.6, 7.8)	(O)	(8.8, 8.2)	(9.0 8.4)	(9.2, 8.6)	(9.4, 8.8)
			(P)	(8.6, 7.1)	(8.6, 6.0)	(8.6, 5.8)	(8.6, 3.1)
			(MP)	(8.7, 5.7)	(8.8, 6.2)	(8.9, 7.9)	(9.1, 8.3)
5.	The laser treatment of WC tungsten carbide powders in the surface of hot-work alloy tool steels	Strategy of an oak in autumn E (8.4, 3.6)	(O)	(8.8, 4.3)	(9.2, 4.8)	(9.4, 7.8)	(9.6, 8.5)
			(P)	(8.4, 6.0)	(8.3, 5.6)	(8.3, 3.1)	(8.3, 2.8)
			(MP)	(8.7, 3.7)	(9.0, 3.8)	(9.2, 3.9)	(9.4, 4.0)

4. Received results of materials science research

In order to precise the value of the potential and attractiveness of laser treatment of hot-work alloy tool steels a series of materials science research using specialised diagnostic and measurement equipment were carried out. The outcarried experimental works bowling down to the laser treatment of five different carbide powders of the X40CrMoV5-1 and 32CrMoV12-28 hot-work alloy tool steels the influence of parameters process like power laser and using powders on shape and surface topography were shown. A melted zone (RZ), heat-affected zone (HAZ) and transition limits between the melted zone and heat-affected zone and between the heat-affected zone and the virgin material (VM) exist in each laser cladded or alloyed surface layer of the tested steel (Figs. 9-12). The thickness of the melted zone and the heat-affected zone depends on the laser beam power. The zone thickness increases along with an increase in laser power with alloying speed and alloying coating thickness being constant. Figures 13 and 14 present the effect of laser power on the thickness of the tested steels' melted zone thickness and heat-affected zone.

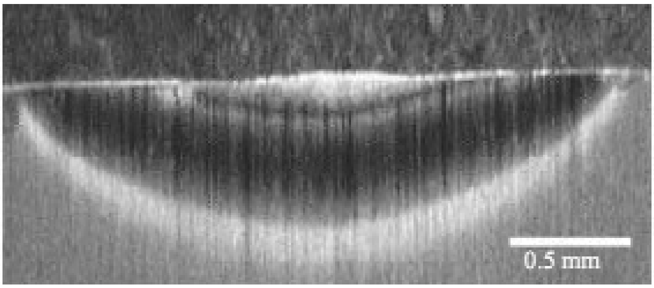

Figure 9. *Surface layer of X40CrMoV5-1 steel after laser cladding, laser power of 1.2 Kw*

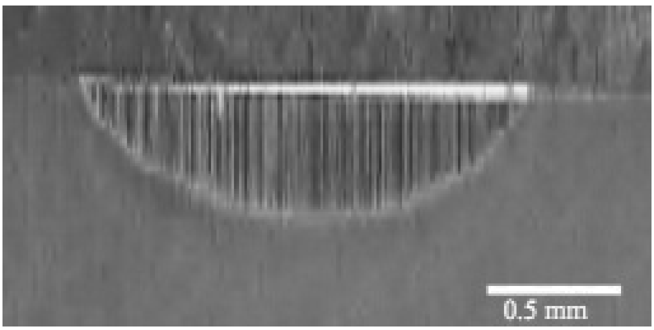

Figure 10. *Surface layer of 32CrMoV12-28 steel after laser cladding, laser power of 1.2 kW*

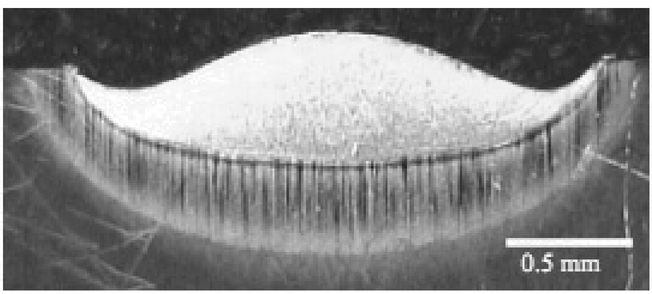

Figure 11. *Surface layer of X40CrMoV5-1 steel after laser alloying with tantalum carbide, laser power of 2.3 kW*

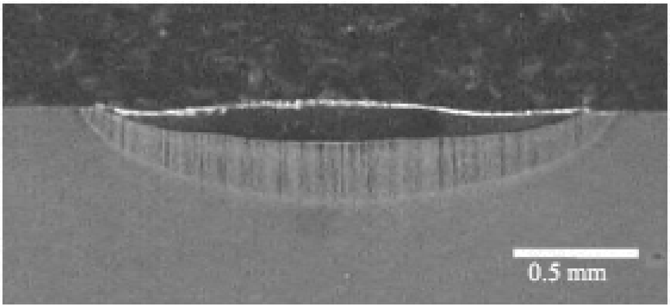

Figure 12. *Surface layer of 32CrMoV12-28 steel after laser alloying with tungsten carbide, laser power of 2.0 kW*

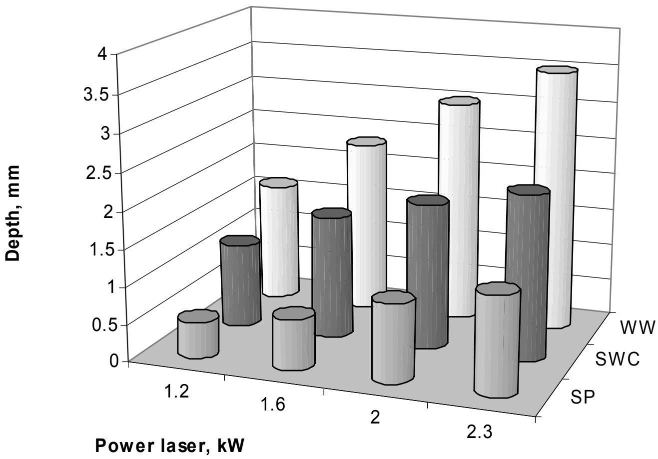

Figure 13. *The effect of laser power on the thickness of RZ and HAZ of X40CrMoV5-1 steel after laser alloying with TaC within the laser power of 1.2-2.3 kW*

The characteristic type of topography and the bead face shape formed from cladding or alloying the surface layers of hot-work X40CrMoV5-1 and 32CrMoV12-28 steel with a HPDL diode microscope within the power range of 1.2-2.3 kW were found based on the observations in a light microscope. The alloying conditions and in particular the laser beam power and the alloying material type have an important effect on the bead face shape. The surface layers produced in the cladding process are characterised by smaller roughness, more regular cladding shape and no cracks as compared to the steel alloyed with carbide powders.

Figures 15-18 show the face view after the laser cladding and alloying of the surface layer depending on the type of the carbide powder and laser power used. Surface roughness is increasing along with rising laser power when carbide powders for alloying are used and when cladding the surface only. Due to small laser beam power (1.2 and 1.6 kW) used for alloying, some grains of the alloying material remain on the surface, thus increasing its roughness, and the high scanning speed influences the specific steel surface shape as a result of fast metal crystallisation according to the heat evacuation direction.

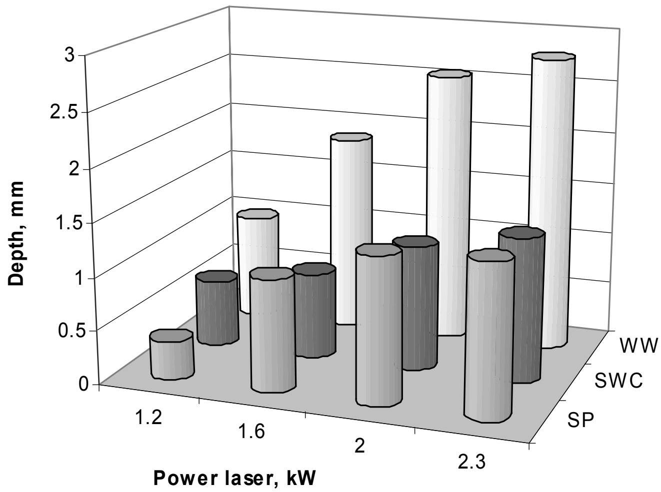

Figure 14. *The effect of laser power on the thickness of RZ and HAZ of 32CrMov12-28 steel after laser alloying with TaC within the laser power of 1.2-2.3 kW*

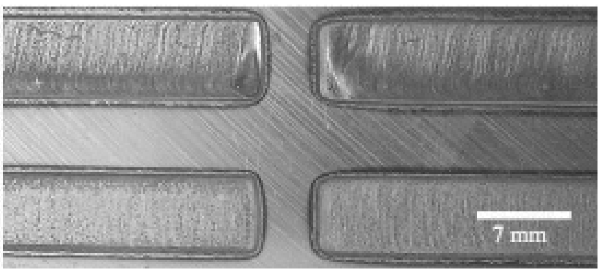

Figure 15. *Face view after laser cladding, X40CrMoV5-1 steel, laser power of 1.2-2.3 kW*

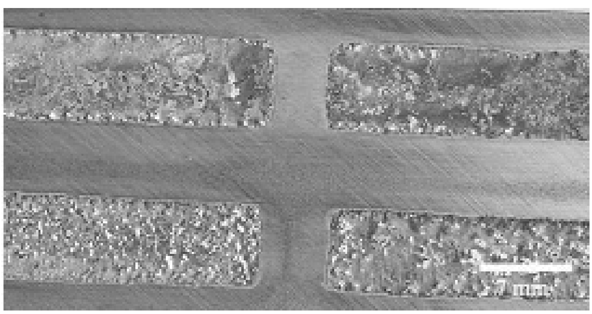

Figure 16. *Face view after laser alloying with tungsten carbide, X40CrMoV5-1 steel, laser power of 1.2-2.3 kW*

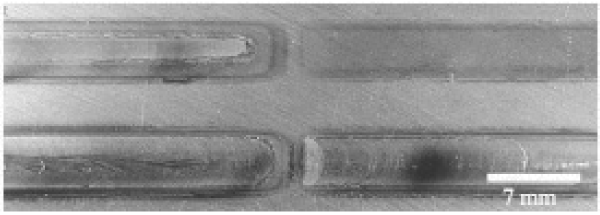

Figure 17. *Face view after laser cladding, 32CrMoV12-28 steel, laser power of 1.2-2.3 kW*

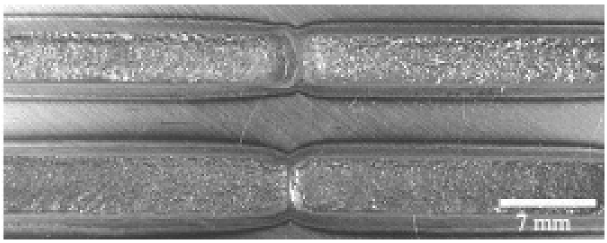

Figure 18. *Face view after laser alloying with vanadium carbide, 32CrMoV12-28 steel, laser power of 1.2-2.3 kW*

Figures 19 and 20 present the effect of laser power on the roughness parameter value for the surface layers of laser-remelted or alloyed steels. Surface roughness is growing along with higher laser beam power as a result of laser cladding or alloying with carbide powders.

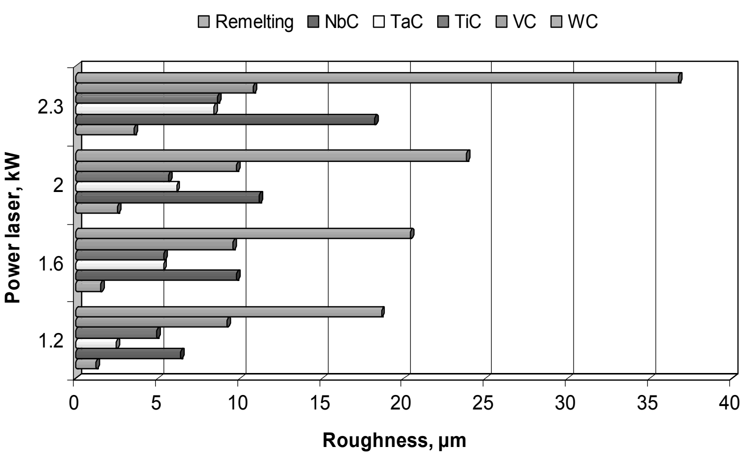

Figure 19. *Average roughness of surface layer for X40CrMoV5-1 steel remelted with laser or alloyed with carbide powders with a 1.2-2.3 kW laser*

It was found based on the observations with a light microscope (Figs. 21-24) that the melted zone of the steel subjected to laser cladding and alloying is of a dendritic structure

(Figs. 25 and 26). The steel structure after cladding and after alloying is characterised by areas with a very differentiated morphology connected with the solidification of the material.

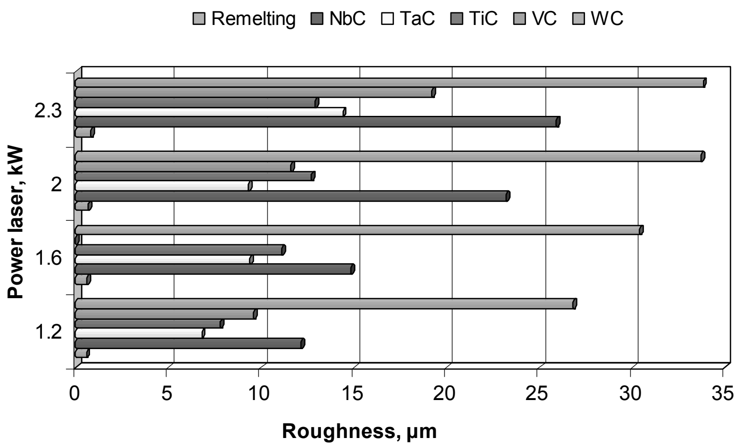

Figure 20. *Average roughness of surface layer for 32CrMoV12-28 remelted with laser or alloyed with carbide powders with a 1.2-2.3 kW laser*

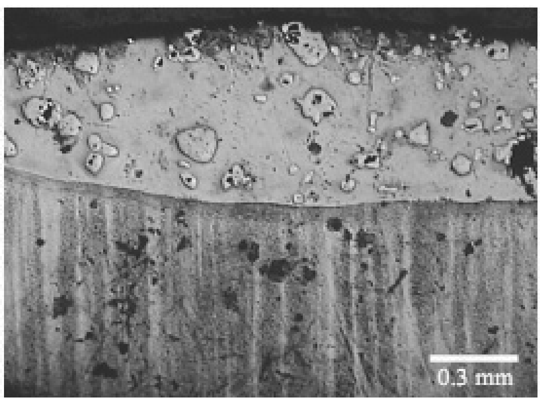

Figure 21. *View of surface layer section after laser alloying of X40CrMoV5-1 steel, tungsten carbide powder, laser power of 2.3 kW*

The clusters of unmelted alloying material carbides occur in the central area of the melted zone. The convective movements present during steel cladding and alloying are "freezing" due to the sudden solidification of the melted zone (Figs. 27 and 28).

The observations carried out with a scanning electron microscope have revealed that the surface layer of the tested X40CrMo5-1 steel subjected to laser cladding and alloying shows a zonal structure, especially the melted zone and the heat-affected zone (Figs. 29 and 30).

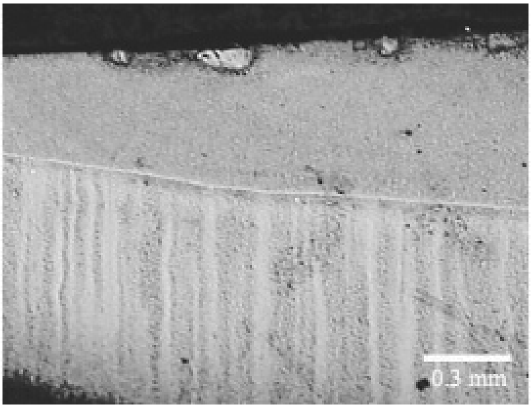

Figure 22. *View of surface layer section after laser alloying, X40CrMoV5-1 steel, vanadium carbide powder, laser power of 2.0 kW*

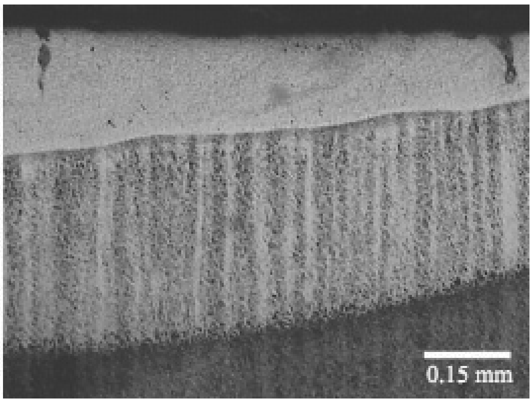

Figure 23. *View of surface layer section after laser alloying, 32CrMoV12-28 steel, vanadium carbide powder, laser power of 1.2 kW*

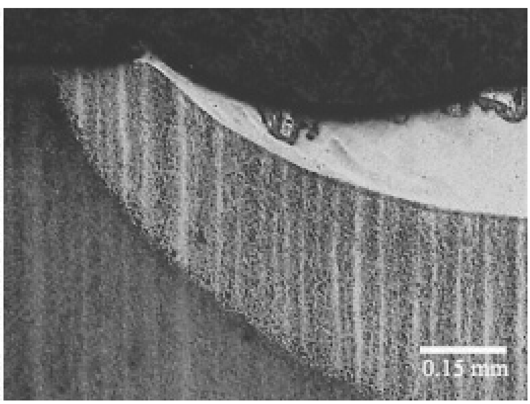

Figure 24. *View of surface layer section after laser alloying, 32CrMoV12-28 steel, vanadium carbide powder, laser power of 2.3 kW*

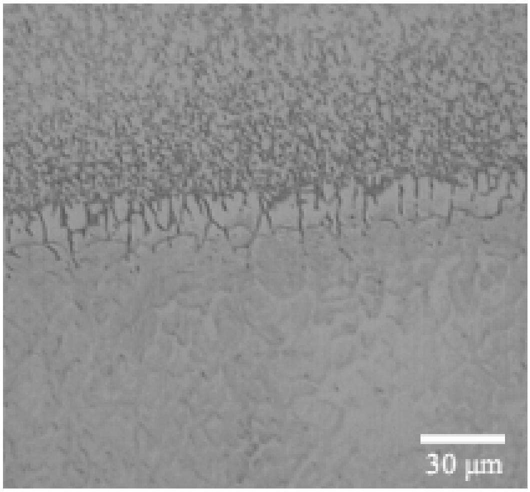

Figure 25. *Melted zone limit of X40CrMoV5-1 steel surface layer, after alloying with titanium carbide, laser power of 2.3 kW*

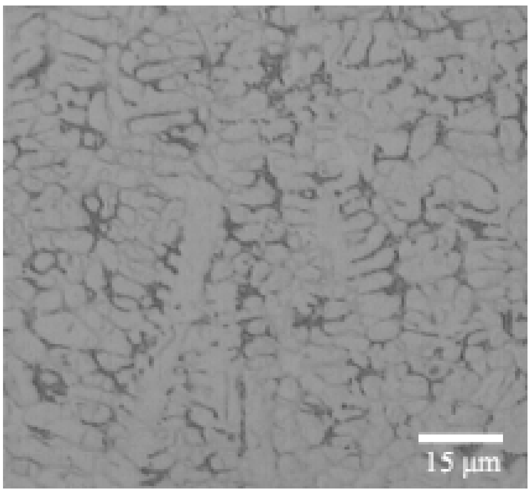

Figure 26. *Central zone of remelted surface layer of X40CrMoV5-1 steel after remelting, laser power of 2.3 kW*

The presence of the elements contained in the tested steel for X40CrMoV5-1 and 32CrMoV12-28W material was found as a result of a linear and local chemical composition analysis using a scattered X-ray radiation spectrometer performed on the lateral microsections of the surface layer remelted or alloyed with laser with different power rating. The presence of the elements contained in the surface layer formed due to alloying with carbide powders was also concluded on the basis of the tests. The local analysis of chemical composition confirms the presence of the elements contained in the tested X40CrMoV5-1 steel: C, Fe, Mn, Si, Cr, W, Mo, V and the elements originating from the alloying material. It was also found in case of 32CrMoV12-28 steel that the following elements are present: C, Fe, Mn, Si, Cr, W, Mo, V,

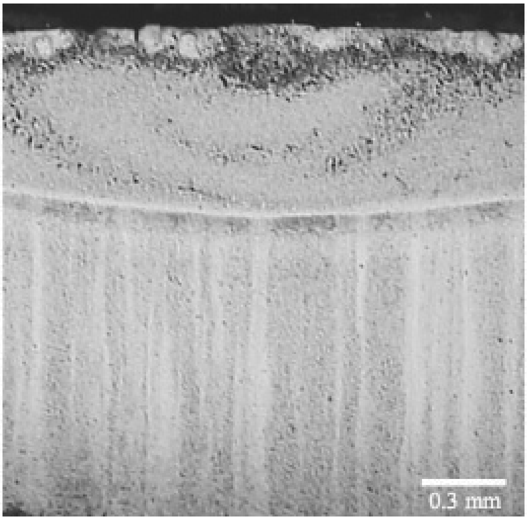

Figure 27. *Melted zone limit of the steel surface layer after alloying with vanadium carbide, laser power of 1.6 kW*

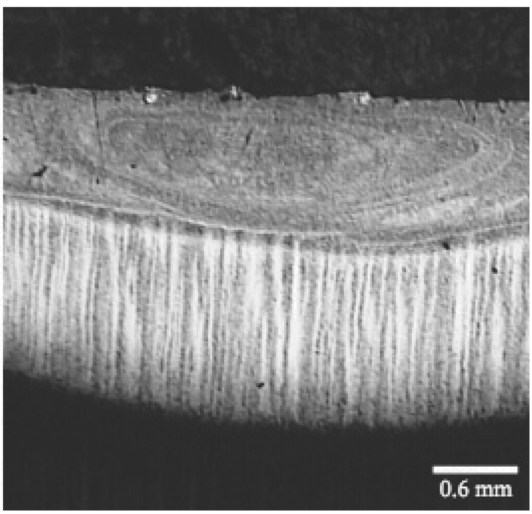

Figure 28. *Melted zone limit of the steel surface layer after alloying with titanium carbide, laser power of 2.0 kW*

however with a different proportion as appropriate for this material. The diagrams of concentration according to scattered radiation energy show the results of the local analysis in the micro-areas of the matrix and carbides (Figs. 31-36).

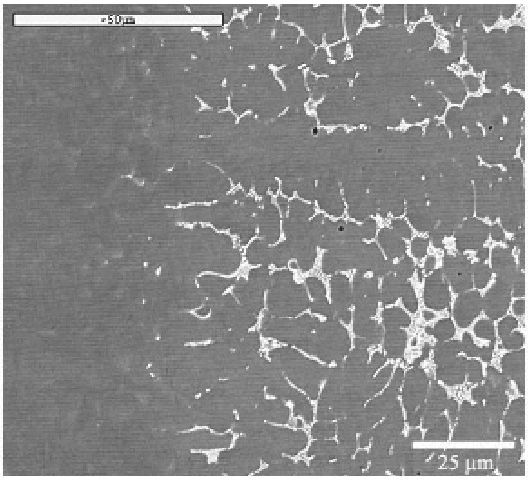

Figure 29. *Zonal structure of the surface layer of X40CrMo5-1 steel alloyed with TaC powder, the laser beam power of 1.6 kW*

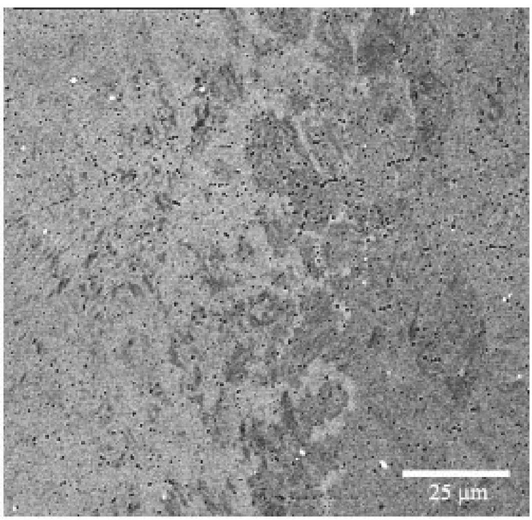

Figure 30. *Zonal structure of the surface layer of X40CrMo5-1 steel remelted with laser, the laser beam power of 1.2 kW*

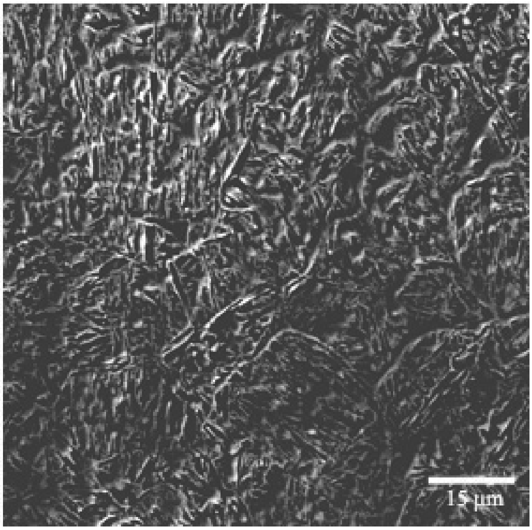

Figure 31. *Surface layer of steel after alloying with vanadium carbide VC powder, the laser beam power of 2.3 kW (SEM)*

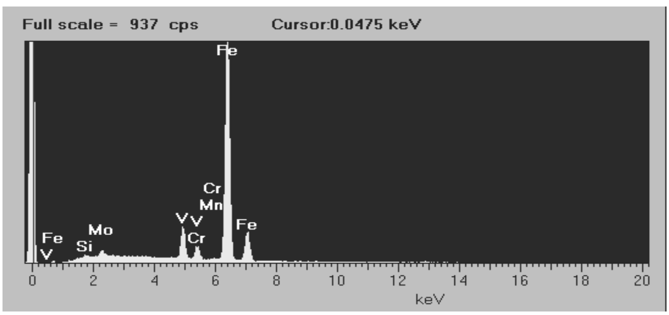

Figure 32. *Diagram of concentration according to energy for the dispersed X-ray radiation for the sample of 32CrMoV12-28 steel after laser alloying with VC vanadium powder*

The tests of thin foils made in a transmission electron microscope with X40CrMoV5-1 and 32CrMoV12-28 steel samples reveal that martensite constitutes the structure of this steel in a quenched and twice-tempered condition (Fig. 37), tempered with dispersion secretions of M_7C_3 carbide. It was found that the respective carbides used for alloying occur on the limits of grains based on the tests of thin foils made using the surface layer of hot-work tool steel alloyed with carbide powders (Figs. 37 and 38). Lath martensite with a high dislocation density constitutes the surface layer matrix after alloying. The tests with a transmission electron microscope have confirmed the presence of particles of all the types of carbides used for alloying: vanadium carbide, wolfram, titanium, niobium and tantalum. The size of the carbide particles found indicate a differentiated size - smaller than this would be shown by the grain size of the ceramic powder used for the tests. This indicates that the particles of carbide powders in the steel matrix have dissolved, however, a more thorough examination made in the future would have permitted to conclude to what extent the alloying material has dissolved in the matrix.

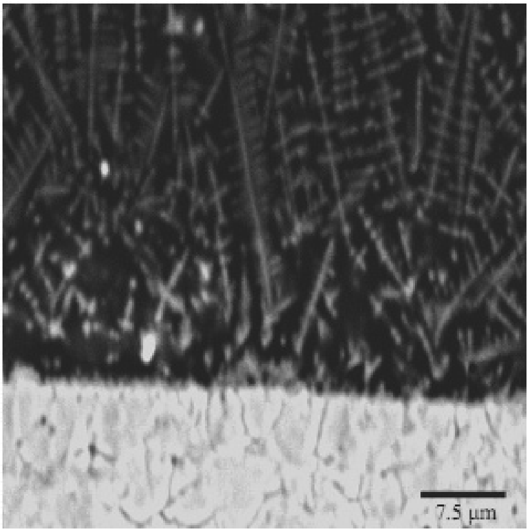

Figure 33. *Surface layer of 32CrMoV12-28 steel after alloying with VC powder, the laser beam power of 2.3 kW*

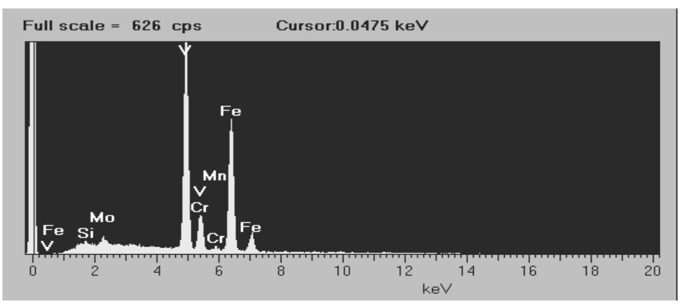

Figure 34. *Diagram of concentration according to scattered X-ray radiation energy for a 32CrMoV12-28 steel sample after laser alloying with VC vanadium powder*

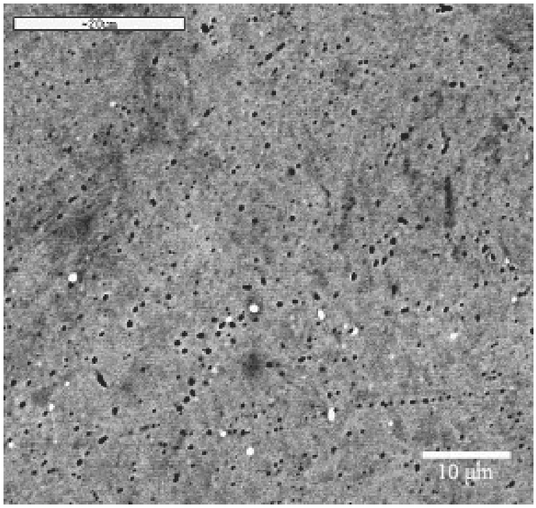

Figure 35. *Central zone of cladding the surface layer of X40CrMoV5-1 steel sample after cladding with laser, the laser beam power of 1.2 kW*

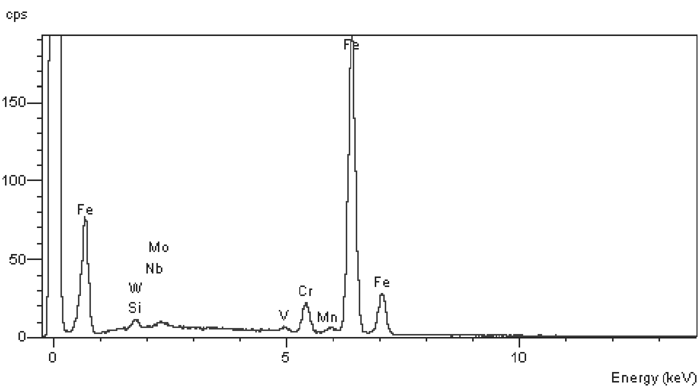

Figure 36. *Diagram of concentration according to scattered X-ray radiation energy for a X40CrMoV5-1 steel sample after cladding with laser, the laser beam power of 1.2 kW*

Laser treatment in the majority of cases increases the hardness of the tested steels and hardness is growing along with higher laser power used for alloying, as shown in Figures 39 and 40. Material hardness after alloying did not rise for titanium carbide only.

The tribological properties of steel are rising along with the growing surface layer hardness after laser alloying. Figs. 41 and 42 present the wear trace of the tested surface layers of hot-work alloy tool steels and Figs. 43 and 44 show the relative mass loss of the sample made of the tested steels according to the alloying material used and the laser power used for laser treatment.

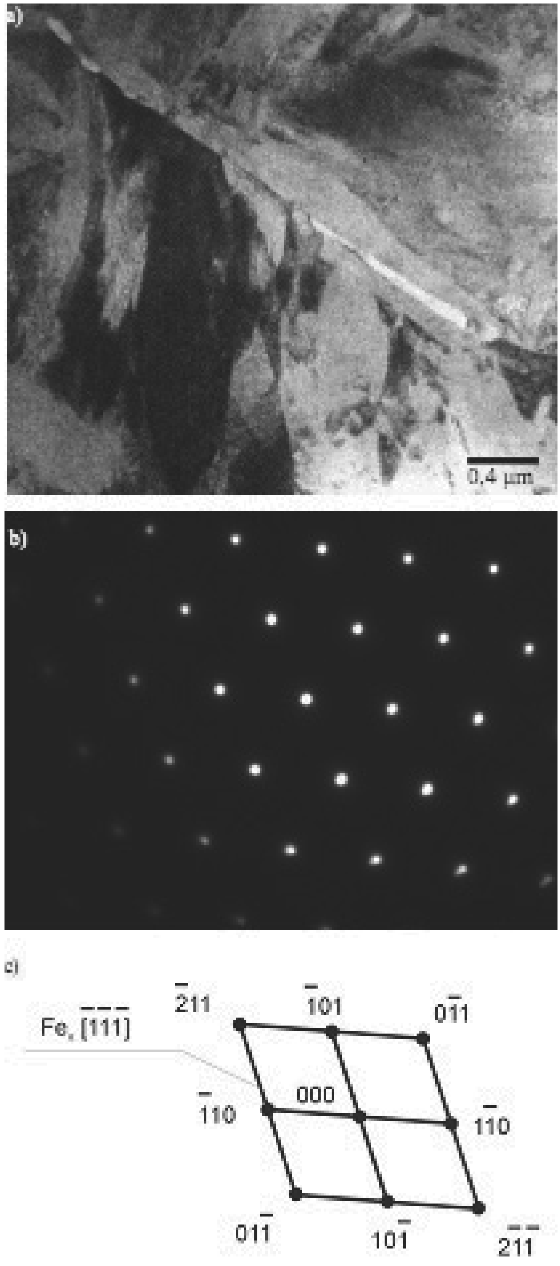

Figure 37. *Structure of thin foil made of 32CrMoV12-28 steel after alloying with VC vanadium carbide with the following parameters: scanning speed – 0.5 m/min, beam power – 2.0 kW, a) image in light field, b) diffraction pattern from the area as in figure a), c) diffraction pattern solution for figure b)*

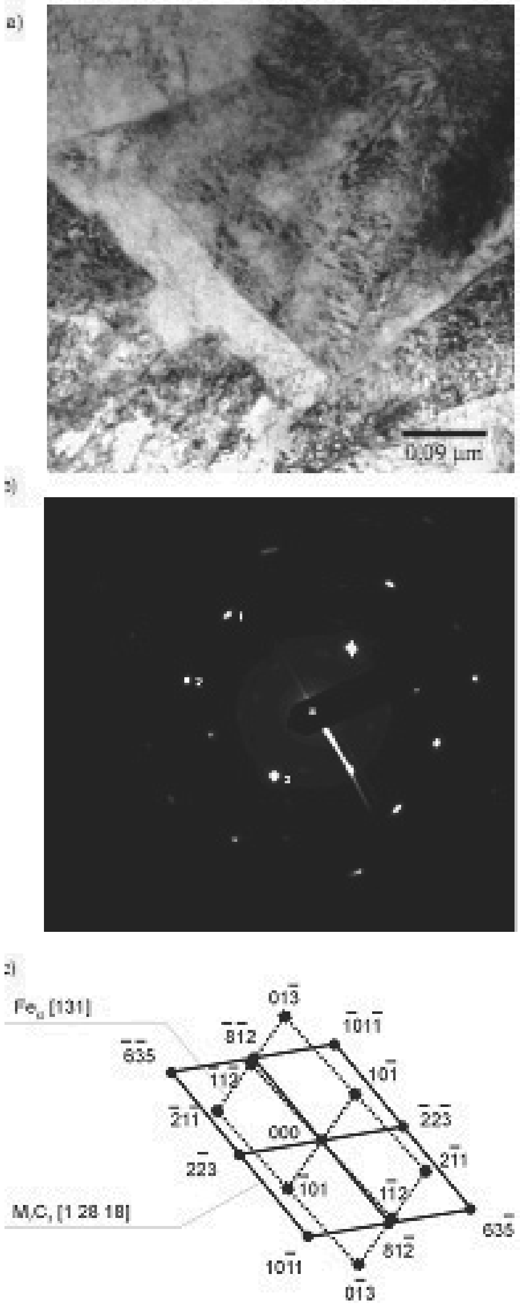

Figure 38. *Structure of thin foil made of X40CrMoV5-1 steel after alloying with NbC niobium carbide, laser beam power of 2.0 kW, a) image in light field, b) diffraction pattern from the area as in figure a), c) diffraction pattern solution for figure b)*

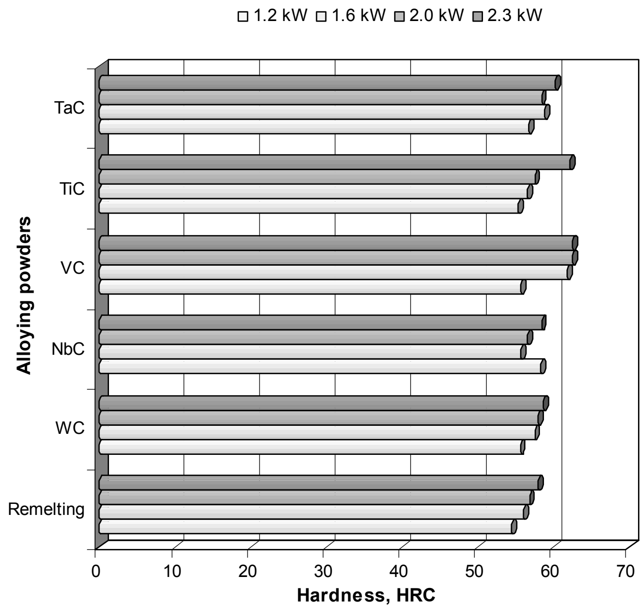

Figure 39. *Variation in surface layer hardness of X40CrMoV5-1 steel remelted or alloyed with the laser power range of 1.2-2.3 kW*

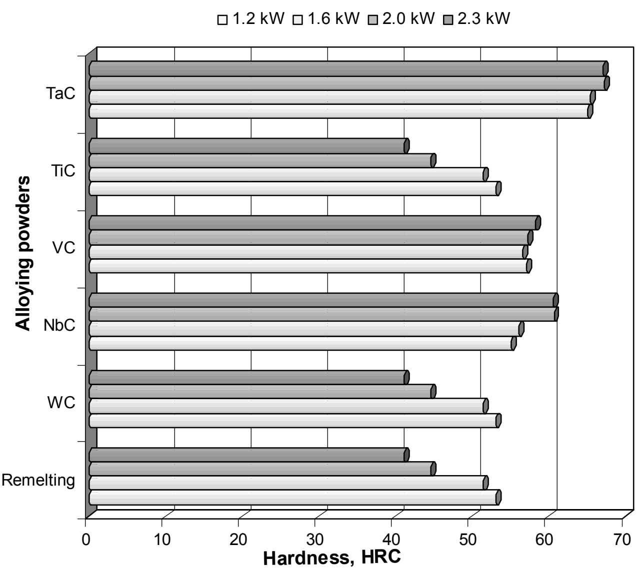

Figure 40. *Variation in surface layer hardness of 32CrMoV12-28 steel remelted or alloyed with the laser power range of 1.2-2.3 kW*

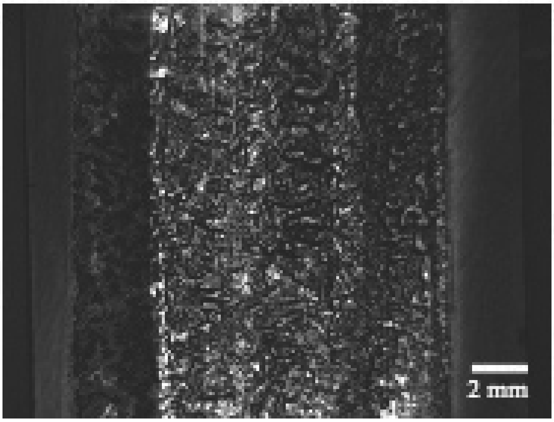

Figure 41. *Wear trace of the surface layer after an abrasion test acc. to ASTM G65 for X40CrMoV5-1 steel alloyed with WC tungsten powder, the laser power of 1.2 kW*

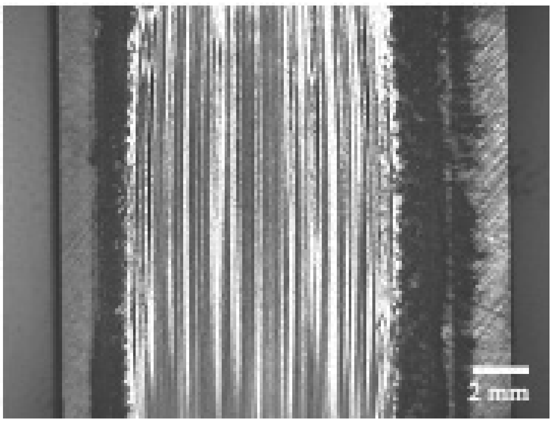

Figure 42. *Wear trace of the surface layer after an abrasion test acc. to ASTM G65 for 32CrMoV12-28 steel alloyed with TiC titanium powder, the laser power of 2.0 kW*

5. Technology roadmaps prepared for laser treated hot-work steels

On the basis of achieved results of experimental-comparative research a series of roadmaps of the analysed groups of technology were created. A representative technology roadmap prepared for the laser treatment of hot-work alloy tool steels using NbC niobium carbide powders in Table 7 is shown. The heading of the technology roadmap contains a basic

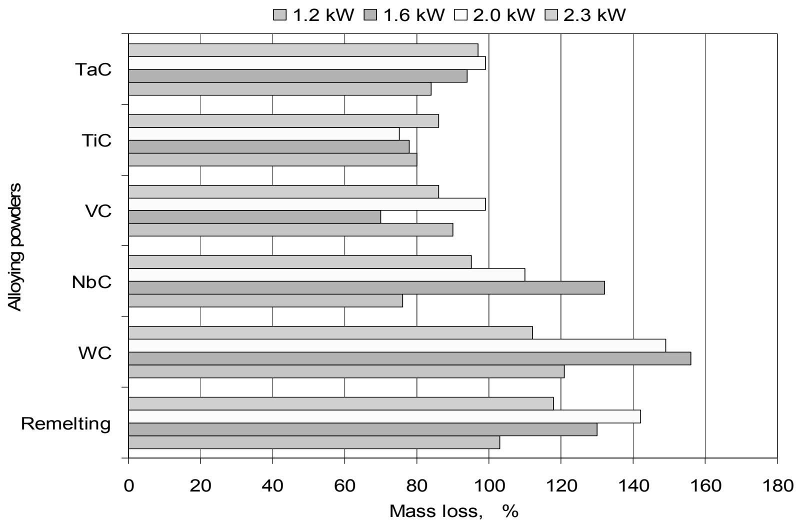

Figure 43. *The relative mass loss measured when testing wear resistance for X40CrMoV5-1 steel (100% – the mass loss of the heat-treated sample, not subjected to laser cladding or alloying with carbide powders)*

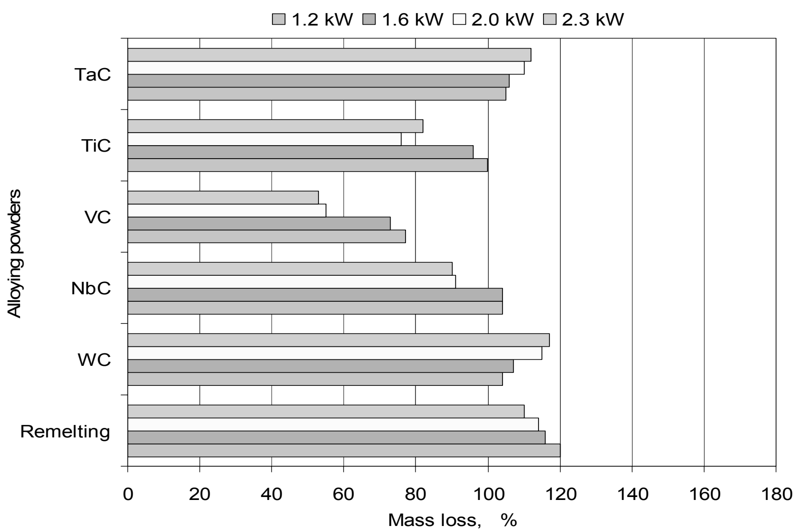

Figure 44. *The relative mass loss measured when testing wear resistance for 32CrMoV12-28 steel (100% – the mass loss of the heat-treated sample, not subjected to laser cladding or alloying with carbide powders)*

***Table 7**. Demonstrating technology roadmap for the laser treatment of hot-work alloy tool steels using NbC niobium carbide powders*

TECHNOLOGY ROADMAP		Technology name: *Laser treatment of hot-work alloy tool steels using NbC niobium carbide powders* Research scope: *Laser technologies in surface engineering*		Catalogue No: *M1-03-2010*
When?	Time intervals	**TODAY 2010**	**2020**	**2030**
	All-society and economic perspectives	Creating scenarios of future events	Development of priority innovation technologies	Statistically high quality of technologies
		Creating the Book of information cards concerning future technologies	Using chances and avoiding difficulties	Sustainable development
		Development of information society and intellectual capital	Cooperation to increase innovativeness and competitiveness of economy and intellectual	Knowledge-based economy
Why?	Strategy for technology	Strategy o an oak in summer		
	Environment influence	Hot summer		
	Technology value	Wide-stretching oak		
What?	**Product**	Casting moulds, stamping dies	Casting moulds, stamping dies, dies	Tools working in high temperature gradient conditions
	Product quality at the background of foreign competitors	Quite high (7)	High (8)	High (8)
	Surface	The following hot-work alloy tool steel grades: X40CrMoV5-1 and 32CrMoV12-28		
	Kind of surface coatings/layers	NbC niobium carbide		
	Improved material properties	Increase of mechanical and functional properties of elements, especially hardness; increase of resistance to heat fatigue and increase abrasive resistance		
	Diagnostic-research equipment	High-Power Diode Laser (HPDL), light, scanning electron and transmission electron microscopes, microhardness tester, screech tester, X-ray diffractometer, X-ray microanalyzer, a device for testing of heat fatigue and abrasive resistance		
How?	**Technology**	Alloying and/or cladding of hot-work alloy tool steels using NbC niobium carbide powders		
	Life cycle period	Embryonic (10)	Experimental (9)	Prototype (8)
	Production type	Unit scale	Medium-scale serial	Large-scale serial
	Production organisation form	Cellular	Cellular	Cellular rhythmic
	Machine park modernity	Excellent (10)	Excellent (10)	Excellent (10)
	Automation & robotisation	Quite high (7)	High (8)	Very high (9)
	Quality and reliability	Quite high (7)	High (8)	Qiute high (7)
	Proecology	High (8)	High (8)	Qiute high (7)
Where?	Organisation type	Research and scientific centres	Research and scientific centres, medium-sized enterprises	Large and medium-sized enterprises
	Represented industrial branches	Heavy and automotive industry	Heavy, automotive and machine-building industry	Automotive and machine-building industry
Who?	Staff education level	High (8)	High (8)	Quite high (7)
	Engagement of scientific-research staff	Very high (9)	High (8)	Quite high (7)
How much?	Capital requirements	Excellent (10)	Very high (8)	Moderate (6)
	Production size determining profitability in firm	Medium (5)	Quite high (7)	High (8)
	Production size in the country	Minimal (1)	Low (3)	Medium (5)

LEGEND: ⇒ Cause and effect connections; ········▶ Capital connections; ·······▶ Time corelations; ⟷ Two-way transfer of data and/or resources characteristic of the technology, including: the name of the technology, the represented research field and the given catalogue number. The horizontal axis of a roadmap corresponds to time intervals, and the vertical axis to seven layers called as following: time, conceptual, product, technological, spatial, staff and quantitative. This enables the setting, in one place, of data on the purpose, subject, manner, place, contractor and cost in relation to the analysed technology, taking into account the changes of these parameters in time. Upper layers placed in the top part of the technology roadmap, specifying general premises, causes and reasons of activity in the process of pulling, act on the fundamental middle layers pertaining to the product and technology. On the other hand, lower layers which specify the organisational-technical details act on the middle layers in an opposite direction, which is referred to in production management as pushing. Technology roadmaps are a very comfortable and practical tool of comparative analysis which facilitates the selection of technologies according to the selected criterion, and when supplemented by operation sheets with precise technological details – they enable the implementation of a given technology in industrial practice. A very large significance of technology roadmaps is their flexibility which enables their supplementing and expanding by new sub-layers depending on the arising needs. On the basis of data contained in given roadmaps prepared for all the analysed groups of laser treated hot-work alloy tool steels the summary presented in Table 8 was outworked.

Table 8. *Selected main source data used for preparation of roadmaps for laser treatment of hot-work steels using: (A) NbC niobium carbide, (B) TaC tantalum carbide, (C) TiC titanium carbide, (D) VC vanadium carbide and (E) WC tungsten carbide*

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
1.	Strategy for technology	2010	Strategy of an oak in summer	Strategy of an oak in summer	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in autumn
		2020	Strategy of an oak in autumn	Strategy of an oak in autumn	Strategy of an oak in summer	Strategy of an oak in summer	Strategy of an oak in autumn
		2030	Strategy of an oak in autumn	Strategy of an oak in autumn	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in autumn
2.	Environment influence	2010	Hot summer	Hot summer	Sunny spring	Sunny spring	Rainy autumn
3.	Technology value	2010	Wide-stretching oak	Wide-stretching oak	Wide-stretching oak	Wide-stretching oak	Wide-stretching oak

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
4.	Product	2010	Casting moulds, stamping dies	Blanking dies, stamping dies, dies	Forgings, dies, casting moulds	Dies	Stamping dies, forgings, dies
		2020	Casting moulds, stamping dies, dies	Blanking dies, stamping dies, dies	Plastic forming dies, punches, stamping dies	Dies	Stamping dies, forgings, dies
		2030	Tools working in high temperature gradient conditions	Stamping dies with long periods of use	Construction elements, biomaterials using other substrate materials	Dies, casting moulds	Stamping dies, forgings, dies
5.	Product quality at the background of foreign competitors	2010	Quite high (7)	High (8)	Moderate (6)	Moderate (6)	Moderate (6)
		2020	High (8)	High (8)	High (8)	High (8)	Quite high (7)
		2030	High (8)	High (8)	High (8)	High (8)	High (8)
6.	Improved material properties	2010-2030	Increase of mechanical and functional properties of elements, especially hardness; increase of resistance to heat fatigue and increase abrasive resistance				
7.	Diagnostic-research equipment	2010-2030	High-Power Diode Laser (HPDL), light, scanning electron and transmission electron microscopes, hardness tester, microhardness tester, screech tester, X-ray diffractometer, X-ray microanalyzer, a device for testing of heat fatigue and abrasive resistance, profilometer, potentiostat				
8.	Life cycle period	2010	Embryonic (10)	Embryonic (10)	Embryonic (10)	Embryonic (10)	Embryonic (10)
		2020	Experimental (9)	Experimental (9)	Experimental (9)	Prototype (8)	Prototype (8)
		2030	Prototype (8)	Growth (7)	Early mature (6)	Early mature (6)	Mature (5)
9.	Production type	2010	Unit scale	Unit scale	Unit scale	Unit scale	Unit scale
		2020	Medium-scale serial	Medium-scale serial	Small-scale serial	Medium-scale serial	Small-scale serial
		2030	Large-scale serial	Large-scale serial	Small-scale serial	Medium-scale serial	Large-scale serial
10.	Production organisation form	2010	Cellular	Cellular non-rhythmic	Cellar	Cellular	Cellular
		2020	Cellular	Cellular non-rhythmic	Cellular	Cellular non-rhythmic	Cellular
		2030	Cellular non-rhythmic	Cellular non-rhythmic	Cellular non-rhythmic	Cellular non-rhythmic	Cellular rhythmic

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
11.	Automation and robotisation	2010	Quite high (7)	Quite high (7)	Quite high (7)	Quite high (7)	Quite high (7)
		2020	High (8)	Very high (9)	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Excellent (10)	Very high (9)	Excellent (10)
12.	Quality and reliability	2010	Quite high (7)	Quite high (7)	Medium (5)	Medium (5)	Medium (5)
		2020	High (8)	Quite high (7)	Moderate (6)	Quite high (7)	Quite high (7)
		2030	Quite high (7)	Quite high (7)	High (8)	High (8)	Very high (9)
13.	Proecology	2010	High (8)	High (8)	Very high (9)	High (8)	High (8)
		2020	High (8)	Quite high (7)	Very high (9)	High (8)	High (8)
		2030	Quite high (7)	Quite high (7)	Very high (9)	High (8)	High (8)
14.	Organisation type	2010	Large and medium-sized enterprises, research and scientific centres, technological parks				
		2020	Large and medium-sized enterprises, research and scientific centres, technological parks				
		2030	Small and medium-sized enterprises, research and scientific centres, technological parks				
15.	Represented industrial branches	2010	Heavy and automotive industry	Heavy and automotive industry	Tool and heavy industry	Automotive industry	Tool and automotive industry
		2020	Heavy, automotive and machine-building industry	Automotive and machine-building industry	Automotive and light industry	Automotive industry	Tool and automotive industry
		2030	Automotive and machine-building industry	Automotive and machine-building industry	Tool, automotive, shipbuilding and biomedical industry	Automotive industry, fine mechanics elements	Automotive industry
16.	Staff education level	2010	High (8)	High (8)	Very high (9)	High (8)	High (8)
		2020	High (8)	High (8)	Quite high (7)	Quite high (7)	Quite high (7)
		2030	Quite high (7)	Moderate (6)	Quite low (4)	Medium (5)	Low (3)

No.	Analysed factor	Time interval	Analysed technology				
			A	B	C	D	E
17.	Engagement of scientific-research staff	2010	Very high (9)	Very high (9)	Very high (9)	Very high (9)	Very high (9)
		2020	High (8)	High (8)	High (8)	Moderate (6)	Medium (5)
		2030	Quite high (7)	Quite high (7)	Medium (5)	Quite low (4)	Quite low (4)
18.	Capital requirements	2010	Very high (9)	Very high (9)	High (8)	Quite high (7)	Medium (5)
		2020	Very high (9)	Very high (9)	Moderate (6)	Medium (5)	Quite low (4)
		2030	Excellent (10)	High (8)	Quite low (4)	Low (3)	Quite low (4)

6. Conclusions

The chapter presents the results of the interdisciplinary experimental-comparative research conducted mostly at the interface of material surface engineering and technology foresight. The purpose of the research was to determine the value of laser treatment of hot-work alloy tool steels compared to other technologies and to identify the recommended strategies and strategic development tracks for these technologies, taking into account the impact of such treatment on hardness, abrasion resistance and coarseness. The presented results of materials science research demonstrate a promising improvement of the functional properties of the tested materials. The melted zone (RZ), the heat-affected zone (HAZ), and the transition zone (TZ) were found in the surface layer; it was also found that laser cladding and/or alloying with carbide powders influences the refinement of the structure for both steel grades within the entire tested laser power range (1.2-2.3 kW). It was also concluded that the ceramic powders of oxides and nitrides do not melt into the tested steel's surface layer during alloying. A fine-crystalline, dendritic structure with the crystallisation direction connected with the dynamic evacuation of heat from the laser beam-affected zone occurs in the melted zone and/or alloyed zone. A high-quality surface layer with no cracks and defects and with its hardness much higher than the substrate material can be produced as a result of the heat treatment and laser cladding and/or alloying of X40CrMoV5-1 and 32CrMoV12-28 tool steel with ceramic powders using a high power laser. The properties of the tested steel and of the structural mechanisms occurring when producing the surface layers can be also thoroughly identified with the testes performed. The laser alloying of the tested steel with ceramic particles complicates, however, predictions as to how the material will behave in use. A very complex system, much more complicated than for hot-work steel unremelted with ceramic powders only, is created as the interaction of stress fields is overlapping with dislocation movements and the presence of microcracks. Improved resistance to abrasive wear and mechanical and tribological properties exhibited by the materials are achievable in particular through alloying with titanium carbide and vanadium carbide particles. Decisive for the further functional properties of the ready product is not only the appropriate selection of the ceramic powder used for alloying, but also its arrangement and volume fraction in the matrix modelled with different process operations. Clear growth in roughness and a higher bead face irregularity are seen when heightening laser power. The phenomenon is linked to the higher absorption of laser radiation by the sample surface. Higher energy absorption is also intensifying the process of cladding the steel surface layer. The heat fatigue resistance tests performed have a major effect on selecting the right ceramic powder for alloying hot-work steel, enabling to categorise the powders used according to their suitability for this type of laser treatment. To sum up one can conclude that titanium carbide TiC and vanadium carbide VC powders are most useful powders considering the improved heat fatigue resistance of both laser-alloyed steel grades. On the other hand, resistance is smaller if tantalum carbide and niobium carbide powders are used that produce alloyed surface layers with very high hardness and resistance to abrasive wear. The tests carried out point out that it is reasonable to use and apply in practise the technology of alloying X40CrMoV5-1 and 32CrMoV12-28 hot-work alloy tool steels grade using a high power diode laser. The final action closing the experimental-comparative works was to create a series of technology roadmaps of the analysed technology groups. The creation and use of this tool allows for presenting, in a uniform and clear format, various types of factors which directly and indirectly characterize given groups of technologies together with the forecast and perspectives of their development during the next twenty years.

A distinguishing characteristic of foresight research is that it looks, often very far, into the future. Therefore, it is reasonable to conclude by indicating the most state-of-the-art and highly promising directions of research which is currently conducted by the Institute of Engineering Materials and Biomaterials of the Silesian University of Technology. The research includes an effort to solve the problem of making a hybrid connection of the technology of sintering and convection cooling, with additional laser treatment, in order to produce sintered steels which are corrosion resistant and have surfaces without any porosity and characterised by improved corrosion resistance and mechanical properties [49]. This modern direction of studies is aimed at producing sintered stainless steel with its surface free of any roughness and offering higher corrosive resistance and mechanical properties. The final outcome of the proposed technology is to achieve a duplex structure in the surface layer on the substrate made of sintered ferritic single-phase steel and austenitic steel that will be characterised by improved anticorrosive properties, in particular improved resistance to pitting corrosion and a duplex structure with enhanced mechanical properties on the substrate made of sintered dual-phase steel characterised by improved corrosive resistance combined with strong grain refining resulting from fast crystallisation, which will additionally improve functional properties. In addition, the modern surface treatment methods combined with the controlled depth of laser beam's interaction and introducing additional alloying materials into the surface layer allows to achieve a gradient structure of the item, which is beneficial considering functional properties. Other state-of-the-art and promising works contain the effect of specific elements introduced into TRIP steel on its structure and properties is also undertaken. Complex hot-work tool steels should be characterised by their functional properties first of all (such as abrasive resistance and resistance to heat fatigue) exceeding other steel grades. Of note are also studies over hot-work tool steel properties are concerned with the impact of micro-additions, especially Ce, Zr and Ti on the progress of structural mechanisms and the properties of thermally-treated steel, with subsequent heat fatigue due to repetitive inductive heating and fast cooling. Studies have also been undertaken on the enrichment of surface of such steel by applying other types of lasers, including disc lasers.

Summing up, it should be underlined that the foresight- materials science research described in this chapter are a fragment of broader individual activity [1-3, 37-43, 50-53] aimed at selecting, researching, characterizing and determining strategic development perspectives of priority innovative material surface engineering technologies in the process of technological e-foresight understanding as computer aided scientific forecasting and shaping of the future in researched area.

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7 Technology validation of coatings deposition onto the brass substrate

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Abstract

Purpose: *The purpose of this chapter is to evaluate strategic development perspectives of manufacturing metallic-ceramic coatings in the process of physical vapour deposition (PVD) on the CuZn40Pb2 brass substrate. The amount of layers applied to the substrate was adopted as the criterion for technology division, thus obtaining three technology groups for foresight research.*

Design/methodology/approach: *The carried out foresight-materials science research included creating a dendrological matrix of technology value, a meteorological matrix of environment influence, a matrix of strategies for technologies, laying out strategic development tracks, carrying out materials science experiments which test the mechanical and tribological properties and the resistance to corrosion and erosion of brass covered with a varied number of layers applied using the method of reactive magnetron evaporation, as well as preparing technology roadmaps.*

Findings: *High potential and attractiveness were shown of the analysed technologies against the environment, as well as a promising improvement of mechanical and tribological properties and an increase of resistance to material corrosion and erosion as a result of covering with PVD coatings.*

Research limitations/implications: *Research pertaining to covering the brass substrate with PVD coatings is part of a bigger research project aimed at selecting, researching and characterizing priority innovative material surface engineering technologies.*

Practical implications: *The presented results of experimental materials science research prove the significant positive impact of covering with PVD coatings on the structure and mechanical* *properties, as well as the resistance to corrosion, erosion and abrasive wear of brass which leads to the justification of their including into the set of priority innovative technologies recommended for application in industrial practice, including in small and medium-size companies.*

Originality/value: *The advantage of the chapter is the specification of the significance of the technology involving covering the brass substrate with mono- and multilayer PVD coatings against the environment, together with the recommended strategies of conduct, strategic development tracks and roadmaps of these technologies, taking into account the impact of the processes of applying these coatings onto the structure and the improvement of the properties of the tested surface layers.*

Keywords: *Manufacturing and processing; Thin & Thick Coatings; Brass substrate; Foresight; Technology Roadmapping*

This chapter has been also published as:

A.D. Dobrzańska-Danikiewicz, K. Lukaszkowicz, Technology validation of coatings deposition onto the brass substrate, Archives of Materials Science Engineering 46/1 (2010) 5-38.

1. Introduction

In accordance with the definition of the Organization for Economic Co-Operation and Development (OECD), knowledge-based economy is based on creating, distribution and the practical application of knowledge and information [1]. This economy promotes companies, including small and medium-size ones, which are innovative, educational and informational-communicative systems, consciously managing knowledge as a strategic resource, taking into account the impact of the micro- and macroenvironment. The innovation of the system is expressed in a constant search and promotion of new technologies in all areas of the company's functioning in order to obtain a specific profit. A company which is an educational system puts emphasis especially on the acquisition and education of employees hired in the research and development field (R+D). On the other hand, the information-communicative system constitutes a basis for providing knowledge to employees by creating safe IT networks and systems and by communicating with the state and European administration. Innovation, education and an effective flow of information at the company level are the main building blocks of an economy based on knowledge and economy competitiveness on a country scale. Decisive for the development of a knowledge-based economy is the development of those economy sectors which are directly related to the development of science and the processing of information as part of the so-called high technology (hi-tech). In this context it seems critical to direct scientific research to the most promising scientific fields and branches which may have large influence on the quick civilisation-economic development of the country based on an IT community. Moreover, attention should be drawn to providing the possibility of a rational practical use of the conducted studies and of creating budgetary preferences for them. The realisation of such defined goals and targets is possible with the use of the e-foresight methodology. E-foresight involves conducting foresight research aimed at selecting priority innovative technologies and strategic development directions for the research field, with the use of the Internet [2], referring to already known and commonly used notions [3, 4] of e-management, e-business, e-trade, e-banking, e-logistics, e-services, e-administration and e-education which always mean conducting specified activities with the use of computer networks. The proposed approach uses the synergy effect and eliminates the unfavourable psychosocial phenomenon called the show-off effect, meaning that during a direct meeting which serves the exchange of views on a specific subject, people are mainly directed at presenting themselves in the best light possible, and not at sharing their knowledge. For the realisation of technological e-foresight, the Computer Aided Foresight Integrated Research Management (CA FIRM) methodology was created [2, 5-8]. This methodology which organizes, improves and modernizes the actual process of foresight research, may be used in practice thanks to working out a concept of functioning in cyber-reality – the Virtual Organisation for Foresight Integrated Research Management (VO FIRM). The following IT tools enable the realisation of such defined goals and targets from the technical angle: the Web Platform for Foresight Integrated Research Management (WP FIRM) and the Neural Networks for Foresight Integrated Research Management (NN FIRM).

The challenges lying ahead of the contemporary economy necessitate reductions in energy consumption and material consumption as a prerequisite for sustainable development and reasonable natural resources management. In the majority of cases the goals are achievable though replacing the traditional materials with those having higher proper strength or better functional properties. A modern approach targeted at matching the material to the construction, not the construction to the material, requires materials manufacturing to be based on the knowledge of materials. The approach also makes it necessary to associate flexibly and skilfully many technological operations (including the surface layer modification technology) for their production to accomplish the intended outcome, i.e. the material having properties necessary for the optimum operation of the designed construction. The functional properties of many products depend not only on the possibility of transmitting mechanical loads through the entire active section of the element made of the material applied or on its physiochemical properties, but very often on the structure and properties of surface layers [9-19]. The products used in the construction, automotive and electronic industry should feature, apart from special aesthetic properties and colour, also high corrosion, erosion and abrasion resistance. Many parts of sanitary fittings, fixtures, builder's hardware are made traditionally of copper and zinc alloy that is cast or worked plastically and frequently surface-plated with electroplating methods, most often nickel and chromium. This poses a major ecological hazard for the environment and people manufacturing such parts. For this reason, other materials are being sought for that could live up to the expectations connected with good functional properties and an environmentally-pure manufacturing technology. Copper and zinc alloys turn out to be still widespread because of their good castability and workability. High requirements concerning properties make it necessary to use other environmentally clean methods offering an opportunity of greater colour differentiation for coatings and more advantageous useful properties [20-37]. The intensive development of issues related to the widely-understood concept of surface engineering can be seen nowadays. The modern surface engineering technologies enable to improve economically the quality and properties of many parts exhibiting greatly enhanced strength under operating conditions than if they had been made entirely of expensive, high-durability materials. Progress in production and in the improvement of operating durability of structural parts and tools used in the different areas of life is achieved as the techniques of depositing thin coatings made of hard ceramic materials resistant to wear are becoming more and more common. A wide selection of the types of coatings and deposition technologies currently available derives from a growing demand in the recent years for the state-of-art material surface modification and protection methods [28-79]. From among a myriad of techniques enhancing the strength of materials, the PVD (Physical Vapour Deposition) methods are enjoying an increasing popularity in industrial practise [80-86]. Actually, PVD coatings are one from the most interesting and intensively developed technologies of protection and modification of product surface. It takes pace, because they give possibility of creation of materials with unique physiochemical properties, such as: extremely high hardness [87-89], high corrosion resistance [90, 91], high oxidation resistance in high temperature [92, 93], as well as high resistance to abrasive and erosion wear [94-96]. Thin, hard PVD coatings on a soft substrate prove to be a beneficial material combination from the tribological perspective. They can be employed in particular for abrasive or erosive destruction by improving resistance to scratches or cracks formed in contact with hard materials. The only limitation for using hard coatings on a soft base are high stresses formed in the coatings themselves and at the substrate material – coating interface.

The favourable properties of copper and zinc alloys, together with the advantages of physical vapour deposition constituted the basis for performing a series of interdisciplinary foresight-materials science research in order to specify the value, attractiveness and potential of the technology of applying hard PVD coatings on the soft brass substrate against the micro- and macroenvironment. The carried out research involved also working out recommended strategies of conduct, setting strategic development tracks and preparing technology roadmaps of analysed technology groups, with special consideration to mechanical and tribological properties and the resistance to corrosion of a material covered with a varied number of layers applied to the brass substrate using the PVD technology. Experimental research were performed onto the CuZn40Pb2 brass substrate, to which layers of Ti/CrN, Ti/TiAlN, and Mo/TiAlN were applied, under suitable pressure in the amount of one, fifteen and one hundred and fifty, respectively. The research of coating microstructures was performed using a metallographic, as well as a scanning and transmission electron microscope. The exploitation properties of the created coatings were determined based on an erosion test. Tests of the electrochemical corrosion of coatings were performed in a tri-electrode chamber in a 1-molar solution of HCl.

Foresight-materials science research carried out as part of this chapter constitute a fragment of broader individual actions aimed at selecting a set of priority innovative technologies of material surface engineering. The overriding aim of these large-scale research is to generate a set of priority innovative surface engineering technologies which contribute to the statistical quality increase of technologies applied in industrial companies, stimulating sustainable development and strengthening knowledge-based economy.

2. Research methodology

The conducted research are interdisciplinary and the used researching methodology pertains mainly to technology foresight [97] being an element of a field called organisation and management and to surface engineering included in a more broadly understood material engineering. At certain stages of the conducted studies, also methods were used which come from artificial intelligence, statistics, IT technology, construction and exploitation of machines, as well as strategic [98], operational [99] and quality [100] management.

According to the adopted methodology, the carried out research include: selecting technology groups for experimental-comparative research, collecting expert opinions, carrying out a multi-criteria analysis and marking its results on the dendrological and meteorological matrix, determining strategies for technologies preceded by rescaling and objectivising research results using formulated mathematical relations, setting strategic development tracks for technologies, carrying out a series of specialist materials science experiments in experienced team using a specialist diagnostic-measuring apparatus and the creation of technology roadmaps. In accordance with the applied methodology of foresight-materials science research, several possibilities of homogenous groups should be singled out from the analysed technologies in order to subject them to planned experimental-comparative nature research. To determine the objective values of given selected technologies or their groups a dendrological matrix of value technology is used, and to determine the strength of positive and negative influence of the environment on a given technology a meteorological matrix of environment influence is used. The methodological construction of those both matrices refers to portfolio methods, commonly known in sciences about management, and first of all to BCG matrix [101]. For the purpose of evaluating technology groups with regard to their values and environmental influence, a ten-point universal scale of relative states was adopted, in which the smallest value 1 corresponds to a minimum level, and the highest value 10 is the level of perfection.

The dendrological matrix of technology value [5] presents graphic results of evaluating specific technology groups, with special attention paid to the potential constituting the real objective value of a given technology and to the attractiveness reflecting how a given technology is subjectively perceived among its potential users. The potential of a given technology group expressed through a ten-point universal scale of relative states, marked on the horizontal scale of the dendrological matrix is the result of a multi-criteria analysis carried out based on an expert opinions. On the vertical scale of the dendrological matrix the level of attractiveness was marked of a given technology group which is the mean weighed expert opinions based on detailed criteria. Depending on the type of potential and level of attractiveness determined as part of the expert opinions, a given technology may be placed in one of the quarters of the matrix. The quarters distinguished in the dendrological matrix of technology value are presented in Table 1.

Table 1. *The quarters of the dendrological matrix of technology value*

Factors		Potential	
		Low	High
Attractiveness	High	A **quaking cypress** which is technology with a limited potential, but highly attractive, what causes that a success of technology is possible	A **wide-stretching oak** which corresponds to the best possible situation in which the analysed technology has both a huge potential and huge attractiveness, which is a guarantee of a future success
	Low	A **sparing aspen** which is technology with a limited potential and limited attractiveness in the range, which a future success is unlikely	A **rooted dwarf mountain pine** which is technology with limited attractiveness, but a high potential, so that its future success is possible

The meteorological matrix of environment influence [5] presents graphic results of evaluating the impact of external factors on specific groups of technologies which had been divided into difficulties with a negative impact and chances which positively influence the analysed technologies. The researching of expert opinions on the subject of positive and negative factors which influence specific technologies was carried out based on a survey comprising several dozens of questions pertaining to the micro- and macroenvironment in strictly defined proportions. External difficulties expressed with the use of a ten-point universal scale of relative states (from 1 to 10), which are the result of a multi-criteria analysis conducted based on the expert opinions, have been placed on the horizontal scale of the meteorological matrix. On the other hand, chances, i.e. positive environment factors being a mean weighed expert opinions based on detailed criteria, were placed on the vertical scale. Depending on the level of influence of positive and negative environment factors on the analysed technology, determined as part of the expert opinions on a ten-point scale, it is placed in one of the matrix quarters. The quarters distinguished in the meteorological matrix of environment influence are presented in Table 2.

Table 2. *The quarters of the meteorological matrix of environment influence*

Factors		Difficulties	
		A small number	A large number
Chances	A large number	**Sunny spring** being the best option denoting friendly environment with lots of opportunities and a little number of difficulties, which means that the success of given technology is guaranteed	**Hot summer** corresponding to a situation in which the environment brings a lot of opportunities, which, however, are accompanied by many difficulties, meaning that the success of technology in the given circumstances is possible, but is a subject to the risk
	A small number	**Rainy autumn** corresponding to the neutral position, in which for given technology traps do not wait, but also the environment does not give too many opportunities	**Frosty winter** corresponding to the worst possible situation in which the environment brings a large number of problems and few opportunities, which means that success in a given environment is difficult or impossible to achieve

The research results presented in a graphical form using a dendrological matrix of technology value and a meteorological matrix of environment influence were put on a **matrix of strategy for technologies** consisting of sixteen fields corresponding to each set of versions resulting from the combination of the types of technology and the types of environments. To facilitate the transfer of specific numeric values from the dendrological matrix [2x2] and the meteorological matrix [2x2] to the matrix of strategies for technologies with the dimensions of [4x4], mathematical relations were formulated which enable the rescaling and objectivising of research results and, based on them, a short computer program was created to enable a quick calculation of the searched values and their placing on the chart. Thus, the following notions were introduced: the relative value of technology V_n and the relative value of environment influence E_n and mathematical dependence allowing to graduate and make objective research results were introduced [5, 8].

The strategic development tracks for different technologies/ groups of technologies in the next step of research were outworked. These strategic development tracks forecast given technology development successively in: 2015, 2020, 2025 and 2030 in three versions: optimistic, pessimistic and most possible ones, followed by their visualisation against a back-ground of a matrix of strategy for technology.

In order to precise the value of the potential and attractiveness of PVD coatings deposited onto the brass substrate **a series of materials science research** using specialised diagnostic and measurement equipment were carried out. The research were made on CuZn40Pb2 copper-zinc alloy samples plated with hard coatings in the PVD process with the chemical composition presented in Table 3. The copper-zinc alloy samples were subjected to mechanical grinding and polishing to ensure the appropriate quality of the sample surface. The methods commonly used in the process of preparing metallographic specimens using Struers equipment were applied during polishing. A diamond abradant with a varied grain size ending with a 1 µm grain was used. The samples, immediately prior to the coating deposition process, were cleaned chemically using a multi-stage washing and rinsing process in washing and degreasing baths, and then they were ion-etched in the chamber of the coating deposition equipment in a pure argon atmosphere in order to clean the coated surfaces and to activate them for 20 min. The 200×100×6 mm water-cooled discs containing pure metals (Cr, Ti, Mo, Zr) and 50% Ti – 50% Al alloys being the substrates of the phases deposited on the charge were used for applying coatings. Current density for both megatons was determined approximately as 0.01 A/cm^2. The coatings were deposited in the atmosphere of inert gas (argon) or/and reactive gas (nitride) being supplied continuously to the working chamber. The distance between each of the discs and the coated samples is 65 mm. The type of coating, current and voltage conditions and the values of pressures prevailing in the equipment chamber during the coating deposition process are presented in Table 4.

Table 3. *Chemical composition of the CuZn40Pb2*

Type	Chemical composition, %							
	Alloy components			Allowable concentration of pollutants				
	Cu	Pb	Zn	Fe	Sn	Al	Ni	other
CuZn40Pb2	56.0-60.0	1.0-3.5	rest	0.5	0.5	0.1	0.5	0.2

Table 4. *Deposition parameters of the coating*

Coating	Substrate bias voltage, V	Working pressure, Pa	Partial pressure, Pa		Number of layers
			nitrogen	argon	
Ti/CrN×1	–50	0.58	0^a. 0.15^b	0.31	1
Ti/CrN×15		0.39	0^a. 0.15^b	0.31	15
Ti/CrN×150		0.46	0^a. 0.15^b	0.31	150
Ti/ZrN×1	– 50	0.34	0^a. 0.10^b	0.29	1
Ti/ZrN×15		0.29	0^a. 0.10^b	0.29	15
Ti/ZrN×150		0.31	0^a. 0.10^b	0.29	150
Ti/TiAlN×1	– 40	0.40	0^a. 0.10^b	0.38	1
Ti/TiAlN×15		0.41	0^a. 0.10^b	0.38	15
Ti/TiAlN×150		0.41	0^a. 0.10^b	0.38	150
Mo/TiAlN×1	– 60	0.49	0^a. 0.11^b	0.45	1
Mo/TiAlN×15		0.46	0^a. 0.11^b	0.45	15
Mo/TiAlN×150		0.50	0^a. 0.11^b	0.45	150

[a] during metallic layers deposition
[b] during ceramic layers deposition
During deposition the substrate temperature was always 300˚C

The metallographic research were carried out with an MEF4A Leica metallographic microscope using a Leica-Qwin computer-aided image analysis system on copper-zinc samples with coatings deposited on their surface. The specimens were prepared using Struers equipment and then etched in an aqueous iron chloride solution (10 g of iron chloride, 30 ml of hydrochloric acid, 100 ml of distilled water) to develop the structure. The structure of the samples produced was observed at lateral fractures with an XL-30 scanning electron microscope by Philips. Secondary electrons detection was used for creating the images of the fractures with the accelerating voltage of 20 kV. The samples with the cut notch were cooled in liquid nitride before breaking to eliminate a plastic deformation and ensure the brittle character of the fracture being created. The phase composition of the researched coatings was determined using a Dron 2.0 diffractometer, and filtered $K\alpha_1$ X-ray radiation was used for stepwise recording with the wave length of λ = 1.79021 nm coming from a lamp with a 35 kV cobalt tube with 8 mA filament current intensity. The measurement was made within the angle range of 2θ within 35 to 100°. An X-ray XRD7 Seifert-FPM diffractometer fitted with a texture attachment was used for evaluating the texture of coatings. The X-ray radiation of a 35 kV Co Kα cobalt tube with 40 mV current intensity was used. The texture of the researched coatings was assessed with the inverse pole figures method.

Internal stresses within the coatings were assessed with the spacing of reflections coming from the planes of crystallographic lattices of the phases forming part of the coatings produced on X-ray diffraction photographs and the Young's modulus values for the respective coatings. Internal stresses σ were determined using the following equation:

$$\sigma = -\frac{E}{2\nu} \cdot \frac{d - d_o}{d_o} \qquad (1)$$

where:

E –Young's modulus,

ν – Poisson's constant,

d – lattice parameter with internal stresses determined with an X-ray diffraction photograph,

d_o – lattice parameter without internal stresses (table value).

The thickness of the coatings produced was measured with a "kalotest" method consisting of measuring the characteristic sizes of a crater formed on the surface of the researched sample with the coating. The measurements were made with a custom-designed device. In addition, to verify the results obtained, the depth of the coatings was measured with a scanning electron microscope at the lateral fractures to their free surface.

The qualitative and quantitative X-ray micro-analysis and the surface distribution analysis of alloy elements in the samples and of the coatings deposited onto their surface was performed at the lateral fractures with a JEOL JCXA 733 X-ray microanalyser with an EDS LINK ISIS X-ray scattered radiation spectrometer by Oxford with the accelerating voltage of 20 kV.

Variations in the chemical concentration of the coating components in the perpendicular direction to the coating surface and concentration changes in the transient zone between the coating and the substrate material were evaluated based on research with a GDOS-75 QDP glow discharge optical spectrometer by Leco Instruments. The following working conditions of the spectrometer's Grimm lamp were determined in the research:

- inner lamp diameter – 4 mm;
- lamp supply voltage – 700 V;
- lamp current – 20 mA;
- working pressure – 100 Pa;
- analysis duration – 400 s.

A Paschen–Runge continuous simultaneous spectrometer with the focal point of 750 mm and the holographic lattice with 2400 lines per millimetre was used in this device. The maximum depth of the chemical composition analysis is 10 μm.

The hardness tests of the deposited coatings hardness were conducted with the Vickers method consisting of measuring the depth of indention that usually does not exceed the decimals of micrometre, and the set pressure does not exceed 0.05 N, which eliminates the impact of the substrate material on the hardness of the coating. The hardness test with the Vickers method was performed with nano-indenting made with the Shimadzu DUH 202 nanohardness tester.

Rigidity S after unloading the sample was calculated to determine Young's modulus using Hardness 4.2 software bundled with the DUH 202 nanohardness tester according to the following formula:

$$S = \frac{dP}{dh} = \beta \cdot \frac{2}{\sqrt{\pi}} \cdot E_r \cdot \sqrt{A_k} \qquad (2)$$

where:

β – the constant resulting from the indenter geometry;

E_r – reduced Young's modulus, kN/mm^2;

A_k – contact area, μm^2.

and a reduced Young's modulus according to the formula:

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu_s^2}{E_s} \qquad (3)$$

where:

E_i – Young's modulus for indenter, kN/mm^2;

E_s – Young's modulus for sample, kN/mm^2;

ν_i – Poisson constant for indenter;

ν_s – Poisson constant for sample.

The adhesion of the coatings to the substrate material was examined with a scratch test used commonly for coatings produced in the processes of physical deposition from the gas phase. The tests were made with a computer-controlled device – Sebastian 5A (Quad Group) fitted with an acoustic detector under the following test conditions:

- load increase rate (dL/dt) – 100 N/min;
- indenter movement rate (dx/dt) – 10 mm/min.

The smallest force at which the coating is damaged, referred to as the critical load L_{C2}, was determined based on the decrease of the acoustic emission value recorded during the measurement and formed at the indenter – tested sample interface. The character of the damage formed was assessed based on observations with a DSM-40 scanning electron microscope by Opton and with a MEF 4A Leica light microscope.

Surface roughness for the polished samples without coatings and with coatings was measured in two mutually perpendicular directions with a Surftec 3+ profilometer by RankTaylor Hobson. The measurement length is l = 0.25 mm, and the measurement accuracy 0.01. The R_a parameter acc. to PN – EN ISO 4287 was adopted as a value describing surface roughness.

Abrasive wear resistance tests with the pin-on-disc method were carried out with the CSEM High Temperature Tribometer. A 6 mm Al_2O_3 ball was used as a counter-sample. The tests were made at a room temperature under the following test conditions:

- pressure force F_N – 5 N;
- movement speed v – 40 cm/s;
- radius r – 10 mm.

A friction coefficient for the researched coatings was determined with a CSEM High Temperature Tribometer. A 100Cr6 steel penetrator with the rounding diameter of 1 mm was used as a counter-sample. The research were made at a room temperature under the following test conditions:

- pressure force F_N – 1 N,
- friction path s – 10 mm,
- movement speed v – 10 mm/s.

The operating properties of the coatings produced were determined with an erosion test with the Falex Air Jet Eroder by Falex Corporation, representing the air jet type devices, where the powder erodent leaving the nozzle at the set pressure is impacting the tested sample surface positioned at the set angle against the nozzle. The tests were carried out under the following conditions:

- nozzle pressure – 270 kPa;
- impact angle – the angle between the sample surface and the nozzle – 90°;
- erodent flow rate – 2 g/min.;
- distance between the sample surface and the nozzle – 20 mm;
- minimum test duration – 0.1 s.

Powder was used as an erodent with a commercial name Dynablast™ manufactured by Norton company with the following components: Al_2O_3 (95.8%), TiO_2 (2.6%), SiO_2 (1%), Fe_2O_3 (0.2%), MgO (0.2%), ZrO_2 (0.1%), other 0.1% being alkali. The average erodent grain size is 70 mm, and Knoop hardness is 21.6 GPa. An additional EDS X-ray analysis was made at 0.1 s intervals (0.2 s for some coatings) to identify an erosion rate to determine if the lines representing alloy elements forming part of the substrate are present in the X-ray radiation energy spectrum produced coming from the craters formed. If such lines appear, this means that the coating is damaged. Besides, a Superprobe 733 electron scanning microscope by JEOL coupled with a computer image analyser was used to evaluate the degree of coating perforation caused by the powder erodent. The perforation degree was evaluated in such a way that the size of the exposed substrate area during the elementary research step was determined or its multiplication was determined using the natural difference between the coating and substrate colour exposed with specific magnification constant for all the samples covering the entire crater area considered as 100%. Erosion resistance is higher the smaller is the share of the coating removed within the set test time.

The research of electrochemical corrosion of the coatings applied were made with a standard laboratory device for the quantitative corrosion test of material properties – a three-electrode chamber in a 1-mole HCl solution with regard to a platinum electrode and calomel electrode. The tests were made with a PGP 201 Potentiostat/Galvanostat device. The following tests were made:

- polarisation tests within the range of -500 mV to 500 mV with the scanning speed of 15 mV/min. to determine corrosion current i_{cor} on the substrate with Tafel's analysis method;
- measurements of corrosion potential E_{cor} after 60 min. of the experiment's progress;
- corrosion speed measurements:

$$v_{cor} = \frac{i_{cor} \cdot M}{\rho \cdot W} \tag{4}$$

where:

v_{cor} – corrosion speed, mm/year;

i_{cor} – current density, A/cm^2;

M – atomic mass, g;

ρ – density, g/cm^3;

W – valence (the electrons lost during the reaction).

The results of the carried out experimental-comparative research constitute source data which serve for creating **technology roadmaps**. The layout of the technology roadmap created for the purpose of the realised research corresponds to the first quarter of the Cartesian coordinate system. Three time intervals were placed on the horizontal axis, pertaining to: the situation as of today (year 2010), in ten years' (in 2020) and in twenty years' time (in 2030). The time horizon of all the research placed on the technology roadmap equals 20 years and is adequate to the dynamics of changes occurring in the surface engineering. On the vertical axis of the technology roadmap seven main layers were placed corresponding to a specific question pertaining to the analysed scope. Each of the main layers has been additionally divided into more detailed sub-layers. The main layers of the technology roadmap were organised in a hierarchical way. The upper part of the technology roadmap contains the most general layers specifying the premises, reasons and causes of realised research which influence the layers placed under them in the process of „pull". The middle part of the technology roadmap pertains to the essence of the analysed problem by characterizing the product and technology used for its manufacturing. The lowest layers of the technology roadmap contain various details of the technical-organisational nature which influence the higher-located layers in the process of „push". In addition, the technology roadmap presents relations between its specific layers and sub-layers, with a division into: cause-and-effect relations, capital relations, time correlations and two-way flows of data and/or resources, visualised using different types of arrows. The technology roadmap is a universal tool which enables presenting, in a unified and clear format, different types of internal and external factors directly and indirectly characterizing a given technology, taking into account the ways of influence, interdependencies and the change of specific factors over time. When needed, the technology roadmap may be supplemented and expanded by additional sub-layers, adapting it, e.g. to the specificity of the carried out scientific-research studies, the requirements of a given industrial field or the size of a company.

This chapter presents results of research which include especially the evaluation of the potential and attractiveness of the analysed technologies against the micro- and macroenvironment. This evaluation was performed based on the opinions of key experts expressed on a ten-point universal scale of relative states; next, a recommended strategy was formulated of conduct with a given technology, together with the anticipated strategic development tracks (sub-chapter 3). Sub-chapter 4 of the chapter contains the results of materials science research which test the microstructure, phase composition and texture, erosion resistance and tribological properties, as well as the resistance to corrosion of monolayer and multilayer coatings applied to the CuZn40Pb2 brass substrate via the PVD technology using the reactive magnetron evaporation method. Based on the results of conducted experimental-comparative research, technology roadmaps were created which present, in a unified and clear format, different types of internal and external factors that directly and indirectly characterize the specific technologies, taking into account the manners of influence, interconnections and the change of specific factors over time, which was presented in sub-chapter 5 of the chapter.

3. Determined technology values and strategic development tracks

Adopting as the division criterion the number of layers which compose the analysed PVD coating, three homogenous groups were selected among the analysed technologies in order to conduct experimental-comparative works. They include:

(A) The production of metallic/ceramic monolayer coatings by means of a physical vapour deposition process onto the CuZn40Pb2 brass substrate,

(B) The production of metallic/ceramic multilayer (in the amount 15) coatings by means of a physical vapour deposition process onto the CuZn40Pb2 brass substrate,

(C) The production of metallic/ceramic multilayer (in the amount 150) coatings by means of a physical vapour deposition process onto the CuZn40Pb2 brass substrate.

The analysed technology groups were evaluated by key experts in terms of their attractiveness and potential, using a ten-point universal scale of relative states. Using a multi-criteria analysis, the mean weighed value was calculated from the analysed detailed criteria selected as part of the attractiveness and potential, and the result obtained for specific technology groups was placed on the dendrological matrix of technology value (Fig. 1). As a result of the carried out analysis, all technology groups were qualified to the most promising quarter of the matrix – the wide-stretching oak which includes technologies of a high potential and large attractiveness. The best result was obtained by the technology group C (7.50, 8.50), involving covering with multilayer (in the amount 150) PVD coatings; a slightly worse result was obtained by the technology group A (7.00, 8.00), involving covering with monolayer PVD coatings; the worst result was obtained by the technology group B (6.60, 7.60), involving covering with multilayer (in the amount 15) PVD coatings.

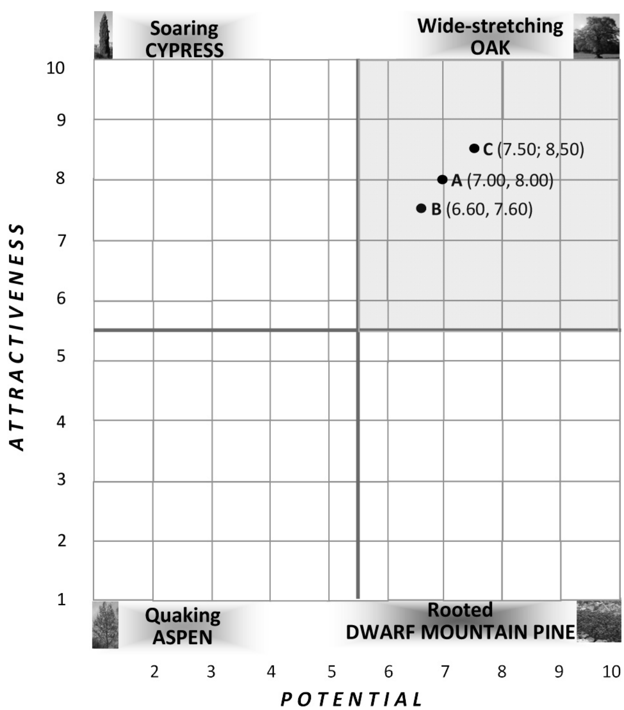


Figure 1. The dendrological matrix of technology value for production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate with: (A) monolayer, (B) fifteen layers, (C) one hundred fifty layers

The meteorological matrix of environment influence is a tool which serves the positive and negative evaluation of environmental impact on the specific technology groups. The results of the multi-criteria analysis performed on the expert opinions obtained during surveying were charted onto the meteorological matrix (Fig. 2). The survey used for conducting research contains several dozens of questions pertaining to the power of positive and negative influence of the micro- and macroenvironment on technologies in strictly determined proportions. The conducted research indicates that in the case of all technology groups subjected to research, the environment is very favourable, bringing many chances and a small amount of difficulties. An illustration of such a state of things is placing all the analysed technology groups in the quarter corresponding to sunny spring, which bodes well for their development. Again, the highest mark was obtained by the technology group marked as C (3.52, 7.42); a slightly lower mark was obtained by technology group A (3.97, 7.22), while the lowest mark – by technology group B (4.36, 6.35).

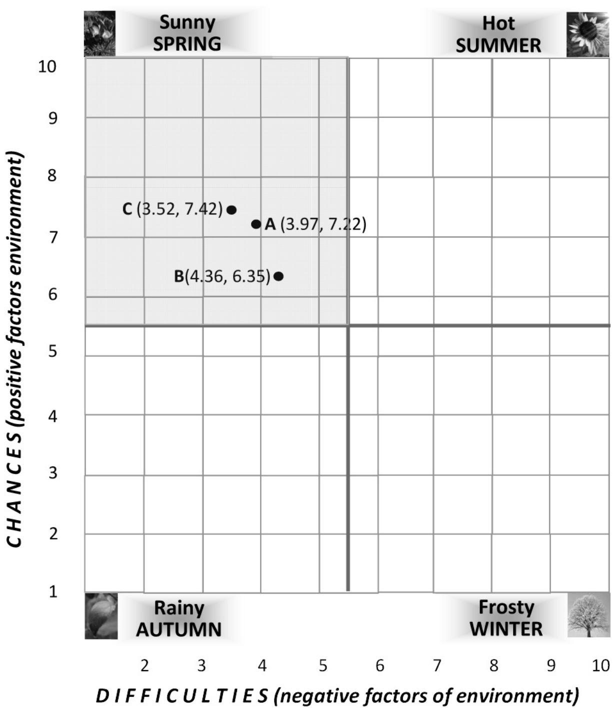


Figure 2. *The meteorological matrix of environment influence for production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate with: (A) monolayer, (B) fifteen layers, (C) one hundred fifty layers*

At the next stage of research works the results of research presented in graphic form using the dendrological matrix of technology value and the meteorological matrix of environment influence have been placed on the matrix of strategies for technologies. (Fig. 3). This matrix shows the graphical location of specific groups of technologies of applying coatings with a varied number of layers onto the brass substrate using the reactive and magnetron evaporation, taking into account their value and impact on the environment, indicating a suitable strategy of conduct. Transferring specific numeric values from the dendrological meteorological matrices onto the matrix of strategies for technologies, of different dimensions, took place using the formulated mathematical dependencies and a simple computer program based on them which allowed for rescaling and objectivizing the research results […]. In the case of all the analysed well-promising technology groups, the use of the strategy of oak in spring is recommended. This strategy involves developing, strengthening and implementing an attractive technology of a high potential in industrial practice in order to achieve spectacular success.

The next stage of research involves the specification of strategic development tracks, based on expert opinions, for specific technologies/technology groups, constituting their development forecast in 2015, 2020, 2025 and 2030 in three variants: optimistic, pessimistic and the most probable, and then visualizing them against a matrix of strategies for technologies. The representative graphic example of a matrix of strategies for technologies with charted strategic development tracks in three variants for covering the brass substrate with multilayer (in the amount of 150) PVD coatings was presented in Figure 4. The most probable strategic development track for this technology group assumes the change of environmental conditions from friendly spring to risky summer, with a simultaneous maintenance of the technology's high attractiveness and strengthening an already high potential, characteristic for a wide-stretching oak. It is anticipated that in the next years the negative environment factors will be slowly neutralised and the analysed technology group will again enter the field of oak in spring, for which the suitable conduct is the development, strengthening and implementation of an attractive technology with a high potential in industrial practice in order to achieve spectacular success.

The optimistic development track of the technology of applying multilayer (in the amount of 150) PVD coatings on the brass substrate assumes that, despite a temporary (in 2015-2020) appearance of numerous difficulties in the environment, it will be possible to make use of the simultaneously appearing chances and that they will, in the future, determine the development

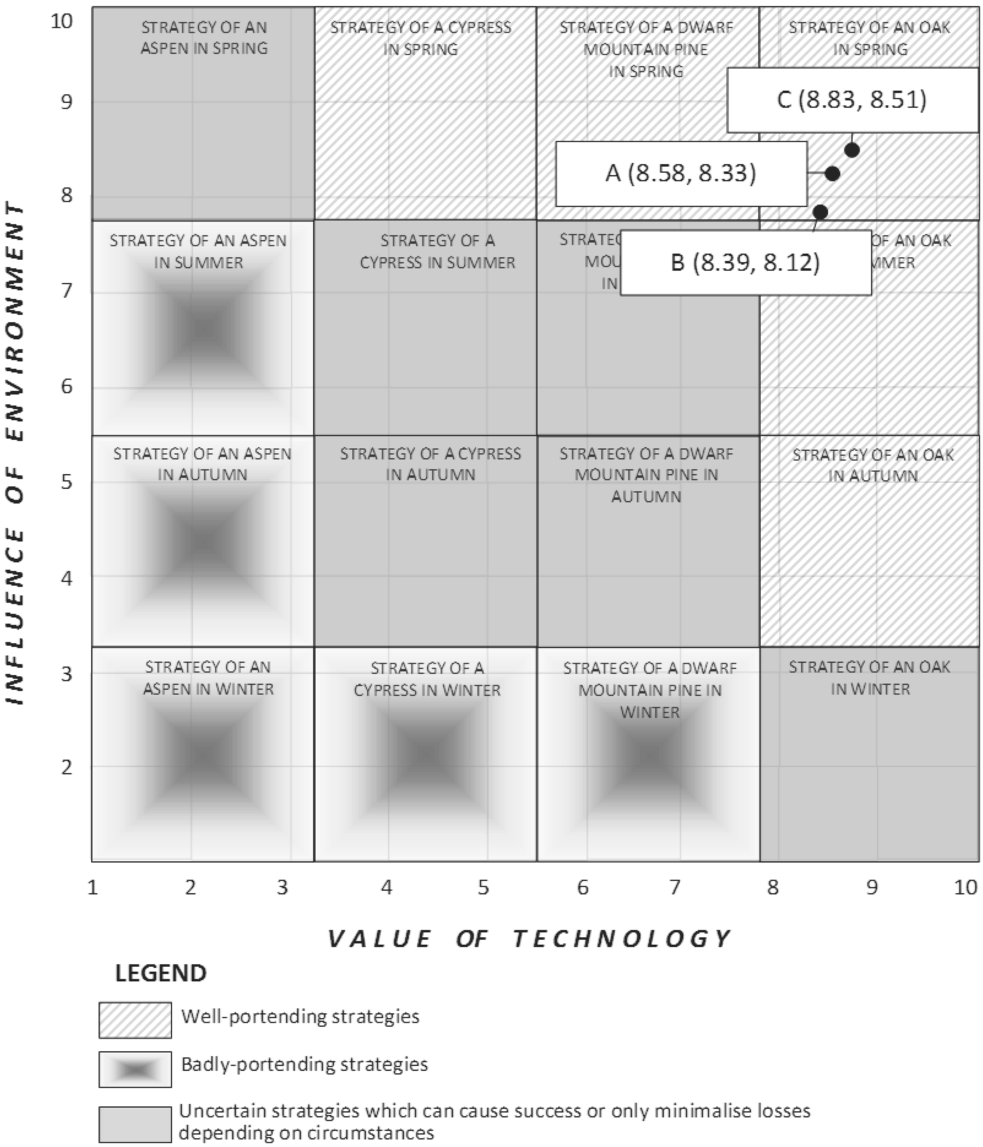


Figure 3. *The matrix of strategies for technologies called production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate with: (A) monolayer, (B) fifteen layers, (C) one hundred fifty layers*

of this technology group ensuring its return to the friendly area of sunny spring already in 2025; this, in connection with the technology's high attractiveness and strengthened potential, will ensure the achievement of spectacular success. The pessimistic variant expressed through the third determined strategic development track of the technology group anticipates the deepening of the crisis in the world and, because of this, an unfavourably developing political and economical situation which will contribute to the appearance of an increasingly larger number of difficulties in the group is attractive and has high potential which should become an environment (2015-2020) and a smaller amount of chances, which in 2025-2030 will lead to the necessity of functioning in the unfavourable conditions of frosty winter. The analysed technology bargaining chip in the highly unfavourable environment conditions. The recommended conduct is waiting through the difficulties and sustenance on the market at all costs, connected with the intensification of the search for new markets, customer groups and new products which are possible to manufacture using a given technology.

Table 5 contains numerical values which are the result of all the conducted research carried out for the three analysed technology groups, corresponding to different amounts of layers which constitute the applied PVD coating. The relatively small differences between the specific analysed technology groups on a macro scale decide on the highly coincident direction of the applied strategic development tracks, together with the appearing slight divergences.

Table 5. *Strategic development tracks of production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic; (MP) – the most probable*

No.	Technology name	Steady state 2010	Type of strategic development tracks	Years			
				2015	2020	2025	2030
1.	Production of metallic/ceramic monolayer coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate	Strategy of an oak in spring A (8.6, 8.3)	(O)	(9.1, 6.0)	(9.1, 7.0)	(9.2, 7.6)	(9.3, 8.7)
			(P)	(8.6, 5.8)	(8.6, 5.6)	(8.7, 2.1)	(8.7, 1.4)
			(MP)	(8.7, 5.9)	(8.8, 6.3)	(8.9, 7.2)	(9.0, 8.1)
2.	Production of metallic/ceramic multilayer (in the amount 15) coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate	Strategy of an oak in spring B (8.4, 8.1)	(O)	(8.5, 5.8)	(8.6, 6.7)	(8.8, 7.2)	(8.9, 8.4)
			(P)	(8.4, 5.6)	(8.4, 2.6)	(8.4, 2.0)	(8.5, 1.2)
			(MP)	(8.4, 5.8)	(8.4, 6.1)	(8.5, 6.9)	(8.6, 7.7)
3.	Production of metallic/ceramic multilayer (in the amount 150) coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate	Strategy of an oak in spring C (8.8, 8.5)	(O)	(9.3, 6.3)	(9.3, 7.4)	(9.4, 8.1)	(9.5, 9.1)
			(P)	(8.8, 5.9)	(8.8, 5.7)	(8.8, 2.4)	(8.8, 1.7)
			(MP)	(8.9, 6.1)	(9.0, 6.7)	(9.1, 7.7)	(9.2, 8.6)

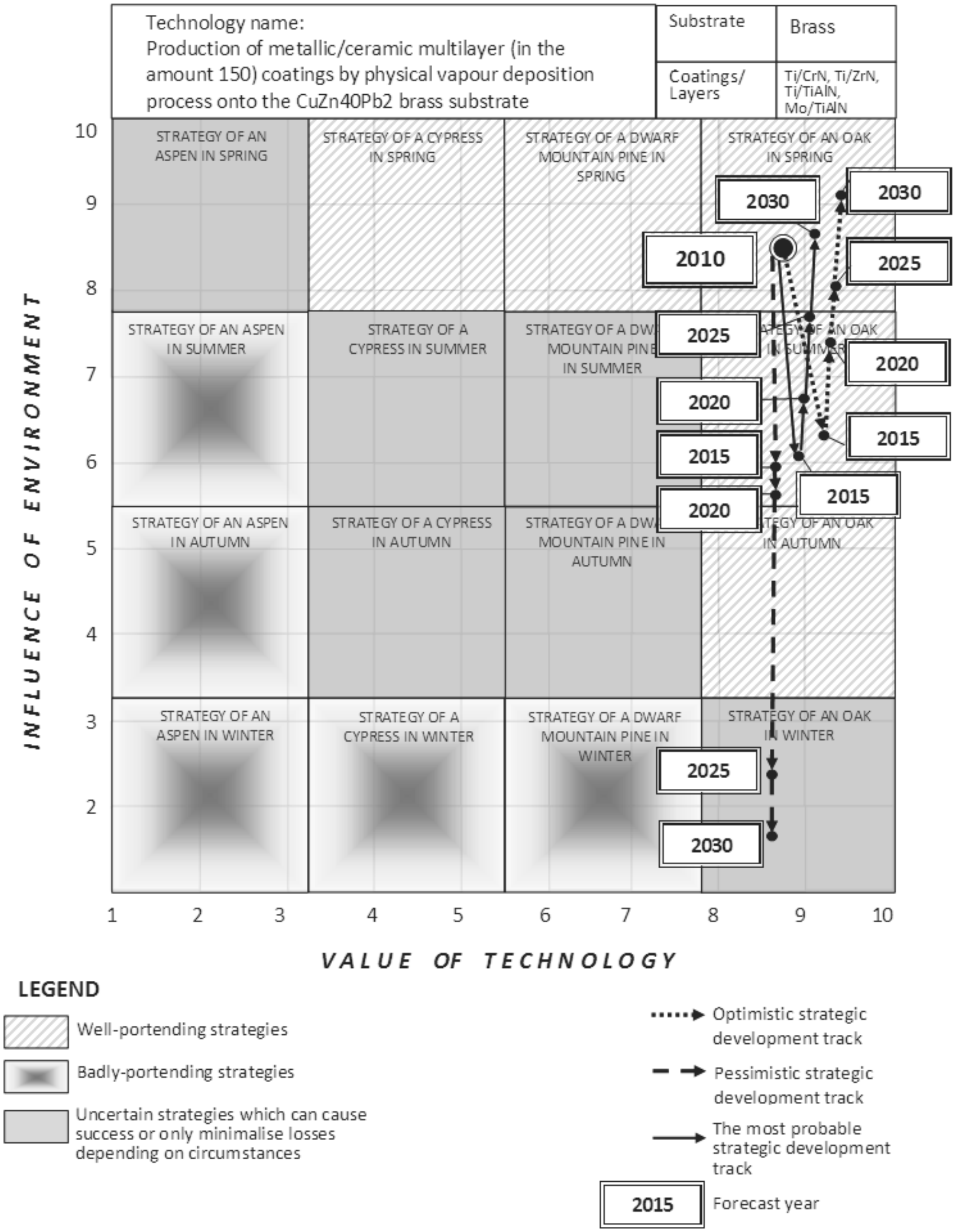


***Figure 4.** The strategic development tracks for demonstration technology called production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate with one hundred fifty (C)*

4. Received results of materials science research

4.1. Coatings structure

It was confirmed based on the metallographic tests made with a light microscope that the tested coatings were deposited with the PVD technique of reactive magnetron atomisation onto a dual-phase substrate ($\alpha+\beta$) of CuZn40Pb2 copper-zinc alloy. The coatings are characterised by the same thickness within their entire area and adhere tightly to the substrate. The dual-phase structure of CuZn40Pb2 alloy shown on the photos consists of phase α (light grains), phase β (dark grains) and of fine, uniformly distributed Pb precipitates (Figs. 5-7).

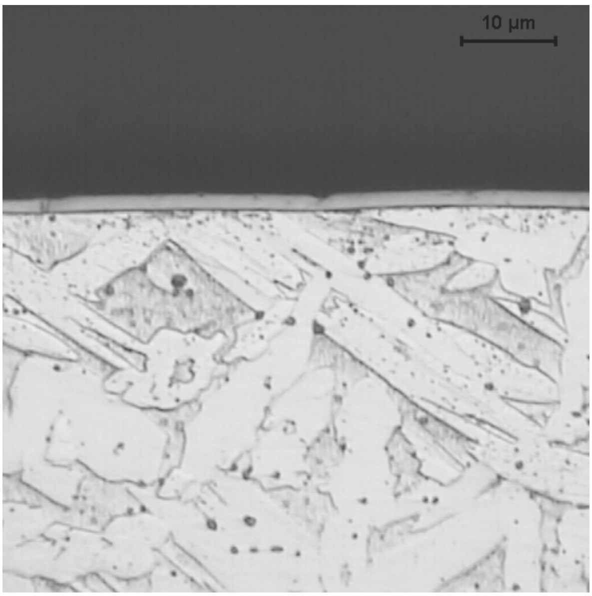


Figure 5. *Mo/TiAlN×1 coating deposited onto the CuZn40Pb2 substrate*

The fractographic tests of fractures in the tested coatings made with an electron scanning microscope confirm the previous claim that the coatings are deposited correctly (Figs. 8-10). The coatings have a compact structure without visible stratifications and defects. A column structure is clearly visible for monolayer coatings (Fig. 8). The fractures of multilayer coatings viewed with a scanning microscope show there is no column structure. The fact that 15 alternate layers for multilayer coatings were applied is confirmed (Fig. 9). The coatings consisting of 150 layers, due to the small thickness of each of the coatings applied, cannot be viewed.

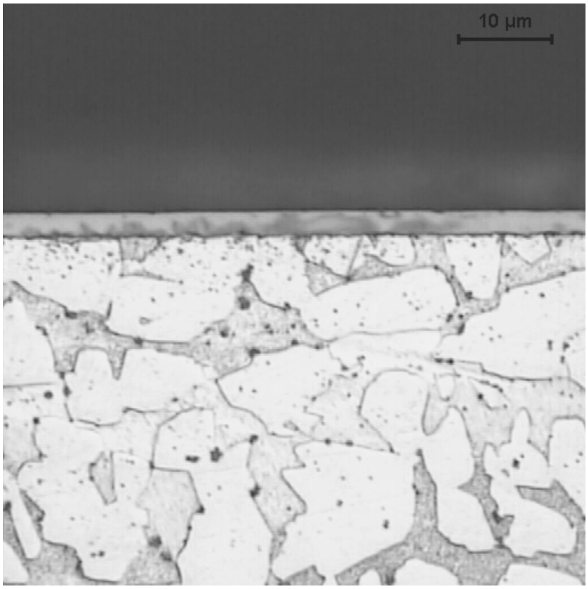


Figure 6. *Mo/TiAlN×15 coating deposited onto the CuZn40Pb2 substrate*

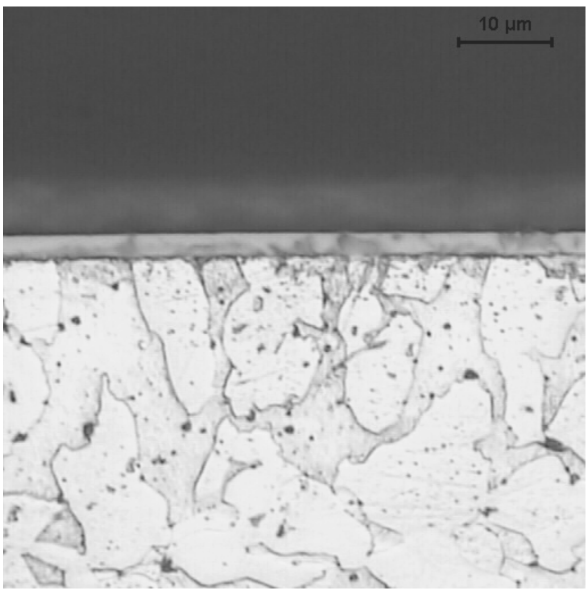


Figure 7. *Mo/TiAlN×150 coating deposited onto the CuZn40Pb2 substrate*

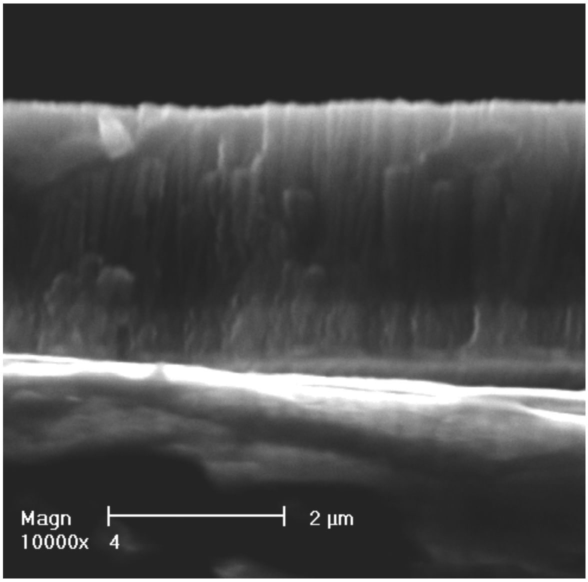


Figure 8. *Fracture of the Ti/TiAlN×1 coating deposited onto the CuZn40Pb2 brass substrate*

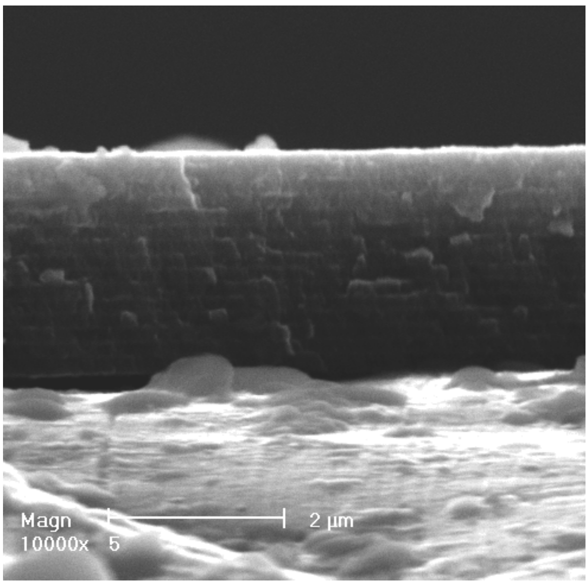


Figure 9. *Fracture of the Ti/TiAlN×15 coating deposited onto the CuZn40Pb2 brass substrate*

The surface morphology of the coatings produced on the copper-zinc alloy substrate is characterised by a high inhomogeneity because numerous bead- or ball-shaped particles are present at the surface (Figs. 11-13) which results from the concept of the PVD coating deposition process applied. This is caused by the presence of metallic droplets in plasma of metal atomised from a magnetron disc that take part in producing the coating. The size of droplet-shaped particles varies and starts with several decimals of micrometer to approx. 4 μm. Double particles and agglomerates formed of several combined particles can also be observed apart from single droplets. Local cracks in the coating are seen if large clusters of solidified particles are formed. Hollows were also observed in the surface of coatings where droplet-shaped particles are deposited that next drop out during a cooling operation after ending the coatings deposition process (Fig. 12).

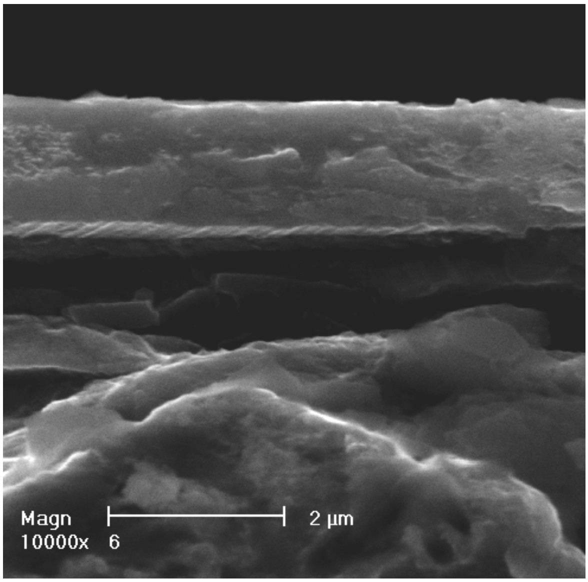


Figure 10. *Fracture of the Ti/TiAlN×150 coating deposited onto the CuZn40Pb2 brass substrate*

The droplets chemical composition tests carried out with an EDS X-ray scattered radiation energy spectrometer reveal that they are formed with pure metals (Ti, Cr, TiAl, Zr, Mo) depending on the coating type. This allows to conclude that these are liquid metal droplets released from a magnetron disc, which are deposited and solidify on the substrate surface. Therefore, different thermal properties (thermal expansion coefficient, heat conductance coefficient) of the particles formed with pure metals and coatings may be decisive for local cracks at the particle-coating interface and for them being dropped out after the end of the process. Oval or elongated-like particles also occur apart from droplet- or ball-shaped particles, which may be caused by the fact they are spattered against the surface in the coating deposition process.

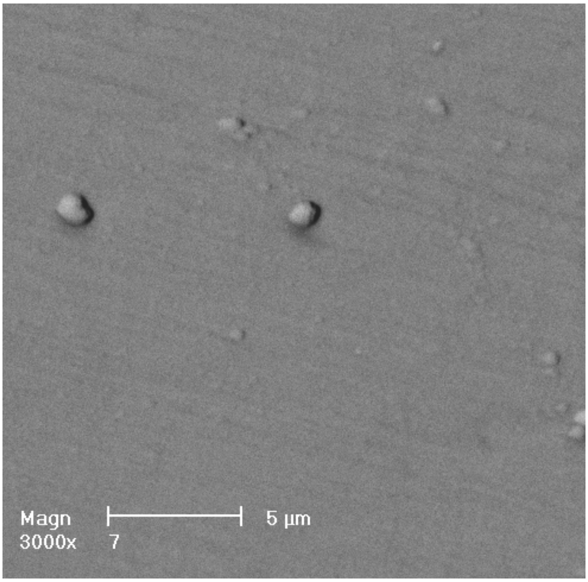


Figure 11. *Topography of the Ti/ZrN×1 coating surface deposited onto the CuZn40Pb2 brass substrate*

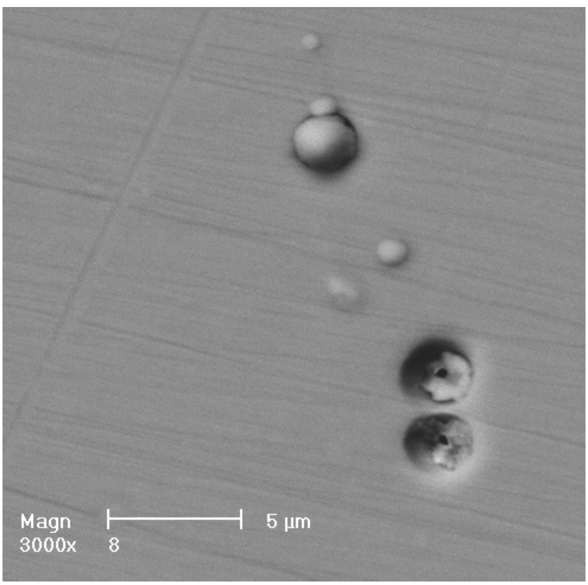


Figure 12. *Topography of the Ti/ZrN×15 coating surface deposited onto the CuZn40Pb2 brass substrate*

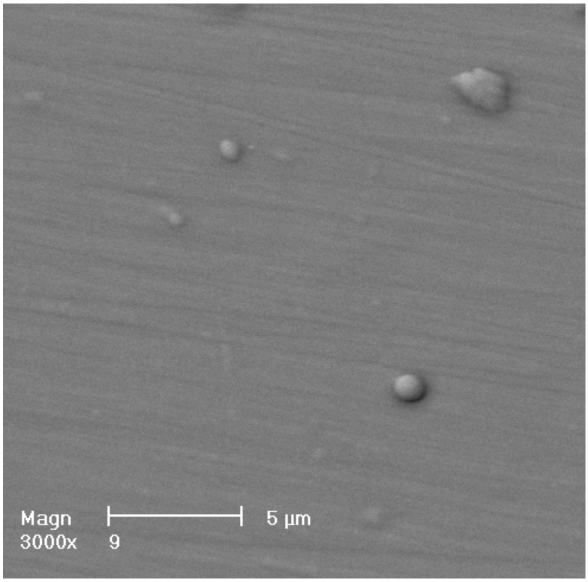


Figure 13. *Topography of the Ti/ZrN×150 coating surface deposited onto the CuZn40Pb2 brass substrate*

It was found based on the tests of thin foils produced from coatings (Fig. 14) that the coatings are composed of fine crystallites. While making observations in a light field and dark field the average size was estimated to be ca. 50-120 nm according to the coating type. The dark field image was created from reflexes {111}.

4.2. Coatings phase and chemical composition

It was determined with the methods of X-ray qualitative phase analysis that CrN phases for Ti/CrN coatings; ZrN for Ti/ZrN coatings; TiAlN for Ti/TiAlN coatings and Mo for Mo/TiAlN coatings show a privileged crystallographic orientation. The diffraction lines of TiAlN phase are moved to higher deflection angles as compared to the TiN phase. This is caused by a lower parameter of the network with an NaCl structure typical for TiN with 0.423 nm to 0.418 nm as Ti atoms (r = 0.146 nm) in the network are replaced with Al atoms (r = 0.143 nm).

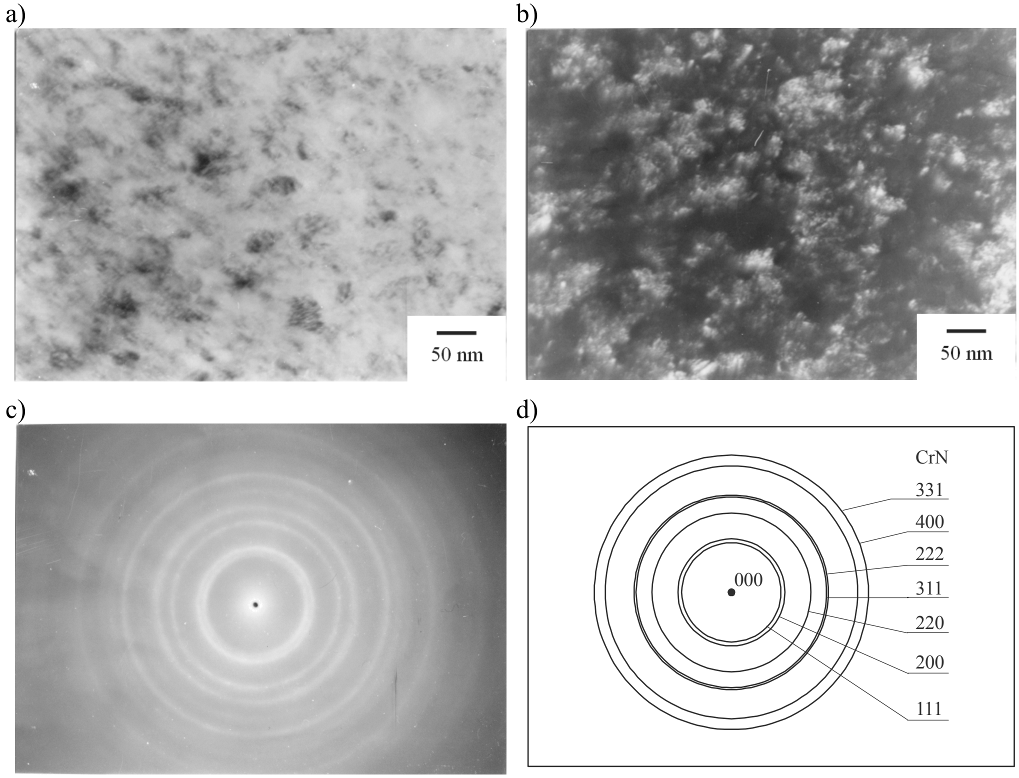


Figure 14. *Structure of the thin foil from the Ti/CrN×1 coating deposited onto CuZn40Pb2 brass substrate, a) light field, b) dark field from the {111} reflex, c) diffraction pattern from the area as in a, d) solution of the diffraction pattern*

The analysis of the tested coatings' texture was carried out with the inverse pole figures method. The texture of the coatings was found to be of axial nature, but the distinguished axis is deflected to the normal in relation to the surface of layers even by several degrees. The diffraction lines of the deposited nitride layers are, however, often very weak. Sometimes they partially overlap with the lines coming from the substrate. For this reason, to be able to present uniformly the texture of the tested coatings, the textures were not analysed with simple pole figures for the sake of inverse pole figures presenting the distribution of the normal to the surface of layers in the basic triangle {100}-{110}-{111}. The intensities of the following diffraction lines were analysed: {111}, {200}, {220} and {311}. Intensity growth for any of the lines corresponds to the existence of the distinguished crystallographic plane corresponding to this line. For data to be fully comparable, pole figures were made as quantitative figures, where level line figures are described as multiple normal densities corresponding to the given crystallographic orientation in relation to the density in the material deprived of a texture. The examples of pole figures are shown in Figs. 15-17, and the summary of texture tests results for PVD coatings are presented in Table 6. The texture of the tested samples is an axial texture, where the distinguished crystallographic axes are the normals to the planes {100}, {110}, {111} or {311}. A double texture exists for most of the coatings where – at different proportions – two planes parallel to the deposition plane are distinguished. Ti/CrN coatings are characterised by a moderately strong double texture where the distinguished planes are: {100} and {111} of CrN phase. Orientation intensity {111} is growing slightly along with an increase in the number of layers and {100} decreases. {100} orientation prevails in all the cases, however. Ti/ZrN are characterised by the same type of a texture, however, the strong component {111} of ZrN phase definitely prevails here. Its intensity is growing along with the growing number of layers in the coating. Ti/TiAlN coatings have a differentiated texture. Ti/TiAlN×1 and Ti/TiAlN×150 coatings have a double texture {110} + {311} of TiAlN phase, and {110} component prevails in Ti/TiAlN×150 coating, and {311} component in Ti/TiAlN×1 coating. The Ti/TiAlN×15 coating has a very weak double texture {100} + {111} of TiAlN phase where the {111} component is slightly stronger. The similar situation occurs in Mo/TiAlN coatings. The Mo/TiAlN×150 coating has a double texture {110} + {311} of TiAlN phase, where {110} component prevails, and Mo/TiAlN×1 and Mo/TiAlN×15 coatings have a texture with a differentiated {311} plane of TiAlN phase, with the texture of Mo/TiAlN×15 coating being slightly stronger.

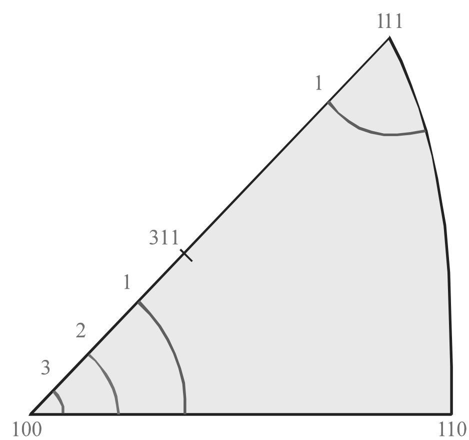


Figure 15. *Inverse pole figures representing the distribution of the normal to the Ti/CrN×1 coating surface in the 001-011-111 base triangle*

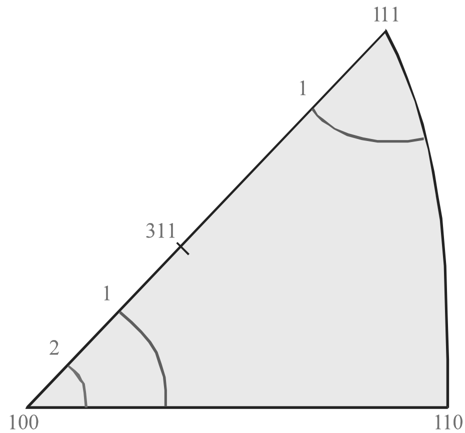


Figure 16. *Inverse pole figures representing the distribution of the normal to the Ti/CrN×15 coating surface in the 001-011-111 base triangle*

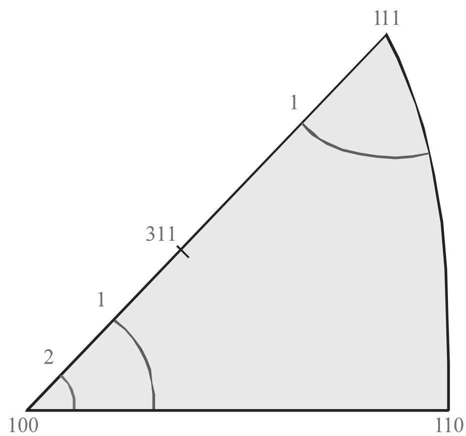


Figure 17. *Inverse pole figures representing the distribution of the normal to the Ti/CrN×150 coating surface in the 001-011-111 base triangle*

It should be assumed for the analysed coatings that the preferred orientation should be {111}, as it is a plane with a dense arrangement of atoms. The tests of the PVD coatings texture provide in the majority of cases a double texture {111} and {100} or {110} and {311}. The changes of crystallographic orientations of the tested coatings result from them being placed relative to the magnetron axis, temperature influence, no constant conditions in the deposition process which results from the cyclic changes in the supply of reactive gases for multilayer coatings and the slightly changing voltage and current conditions, which in turn influences the change of the energy vector resultant direction according to which condensate is oriented.

Table 6. *Summary results of the coatings textures*

Coating	Texturing level of the coatings referring to the discriminated plane			
	{100}	{110}	{111}	{311}
Ti/CrN×1	3	–	1	–
Ti/CrN×15	2	–	1	–
Ti/CrN×150	2	–	1	–
Ti/ZrN×1	1	–	8	–
Ti/ZrN×15	1	–	8	–
Ti/ZrN×150	1	–	16	–
Ti/TiAlN×1	–	2	–	3
Ti/TiAlN×15	1	–	2	–
Ti/TiAlN×150	–	3	–	1
Mo/TiAlN×1	–	–	–	2
Mo/TiAlN×15	–	–	–	2
Mo/TiAlN×150	–	3	–	1

Changes to the concentration of the coatings components and the substrate material according to the number of layers deposited were made in a glow discharge optical spectrometer (GDOS). For Ti/CrN×1 (Fig. 18) and Ti/CrN×15 (Fig. 19) coatings, the percentage atomic concentration of nitride is smaller by approx. 10-15% than the atomic concentration of chromium forming the nitride layer. The nitride and chromium concentration in the Ti/CrN×150 (Fig. 20) coating decreases relative to the maximum concentration of, respectively, the area of 45% and 35%, in atomic terms, to approx. 35% and 25% at the depth of 2 μm, whereas the titanium concentration increases from 20% at the area up to approx. 40% at the depth of 2 μm. The decisive reason for such distribution of concentrations for the analysed elements is the impact of the coating deposition conditions. A varying concentration of the elements forming Ti/CrN coatings signifies its chemical inhomogeneity. The chemical composition of Ti/ZrN coatings (Figs. 21-23) also deviates from the equilibrium composition. Nitride concentration is regularly decreasing to the maximum level, depending on the number of layers in the coating at the surface of 55-75%, in atomic terms, of 40-60% at the depth of approx. 1 μm, and the Zr concentration is rising from 25-45% at the surface up to 40-60% at the depth of approx. 1 μm. The similar situation occurs for Ti/TiAlN coatings (Figs. 24-26) where variations in the concentration of elements for pure metals Ti and Al forming the coating and of nitride are considerable. In case of Mo/TiAlN coatings (Figs. 27-29), the atomic concentration of molybdenum is larger than the total atomic concentration of TiAlN coating components. This is related to the longer deposition time of Mo layers as compared to the alternate TiAlN layer. Large changes in the concentration of elements occurring in multilayer components can be explained with the fact that there are no stable constant conditions in the coatings deposition process in the furnace. This is related to very rapid cyclic changes occurring over time in the supply of reactive gases depending on whether a layer of pure metal is deposited (e.g. Ti) or a nitride layer. Hence the lack of "ideal" conditions (the lack of pure argon only in the furnace atmosphere if Ti or Mo layers are deposited, the residues of reactive gas remaining in the furnace) makes it impossible to achieve coatings with the chemical composition close to the equilibrium composition.

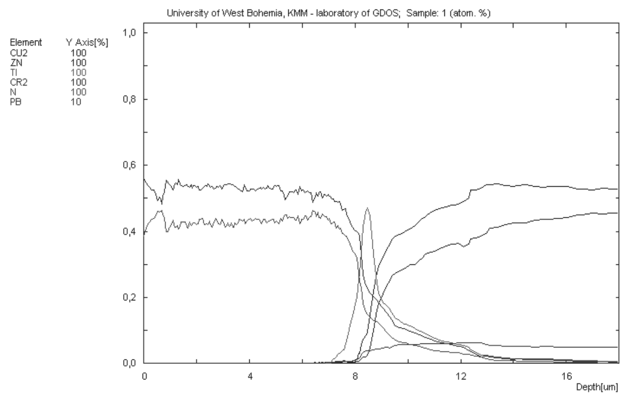


Figure 18. *Changes of constituent concentration of the Ti/CrN×1 and the substrate materials*

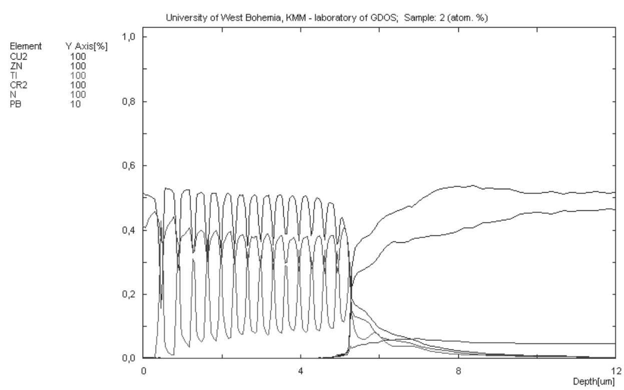


Figure 19. *Changes of constituent concentration of the Ti/CrN×15 and the substrate materials*

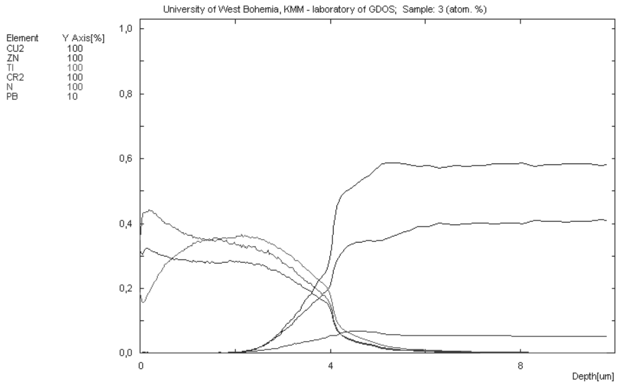


Figure 20. *Changes of constituent concentration of the Ti/CrN×150 and the substrate materials*

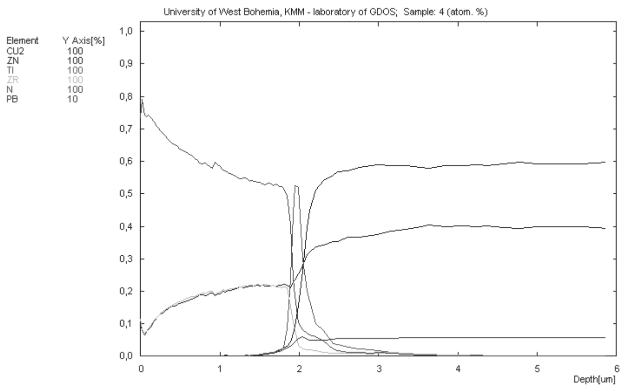


Figure 21. *Changes of constituent concentration of the Ti/ZrN×1 and the substrate materials*

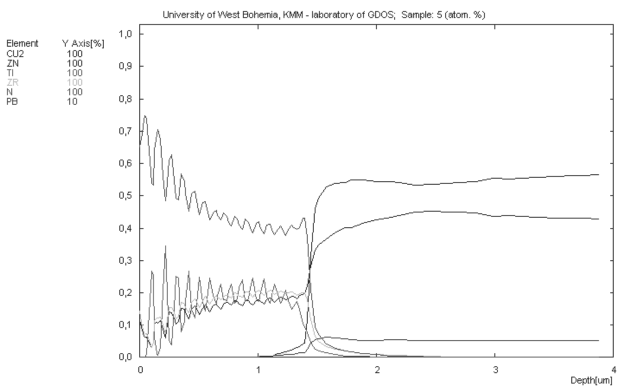


Figure 22. *Changes of constituent concentration of the Ti/ZrN×15 and the substrate materials*

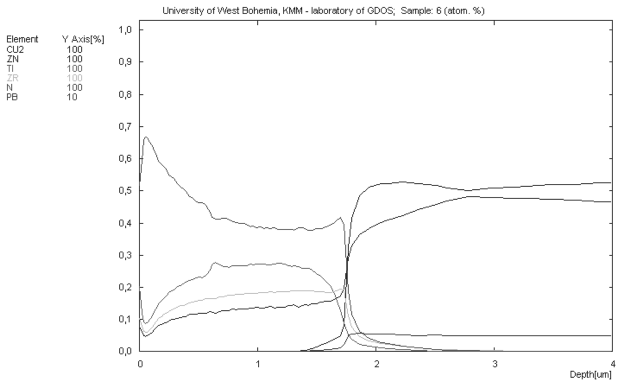


Figure 23. *Changes of constituent concentration of the Ti/ZrN×150 and the substrate materials*

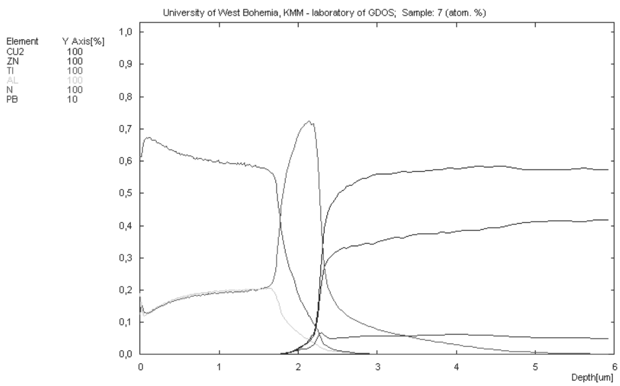


Figure 24. *Changes of constituent concentration of the Ti/TiAlN×1 and the substrate materials*

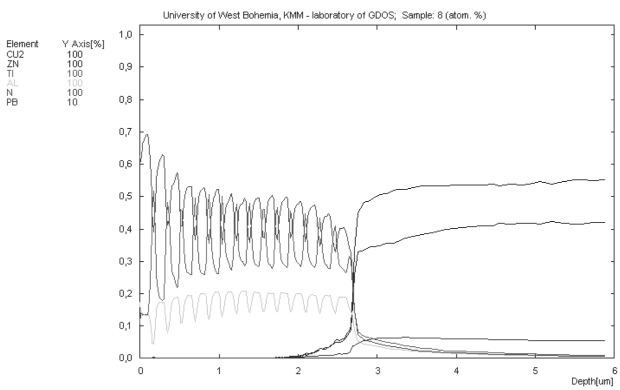


Figure 25. *Changes of constituent concentration of the Ti/TiAlN×15 and the substrate materials*

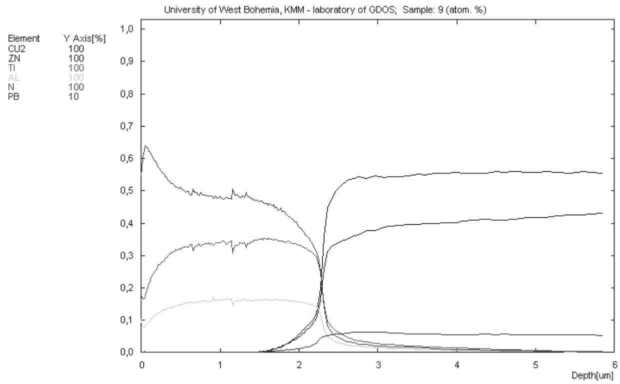


Figure 26. *Changes of constituent concentration of the Ti/TiAlN×150 and the substrate materials*

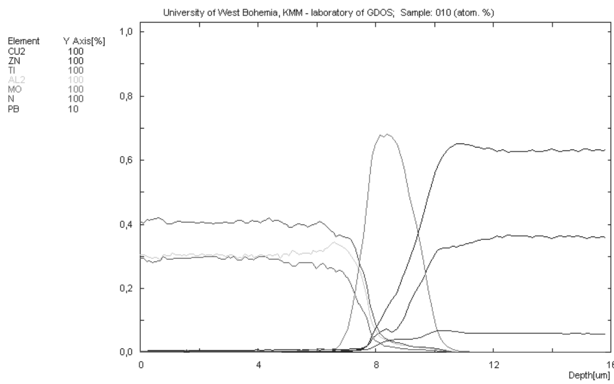


Figure 27. *Changes of constituent concentration of the Mo/TiAlN×1 and the substrate materials*

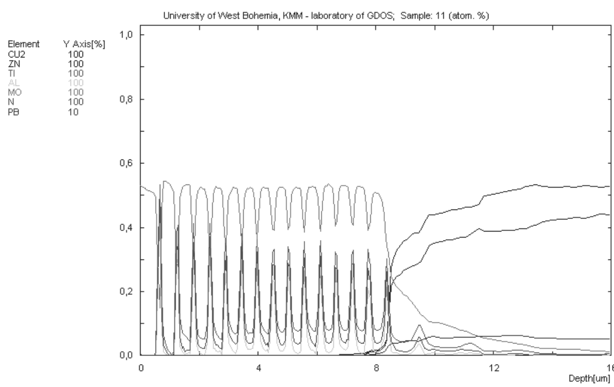


Figure 28. *Changes of constituent concentration of the Mo/TiAlN×15 and the substrate materials*

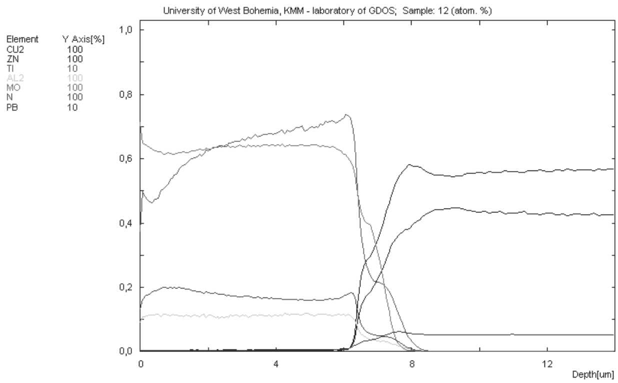


Figure 29. *Changes of constituent concentration of the Mo/TiAlN×150 and the substrate materials*

Research with the glow discharge optical spectrometer GDOS point out that the concentration of elements forming part of the substrate is growing in the bonding zone for the analysed cases starting with the substrate surface, and the concentration of the elements forming the coating is lowering. It may signify that a transient layer exists between the substrate material and the coating which improves the adhesion of the deposited coatings to the substrate, despite the fact that the results cannot be interpreted unequivocally in connection with the inhomogeneous evaporation of the material from the sample surface. The existence of the transient layer is a result of the higher desorption of the substrate surface and the defects occurring in the substrate as well as the displacement of elements in the bonding zone due to the activity of high-energy ions.

4.3. Coatings corrosion resistance

It was found based on the electrochemical corrosion tests that the coatings deposited in the PVD process on the copper-zinc alloy substrate may protect the substrate material effectively against the corrosive effect of an aggressive agent. An analysis of anodic polarisation and corrosive potential curves (Figs. 30-33) and a corrosion rate curve confirm the better corrosion resistance of the samples with coatings deposited as compared to the substrate (Table 7).

A current density in anodic scanning is always smaller than for an uncoated sample (12.4 μA/cm^2), which pinpoints a good protective effect.

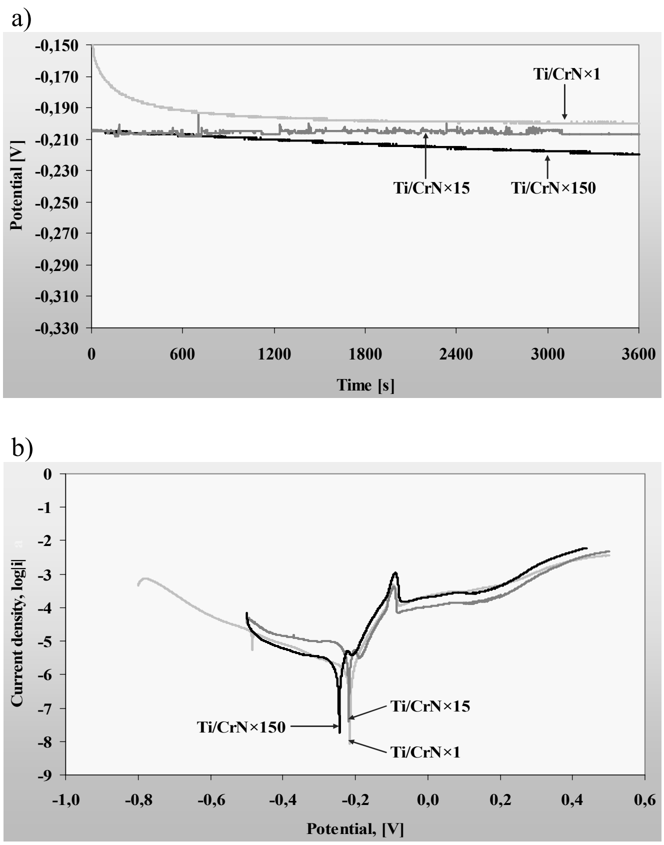
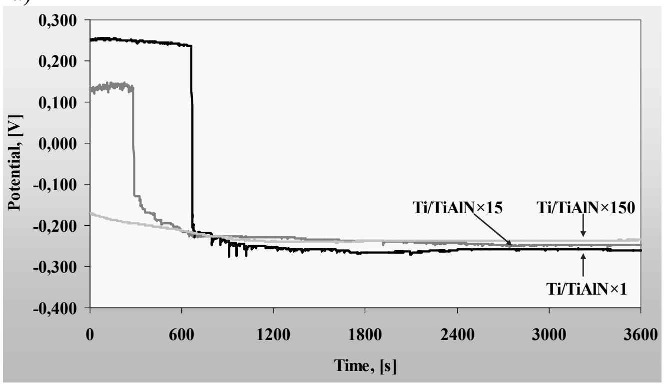


Figure 30. *a) Open circuit potential curves, b) potentiodynamic polarisation curves of the Ti/CrN coatings in 1 M HCl solution*

The progress of polarisation curves signifies the active quench annealing of the CuZn40Pb2 copper-zinc alloy surface uncoated with any layer. The smallest corrosion current density i_{cor}, thus the smallest anodic quench annealing of coatings and the related best corrosion protection properties are exhibited, regardless the type, the coatings with 150 and 15 layers. This can be explained with the fact that the system of multilayer deposition of coatings offers greater opportunities of preventing the cause of corrosion such as scratches or crevices. Small pores and cracks in the coating and the difference between a large area of cathode (coating), and a small area of anode (bottom of pores) is reducing the corrosion protection of coatings.

a)



b)

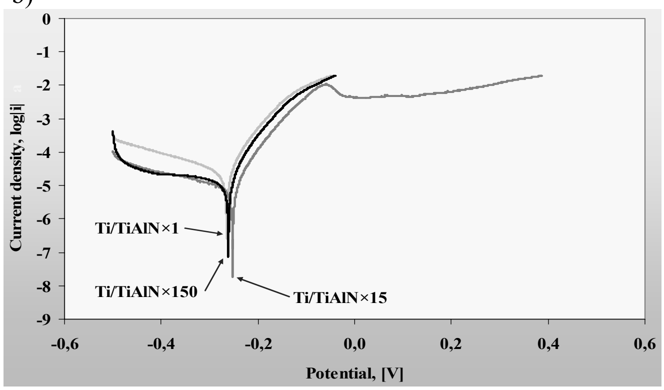


Figure 31. *a) Open circuit potential curves, b) potentiodynamic polarisation curves of the Ti/TiAlN coatings in 1 M HCl solution*

The defects and damages occurring on a single coating deposited in the deposition process can be neutralised or "masked" with the subsequent coating layers being deposited. This way, the path of the corrosion factor is extended or closed. Hence, with 150 layers, the corrosion factor needs more time to penetrate through coating defects than for 1 or 15 layers. The cathodic progress of curves shows that the reactions taking place on the substrate covered with coatings are being strongly inhibited. The anodic behaviour of the tested configurations may prove that coatings are porous or damaged. Many of the coatings were subject to anodic self-passivation, whereas the passive condition takes place within the narrow range of potential. An increase in anodic current related to transpassivation was observed within the potentials of 0.2-0.4 mV. One can estimate corrosion currents and corrosion rate from the progress of polarisation curves (Table 7).

a)

b)

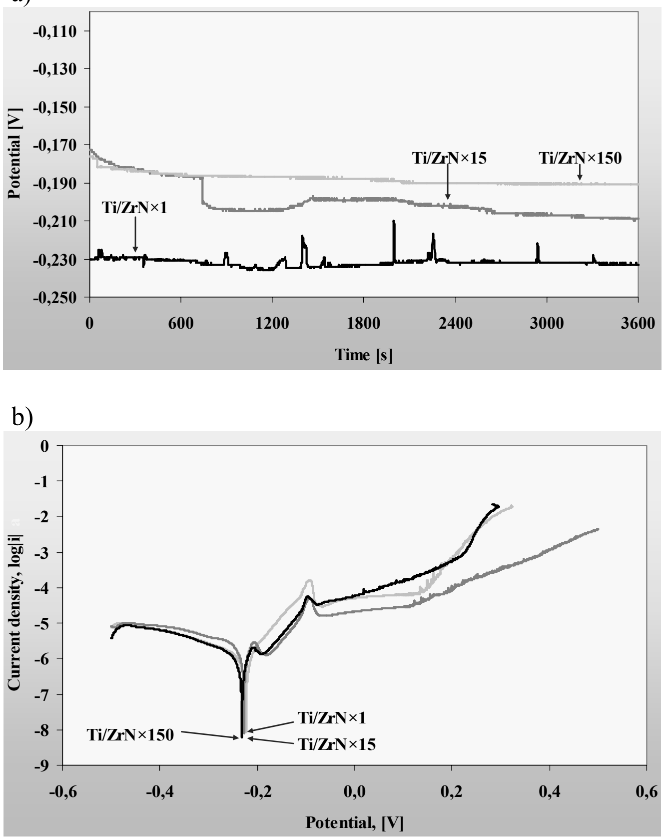


Figure 32. *a) Open circuit potential curves, b) potentiodynamic polarisation curves of the Ti/ZrN coatings in 1 M HCl solution*

The tests results for corrosion potential E_{cor} confirm improved corrosion resistance for multilayer coatings. Note-worthy is the fact that after 60 minutes of the experiment, the corrosion potential is clearly rising (from –315 mV to –254 mV) for the copper substrate uncoated with any coating, therefore it is subject to auto-passivation.

The results of impedance measurements (Table 7) confirm the higher corrosion resistance of copper-zinc alloy with coatings applied. The load transmission resistance is higher for the coated samples. This signifies that the coatings act as a diffusion barrier. Multilayer coatings have the best protection properties which is proved by the results of polarisation tests.

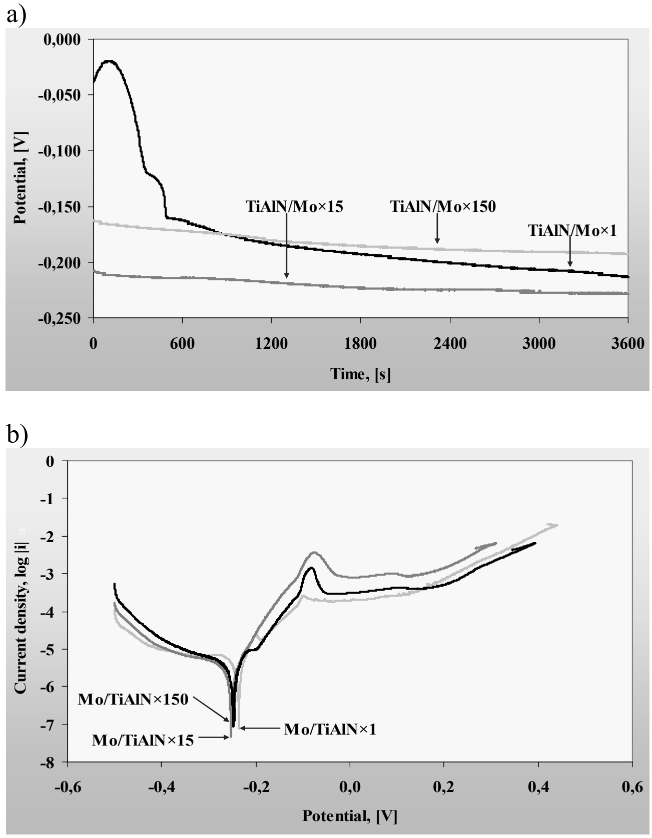


Figure 33. *a) Open circuit potential curves, b) potentiodynamic polarisation curves of the Mo/TiAlN coatings in 1 M HCl solution*

Table 7. *Summary results of the electrochemical corrosion investigation*

Coating	Corrosion potential E_{cor}, mV	Current density i_{cor}, μA/cm^2	Resistance polarisation R_p, kΩ·cm^2	Corrosion rate, mm/year
Ti/CrN×1	- 220	2.2	2.4	0.027
Ti/CrN×15	- 208	1.0	9.2	0.013
Ti/CrN×150	- 202	0.6	7.8	0.008
Ti/ZrN×1	- 238	0.6	7.1	0.008
Ti/ZrN×15	- 211	0.4	12.7	0.005
Ti/ZrN×150	- 191	0.2	12.9	0.003
Ti/TiAlN×1	- 297	5.1	0.58	0.063
Ti/TiAlN×15	- 268	3.4	1.4	0.032
Ti/TiAlN×150	- 243	1.1	1.1	0.014
Mo/TiAlN×1	- 220	5.4	2.9	0.066
Mo/TiAlN×15	- 228	2.1	4.9	0.025
Mo/TiAlN×150	- 194	1.3	4.1	0.015
Substrate	- 254	12.4	2.18	0.167

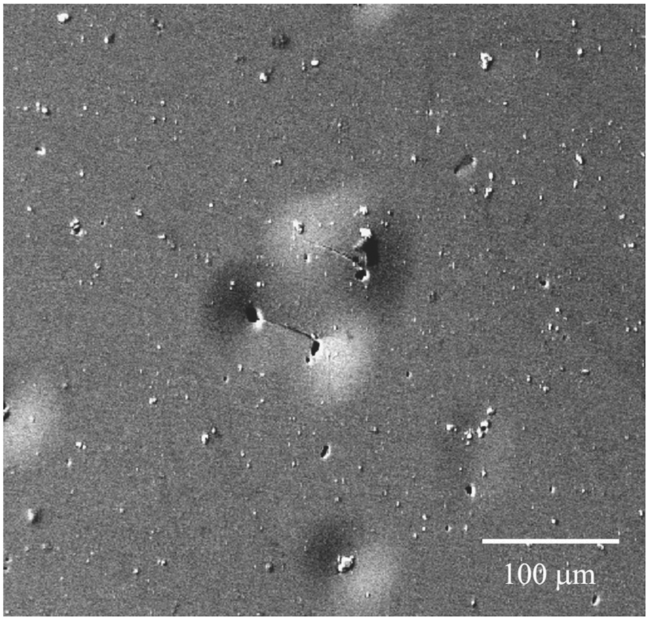


Figure 34. *Effect of the electrochemical corrosion on the Ti/CrN×15 coating*

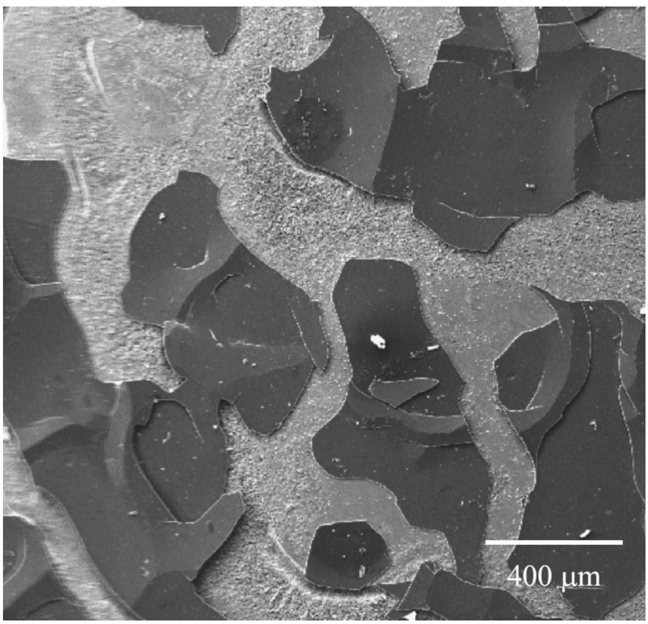


Figure 35. *Effect of the electrochemical corrosion on the Ti/TiAlN×1 coating*

Changes to the colour of coatings and their increased roughness caused by intensive surface quench annealing was observed during the activity of the aggressive factor. Microscope observations allow to conclude that the coating damage process due to electrochemical corrosion progresses in two ways. In the first case, coatings are damaged in numerous locations and the damage area itself is small (Fig. 34). In the other case, the coatings damaged by the aggressive factor embrace the large areas of coatings, hence damage appearance changes or some parts of coatings detach from the substrate material (Fig. 35).

4.4. Coatings mechanical properties

The tested CuZn40Pb2 copper-zinc alloy samples prepared for being deposited by polishing using a diamond suspension show the roughness of R_a= 0.01 μm. R_a roughness rises significantly between 0.15-0.29 μm (Table 8) if coatings are deposited. The topography of the coatings surface described earlier in the chapter undoubtedly contributes to such larger surface roughness. Those two factors are also crucial for the friction coefficient values within the range of 0.33-0.52 (Table 9) and fully correlate with the R_a roughness parameter values obtained for the same coatings.

Table 8. *Summary results of the mechanical properties*

Coating	Hardness, DHV 0.0025	Roughness R_a, μm	Young`s modulus, GPa	Stiffness, mN/μm	Thickness, μm
Ti/CrN×1	2450	0.19	258	330	5.8
Ti/CrN×15	2350	0.24	235	251	4.5
Ti/CrN×150	1800	0.22	228	216	3.1
Ti/ZrN×1	3100	0.20	291	224	2.1
Ti/ZrN×15	2700	0.19	343	265	1.6
Ti/ZrN×150	2200	0.20	290	253	1.9
Ti/TiAlN×1	2400	0.22	348	274	2.3
Ti/TiAlN×15	2100	0.22	259	253	2.7
Ti/TiAlN×150	1850	0.25	210	195	2.2
Mo/TiAlN×1	2400	0.18	302	236	6.2
Mo/TiAlN×15	2200	0.19	293	226	6.5
Mo/TiAlN×150	2000	0.25	297	250	5.9

Dynamic hardness was determined at the load of 0.025 N. A clear effect of the number of layers applied onto the substrate on the obtained dynamic hardness values was found

(Table 8). Monolayer coatings show the largest hardness. As the number of layers rises, their hardness lowers. This stems from the larger hardness of a single thick nitride layer, e.g. ZrN or TiN than from the system of 15 or 150 layers where the properties and hardness on the alternate metallic (soft) layer and the nitride (hard) layer differ. The value of the elongation elasticity factor E and of the coating rigidity factor (Table 8) were determined using Hardness 4.2 software fitted with the ultra-microhardness tester and with the relationship between the load and indenter depth into the tested layer. The rigidity of the tested layers is within 195-330 mN/μm, and Young's modulus for the applied coatings is within 348-210 GPa. Similarly as for hardness, smaller elongation elasticity values for multilayer coatings can be observed.

The tests of internal stresses existing within coatings were carried out with X-ray analysis methods. The values obtained (Table 9) indicate that internal compressive (negative) stresses occur in coatings. Their strength properties grow as a result. Smaller values of internal stresses in multilayer coatings result from a possibility of reducing them at the subsequent alternate hard nitride and soft pure metallic layers.

Table 9. *Summary results of the mechanical properties*

Coating	Critical load L_{C2}, N	Friction distance, m (pin-on-disc test)	Friction coefficient, μ	Residual stresses, GPa
Ti/CrN×1	50	100	0.37	-2.6
Ti/CrN×15	48	98	0.47	2.3
Ti/CrN×150	47	24	0.43	-0.1
Ti/ZrN×1	45	16	0.38	-8.9
Ti/ZrN×15	41	10	0.38	-4.7
Ti/ZrN×150	37	7	0.40	-1.6
Ti/TiAlN×1	41	10	0.43	-11.9
Ti/TiAlN×15	40	5	0.42	2.1
Ti/TiAlN×150	38	2.5	0.48	-0.2
Mo/TiAlN×1	48	75	0.35	-1.3
Mo/TiAlN×15	45	16	0.37	-1.3
Mo/TiAlN×150	40	3	0.47	-0.1

The values of the critical load L_{C2} were determined with the scratch test method with linear load growth characterising the adhesion of the tested coatings to the substrate material caused mainly by adhesion and diffusion forces. The summary of tests results is presented in Table 9. The critical load L_{C2} was determined as corresponding to a sudden decline in acoustic emission being the friction coefficient change signal between the diamond indenter and the coating subject to partial cracking (Figs. 36-38) and based on an optical observation with a light microscope. The results obtained allow to conclude that the critical load L_{C2} at which coatings are damaged is reduced along with more layers in the coating. An additional thin intermediate layer in monolayer coatings improves the adhesion of nitride coating to the substrate as it counteracts the propagation of cracks and reduces stresses within the coating-substrate zone. For multilayer coatings, the coating is deformed as the indenter presses against the coating because the softer and more flexible layers deposited with pure metals are more strongly deformed than the hard nitride layers. Bending stresses are produced that are destroying the system of alternate hard and soft layers due to cracks in nitride layers subjected to an excessive deformation. The relatively softer and more flexible pure metal layers are not able to counteract effectively the wear in contact with other materials, unlike the thick layers.

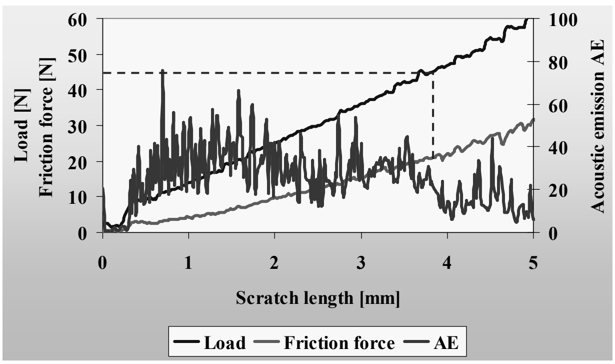


Figure 36. *Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the Ti/ZrN×1 coating*

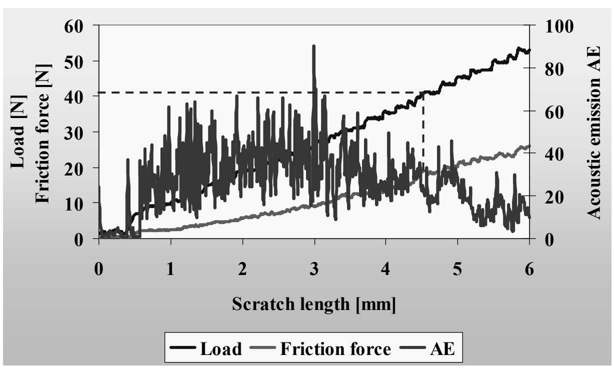


Figure 37. *Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the Ti/ZrN×15 coating*

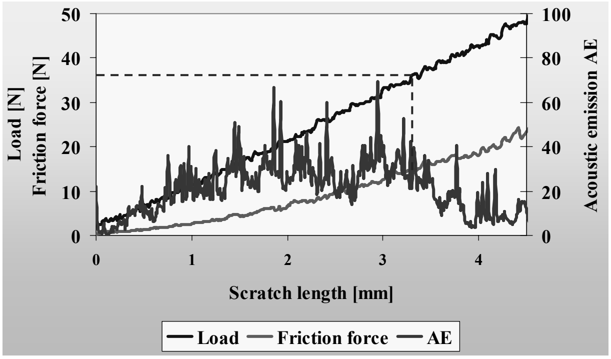


Figure 38. *Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the Ti/ZrN×15 coating*

To determine the nature of damages created during the adhesion test, they were tested with a scanning electron microscope (Fig. 39).

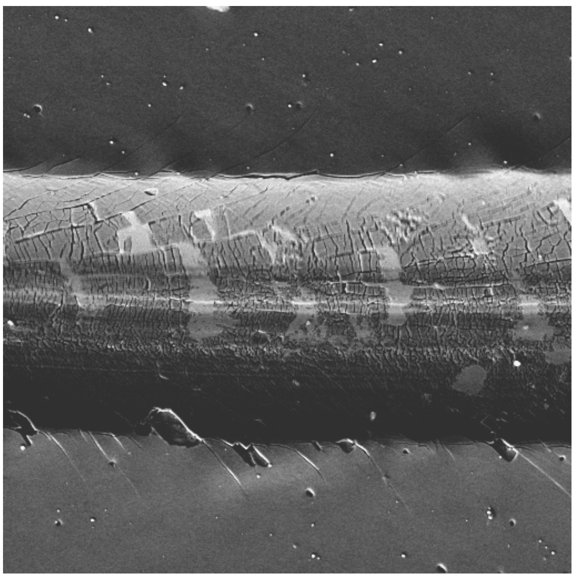


Figure 39. *Characteristic failure of the Ti/CrN×1 coating developed during the scratch test, SEM*

In general, the first symptoms of a damaged coating in the majority of the tested coatings deposited with the PVD technique are arch-like cracks caused by stretching and chipping at the bottom of the scratch formed during the adhesion test. In few cases, minor chipping occurs at the scratch edges. As the load increases, semicircles are developed caused by confermal cracking leading to delamination and cracks as a result of which local coating delamination occurs. After an attempt to fracture copper-zinc alloy with coatings deposited made after pre-cooling in liquid nitrogen, no delamination was identified in any case along the substrate-coating separation surface, signifying that the coatings adhere well to the substrate. The coatings are almost completely delaminated at the maximum load, however, in all the examined cases, which may be caused by a large difference in mechanical and physical properties between the soft substrate material and the hard coating.

4.5. Coatings tribological properties

Erosion tests were conducted to identify the functional and operating properties of coatings. Considering the specific properties of the substrate material (relatively soft compared to coatings), it is impossible to make a standard 3D profile analysis of craters formed in erosion tests as the erodent indents into the soft substrate at the bottom of the craters. The profile of the generated damage changes as a result enabling to assess correctly the degree of damaging the coatings. As the nozzle is positioned perpendicular to the surface of coatings, short intervals were made between the subsequent test steps. Colour metallography methods applying a computer image analysis with a scanning electron microscope were utilised to evaluate the degree of the tested coatings' damage (Fig. 40) by taking advantage of the different colours of the substrate and coatings (green colour – coating, red – substrate, blue – erodent), what unfortunately can not be show at black and white pictures.

The observations of coatings damaged in the erosion test with a scanning electron microscope enable also to determine the percentage of the damaged area in respect of the total observation field area agreed in a way ensuring that the largest area of the formed crater is covered, using stereological methods for this purpose. The data shown in Figures 41-44 prove that the best resistance to the erodent is exhibited by monolayer coatings, especially Ti/CrN×1 coating, where after 1 second of the test only 1.1% of the substrate is exposed by the impacting erodent particles and the percentage degree of coating damage is approx. 11.5% (percentage of the exposed substrate + erodent particles driven into the coating and into the substrate material) as a result of which approx. 88% of the coatings area does not show any damage. In case of the same coating consisting of 15 layers, the percentage share of the coatings area not exhibiting any damage due to the destructive activity of the erodent after 1 s is 53%, and for 150 layers – 17.5%. This means that over 80% of the coatings area is damaged and its erosion resistance is over fourfold smaller than a monolayer coating after the same test duration. The perforation rate for other multilayer coatings is much greater thus compromising their resistance to erosive damage. For Ti/TiAlN coatings after 0.3 s of the test, depending on the number of layers, the perforation rate varies within 14-83%, for Ti/ZrN coatings after 0.4 s of the test – 27-78%, and for Mo/TiAlN coatings, after 0.4 s the perforation rate for coatings is within 35-69%.

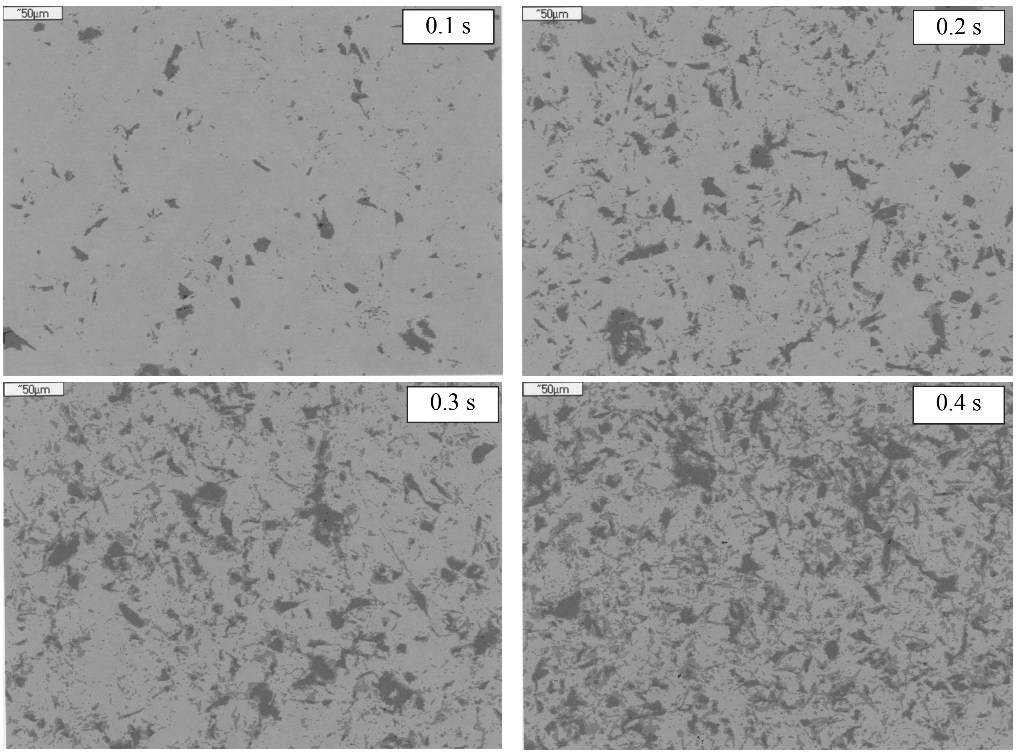


Figure 40. *Surface of the Ti/ZrN×1 coating in various of the erosion test stages, depending on the SEM test duration*

The subsequent damage process stages can be identified due to the acting erodent particles by analysing the coatings surface. The erodent, in the initial stage of the erosion test's duration, when driving into the coating material, causes minor coating damages due to abrasion or cracking in various locations. The spots occur on the surface where the coating is intact and small areas

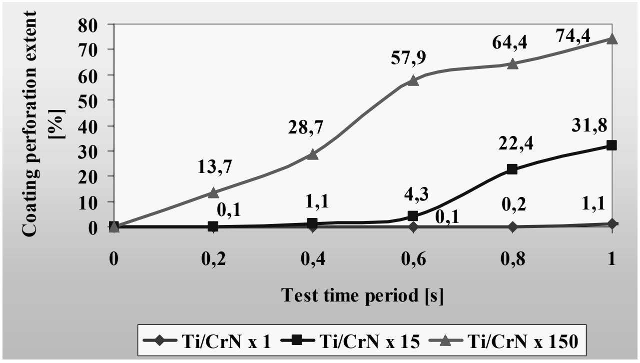


Figure 41. *Extent of perforation of the Ti/CrN coatings deposited onto the CuZn40Pb2 substrate depending on the erosion test time period*

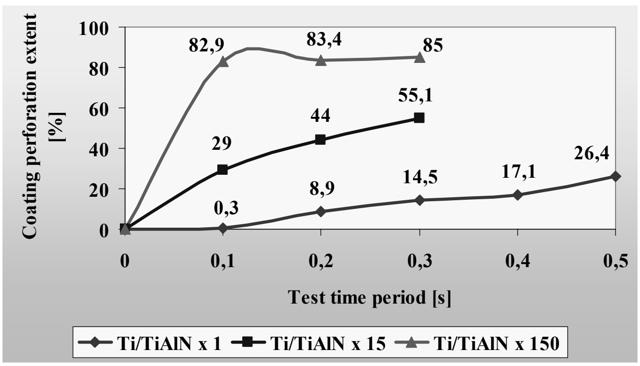


Figure 42. *Extent of perforation of the Ti/TiAlN coatings deposited onto the CuZn40Pb2 substrate depending on the erosion test time period*

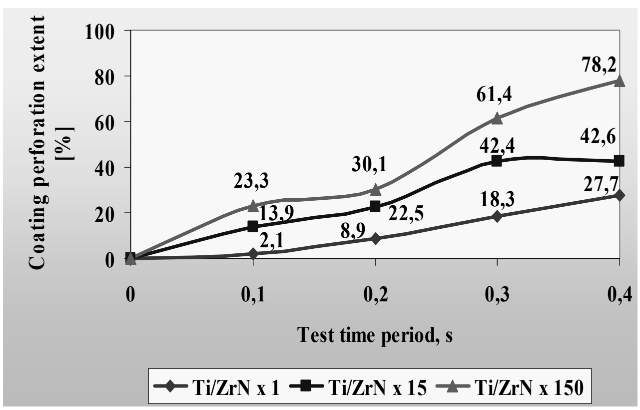


Figure 43. *Extent of perforation of the Ti/ZrN coatings deposited onto the CuZn40Pb2 substrate depending on the erosion test time period*

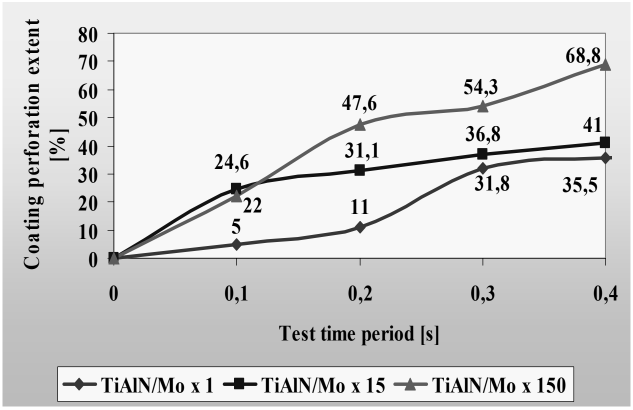


Figure 44. *Extent of perforation of the Mo/TiAlN coatings deposited onto the CuZn40Pb2 substrate depending on the erosion test time period*

where the hard erodent particles have been most of all fixed. The new centres of coating damage occur in the next stages and the area is expanding where coatings have been damaged. The places with the fused erodent particles should also be considered an area where the coating has been damaged. Erosion is gaining momentum over time and the coating areas damaged to date are combined forming large stretches with a crater being developed as a consequence. The crater is next being deepened and ultimately the vast areas of the substrate surface become exposed causing the coating material to be completely damaged with erodent particles. An abrasion strength test with the pin-on-disc method was performed to determine fully the functional and operating characteristic of the coatings deposited in the PVD process onto the copper-zinc alloy substrate. It is one of the most popular tribological tests identifying the impact of abrasion on abrasive wear resistance. The time of damaging the coatings due to the acting countersample was determined in a curve analysis presenting the friction coefficient according to the friction path (Figs. 45-47) and with an optical observation in a light microscope. Almost all the friction curves have a similar characteristic with the initial transient state having stabilised progress. The friction coefficient in some cases at the start of the test increases abruptly along with the growing friction path until the set condition is reached, which usually occurs when the countersample covers 3×10 m. The damage of coatings corresponds to a sudden change in the friction coefficient charts due to strong oscillations on the curve or by a sudden increase or decrease of the friction coefficient and due to the changing curve progress character on the chart. It was found after carrying out the tests that monolayer coatings, in particular

Ti/CrN×1, have the largest path after which coatings are damaged due to friction by the countersample and this is consistent with the results obtained after making erosion tests (Table 9). Similar as in that case, the smallest resistance to abrasive wear was found for multilayer coatings, with, accordingly, 150 and 15 layers. The system of alternate, very thin hard nitride layers and soft layers deposited with pure metals does not secure sufficient wear protection, as contrary to monolayer coatings where a single, thick nitride layer represents good protection for various functional materials during the alternate activity of the abrading elements in a relative motion.

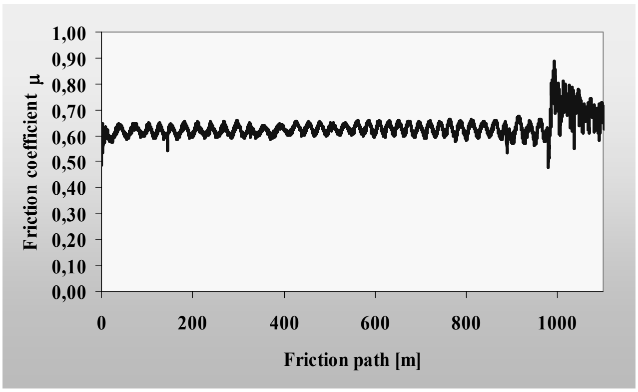


Figure 45. *Plot of the friction coefficient depending on friction path for the pin-on-disc of the Ti/CrN×1 coating*

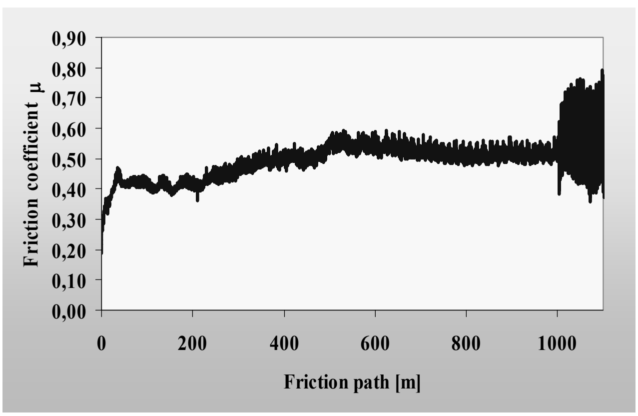


Figure 46. *Plot of the friction coefficient depending on friction path for the pin-on-disc of the Ti/CrN×15 coating*

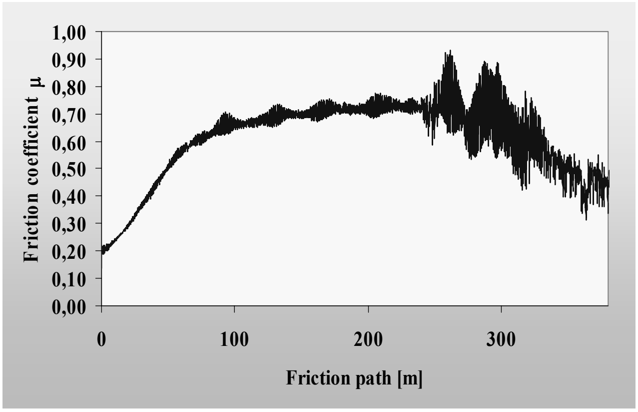


Figure 47. *Plot of the friction coefficient depending on friction path for the pin-on-disc of the Ti/CrN×150 coating*

5. Outworked technology roadmaps

A series of roadmaps of the analysed groups of technology were created on the basis of achieved results of experimental and comparative research. A representative roadmap prepared for production of metallic/ceramic multilayer (in the amount 150) coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate in Table 10 is shown. The horizontal axis of a roadmap corresponds to time intervals, and the vertical axis to seven layers called as following: time, conceptual, product, technological, spatial, staff and quantitative. The upper layers were divided into more sublayers arranged hierarchically detailed starting from the upper ones being the most general one determining premises, causes and reasons of the realised actions through the middle ones characterizing a product and technology in lower layers of the technology precising an organisational and technical details at the end. Technology roadmaps are a very comfortable and practical tool of comparative analysis which facilitates the selection of technologies according to the selected criterion, and when supplemented by operation sheets with precise technological details – they enable the implementation of a given technology in industrial practice. A very large significance of technology roadmaps is their flexibility which enables their supplementing and expanding by new sub-layers depending on the arising needs. On the basis of data contained in given roadmaps prepared for all the analysed groups of technology the summary was outworked, which is presented in Table 11.

Table 10. *Demonstrating technology roadmapping for production of metallic/ceramic multilayer (number of layers 150) coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate*

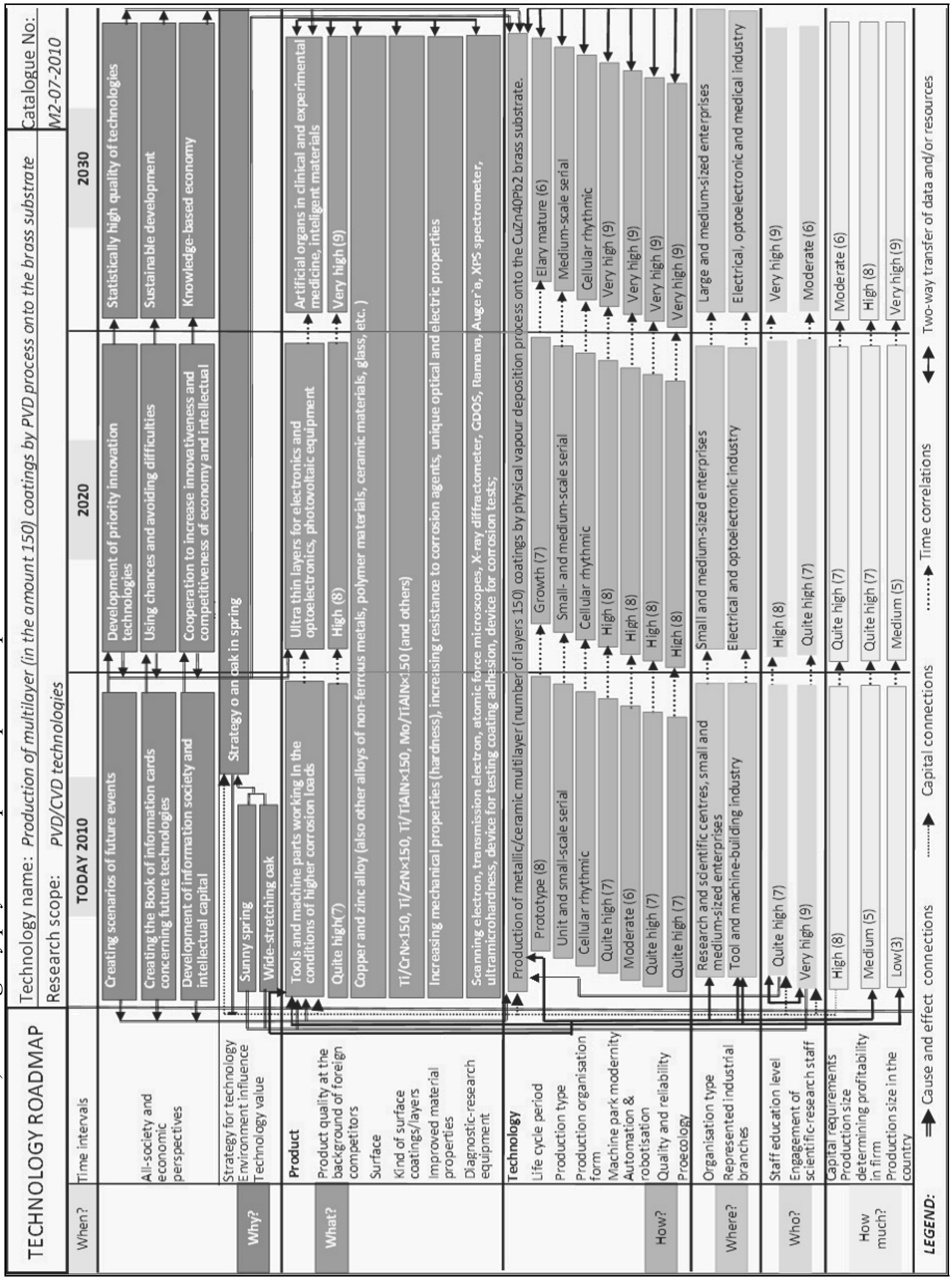


Table 11. *Selected main source data used for preparation of roadmaps for production of metallic/ceramic coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate with: (A) monolayer, (B) fifteen layers, (C) one hundred fifty layers*

No.	Analysed factor		Time interval	Analysed technology		
				A	B	C
1.	All-society and economic perspectives	1st trend	2010	Creating scenarios of future events		
			2020	Development of priority innovation technologies		
			2030	Statistically high quality of technologies		
		2nd trend	2010	Creating the Book of information cards concerning future technologies		
			2020	Using chances and avoiding difficulties		
			2030	Sustainable development		
		3rd trend	2010	Development of information society and intellectual capital		
			2020	Cooperation to increase innovativeness and competitiveness of economy and intellectual capital		
			2030	Knowledge-based economy		
2.	Strategy for technology		2010	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in spring
			2020	Strategy of an oak in summer	Strategy of an oak in summer	Strategy of an oak in summer
			2030	Strategy of an oak in spring	Strategy of an oak in spring	Strategy of an oak in spring
3.	Environment influence		2010	Sunny spring		
4.	Technology value		2010	Wide-stretching oak		
5.	Product		2010	Tools and machine parts working in the conditions of higher mechanical and thermal loads (e.g. matrices for plastic working)	Tools and machine parts working in the conditions of higher mechanical, thermal and corrosion loads	Tools and machine parts working in the conditions of higher corrosion loads
			2020	Ultra thin layers for electronics and optoelectronics, photovoltaic equipment	Ultra thin layers for electronics and optoelectronics, photovoltaic equipment	Ultra thin layers for electronics and optoelectronics, photovoltaic equipment
			2030	Artificial organs in clinical and experimental medicine, photovoltaic equipment	Artificial organs in clinical and experimental medicine, photovoltaic equipment	Artificial organs in clinical and experimental medicine, intelligent materials,
6.	Product quality at the background of foreign competitors		2010	Quite high (7)	Quite high (7)	Quite high (7)
			2020	High (8)	High (8)	High (8)
			2030	Very high (9)	Very high (9)	Very high (9)

No.	Analysed factor	Time interval	Analysed technology		
			A	B	C
7.	Improved material properties	2010	Increasing mechanical properties (hardness), increasing resistance to corrosion agents and unique optical and electric properties		
		2020			
		2030			
8.	Diagnostic-research equipment	2010	Scanning electron, transmission electron, atomic force microscopes, X-ray diffractometer, GDOS, Raman, Auger, XPS spectrometer, ultramicrohardness, device for testing coating adhesion, device for corrosion tests		
		2020			
		2030			
9.	Life cycle period	2010	Prototype (8)	Prototype (8)	Prototype (8)
		2020	Growth (7)	Growth (7)	Growth (7)
		2030	Early mature (6)	Early mature (6)	Early mature (6)
10.	Production type	2010	Unit- and small-scale serial	Unit- and small- scale serial	Unit- and small-scale serial
		2020	Small- and medium-scale serial	Small- and medium-scale serial	Small- and medium-scale serial
		2030	Medium- scale serial	Medium- scale serial	Mass
11.	Production organisation form	2010	Cellular rhythmic	Cellular rhythmic	Cellular rhythmic
		2020	Cellular rhythmic	Cellular rhythmic	Cellular rhythmic
		2030	Cellular rhythmic	Cellular rhythmic	Cellular rhythmic
12.	Machine park modernity	2010	Quite high (7)	Quite high (7)	Quite high (7)
		2020	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Very high (9)
13.	Automation and robotisation	2010	Moderate (6)	Moderate (6)	Moderate (6)
		2020	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Very high (9)
14.	Quality and reliability	2010	Quite high (7)	Quite high (7)	Quite high (7)
		2020	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Very high (9)
15.	Proecology	2010	Quite high (7)	Quite high (7)	Quite high (7)
		2020	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Very high (9)
16.	Organisation type	2010	Research and scientific centres, small- and medium- sized enterprises		
		2020	Small- and medium- sized enterprises		
		2030	Large- and medium- sized enterprises		
17.	Represented industrial branches	2010	Tool and machine-building industry	Tool and machine-building industry	Tool and machine-building industry
		2020	Electrical and optoelectronic industry	Electrical and optoelectronic industry	Electrical and optoelectronic industry
		2030	Electrical, optoelectronic and medical industry	Electrical, optoelectronic and medical industry	Electrical, optoelectronic and medical industry

No.	Analysed factor	Time interval	Analysed technology		
			A	B	C
18.	Staff education level	2010	Quite high (7)	Quite high (7)	Quite high (7)
		2020	High (8)	High (8)	High (8)
		2030	Very high (9)	Very high (9)	Very high (9)
19.	Engagement of scientific-research staff	2010	Very high (9)	Very high (9)	Excellent (10)
		2020	Quite high (7)	Quite high (7)	Excellent (10)
		2030	Moderate (6)	Moderate (6)	High (8)
20.	Capital requirements	2010	High (8)	High (8)	Quite high (7)
		2020	Moderate (6)	Moderate (6)	High (8)
		2030	Quite low (4)	Quite low (4)	Very high (9)
21.	Production size determining profitability in firm	2010	Moderate (6)	Medium (5)	Medium (5)
		2020	Quite high (7)	Quite high (7)	Quite high (7)
		2030	High (8)	High (8)	High (8)
22.	Production size in the country	2010	Low(3)	Very low (2)	Low (3)
		2020	Medium (5)	Quite low (4)	Medium (5)
		2030	High (8)	Quite high (7)	Very high (9)

6. Conclusions

In the chapter are presented the results of interdisciplinary experimental-comparative research aimed at specifying the values of technologies for applying coatings in the processes of physical vapour deposition on the soft brass substrate against the micro- and macroenvironment. Especially the potential and attractiveness were determined of the analysed technology groups, taking into account the differences resulting from the amount of applied layers. Moreover, the recommended strategies of conduct and forecast strategic development tracks were determined, taking into account the influence of coatings applied in the processes of physical vapour deposition on the mechanical and tribological properties, as well as the resistance to corrosion of elements covered with them.

The presented results of materials science research indicate a promising improvement of the tested material's resistance to corrosion. The manufacturing of items used, in particular, in the housing sector and automotive industry, using only the PVD techniques for depositing multilayer coatings and monolayer coatings with a thin intermediate layer is a cleaner and more environmental-friendly technology. The configuration of the deposited coatings applied ensures also very good adhesion to the substrate, the desired, very high corrosion resistance, the required resistance to abrasive wear and small roughness combined with a high gloss expected for aesthetic reasons. The solution proposed allows to eliminate completely the highly harmful electroplating processes, beneficial for the improved staff working conditions and eliminating completely a hazard for the staff's health and life. Moreover, any problems related to the management of electroplating wastes are eliminated thus reducing considerably the manufacturing costs.

The corrosion current density – corrosion rate has been determined by analysing anodic polarisation curves. The curve confirms the better corrosion resistance of samples with coatings deposited with the PVD technique as compared to the sample not coated with copper and zinc alloy (12.4 $\mu A/cm^2$). The value of the corrosion current density for Ti/ZrN×150, Ti/ZrN×15 coating and Ti/CrN×150 coating deposited in the PVD process is within 0.2-0.6 $\mu A/cm^2$, which implies the good anticorrosive properties of PVD coatings, especially multilayer coatings. The results of the testes show that growth in the number of layers in a coating decreases the current density values reducing the corrosion rate. The results of impedance measurements show that the resistance of load transmission is higher for samples with coatings deposited, signifying that they fulfil their role as a diffusion barrier.

It is important to compare the properties of coatings in operating conditions. The greatest resistance to the erodent's activity is exhibited by monolayer coatings, especially Ti/CrN×1. The erosive resistance of monolayer coatings is approx. 4 times higher than of multilayer coatings. This most likely stems from the fact that the alternate soft layers of pure titanium are present. The number of layers in a coating is decisive for the density of the places where the erosion damage is initiated.

The hard PVD coatings deposited by reactive magnetron sputtering method demonstrate structure composed of fine crystallites. Examinations of the PVD coating textures reveal that in most cases they have the binary textures {111} and {100} or {110} and {311}. The optimum mechanical properties and abrasion wear resistance were obtained for monolayers, whereas increase of the number of layers to 15 or 150 results in reducing their life four times. It was found out that coatings deposited by PVD method on the brass substrate improve significantly its corrosion resistance. By employing PVD techniques for depositing coatings, the mechanical and useful properties of the parts coated with them can be improved. Depending on the expected functional properties, multilayer coatings can be used largely enhancing corrosion resistance or hard monolayer coatings resistant to abrasive wear and erosion. The work shows that a "universal" coating cannot be accomplished and the analysis of operating conditions for coatings have the main effect on its selection and use.

The final action closing the experimental-comparative works was to create a series of technology roadmaps of the analysed technology groups. The creation and use of this tool allows for presenting, in a uniform and clear format, various types of factors which directly and indirectly characterize given groups of technologies together with the forecast and perspectives of their development in different time intervals of the adopted time horizon. Technology roadmaps function as a tool of comparative analysis, facilitating the selection of the best technology in terms of the specified selected criterion, and when supplemented by operation sheets with precise technological details – they enable the implementation of a selected technology in industrial practice.

Analysing given research results man should say that the manufacturing of items used, in particular, in the housing sector and automotive industry, using only the PVD techniques for depositing multilayer coatings and monolayer coatings with a thin intermediate layer is a cleaner and more environmental-friendly technology. The configuration of the deposited coatings applied ensures also very good adhesion to the substrate, the desired, very high corrosion resistance, the required resistance to abrasive wear and small roughness combined with a high gloss expected for aesthetic reasons. The solution proposed allows to eliminate completely the highly harmful electroplating processes, beneficial for the improved staff working conditions and eliminating completely a hazard for the staff's health and life. Moreover, any problems related to the management of electroplating wastes are eliminated thus reducing considerably the manufacturing costs.

The technology of physical deposition from the gas phase presented in this work does not constitute the pool of "popular" methods, but the technologies most prospective for the world economy taking into account global trends, development tendencies as well as limitations related to the substrate material, the expected properties of the coatings produced or costs. The positive aspects connected with the attractiveness of the technology and a large potential and a future strategy for Poland should be regarded as the opportunity to achieve synergies in combination with other technologies (rapid development of hybrid technologies should be seen in the coming years), the power of positive effect for the natural environment, strategic importance of the technology for domestic and international economy, share in GDP generation, creation of jobs, deployment of the technology in the small and medium-sized enterprises sector and its impact on enterprise competition and development opportunities in the research area.

The forecast directions of the future development of PVD technologies anticipate the use of process temperatures close to 600ºC aimed at increasing the adhesion of the coating to the substrate material by obtaining a partially diffuse connection. It is anticipated that the future lies in intelligent and hybrid nanostructural coatings (including nanocomposites). Big hopes are also put on the use of modulated multilayer coatings with a large number of single layers. Special attention should be paid to coatings constructed from a small number of single layers whose concept is based on the disturbance of the column growth of crystals during their deposition in the PVD process; coatings with a large number of non-isostructural layers, as well as coatings with a large number of isostructural layers, so-called superstructures.

Summing up, it should be underlined that the foresight- materials science research described in this chapter are a fragment of broader individual actions [102-107] aimed at selecting, researching, characterizing and determining strategic development perspectives of priority innovative material surface engineering technologies in the process of technological e-foresight.

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8 Long-term development directions of PVD/CVD coatings deposited onto sintered tool materials

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Abstract

Purpose: *The purpose of this chapter is to evaluate strategic development perspectives of physical/ chemical vapour deposition of monolayer, multilayer and gradient coatings onto sintered tool materials with cemented carbides, cermets and tool ceramics substrates. The coating type was adopted as the criterion for technology division, thus obtaining eight technology groups for carried out research.*

Design/methodology/approach: *In the framework of foresight-materials science research: a group of matrices characterising technology strategic position was created, materials science experiments using high-class specialised equipment were conducted and technology roadmaps were prepared.*

Findings: *High potential and attractiveness were shown of the analysed technologies against the environment, as well as a promising improvement of mechanical and functional properties as a result of covering with the PVD/CVD coatings.*

Research limitations/implications: *Research pertaining to covering sintered tool materials with the PVD/CVD coatings is part of a bigger research project aimed at selecting, researching and characterizing priority innovative material surface engineering technologies.*

Practical implications *The presented results of experimental materials science research prove the significant positive impact of covering with the PVD/CVD coatings on the structure and mechanical properties of sintered tool materials, which leads to the justification of their including into the set of priority innovative technologies recommended for application in industrial practice.*

Originality/value: *The advantage of the chapter are results of comparative analysis of sintered tools materials with different types of coatings deposited in the PVD/CVD processes together with the recommended strategies of conduct, strategic development tracks and roadmaps of these technologies.*

Keywords: *Manufacturing and processing; Thin and thick coatings; Sintered tool materials; Foresight; Technology Roadmapping*

This chapter has been also published as:

A.D. Dobrzańska-Danikiewicz, K. Gołombek, D. Pakuła, J. Mikuła, M. Staszuk, L.W. Żukowska, Long-term development directions of PVD/CVD coatings deposited onto sintered tool materials, Archives of Materials Science and Engineering 49/2 (2011) 69-96.

1. Introduction

A recently announced "Europe 2020" strategy replacing the current Lisbon Strategy envisages comprehensive actions at a European, national and regional level aiming to support a more effective, competitive and low-emission economy based on knowledge and innovation ensuring high employment and social and territorial cohesion. One of the five strategic goals of the contemporary Europe in the field of economy provides for the growing level of R&D and innovation investments. 3% of the EU's GDP will be spent in total for this purpose until 2020 from public and private funds. For the economic and social effects achieved to be satisfactory and as expected, the above financial resources have to be invested properly in the fields of science and industries generating the highest added value in the future. The above reasons have been decisive in the last decade for widespread interest in foresight research, also in respect of material engineering, being the subject of numerous projects implemented and currently held globally [1-3] and in Poland [4-8]. An analysis of such projects' outcomes and scope combined with literature review [9] and the observation of development trends in the industry indicating the sharp growth of materials surface engineering in many technically advanced countries, set a basis for conducting extensive research aimed at identifying the priority, innovative technologies and strategic directions of development in this field [10]. The relevance and adequacy of the assessments is ensured by the synergic interaction of materials science research and foresight methods. The foresight-material science research has been carried out using the custom methodology [11] of the Computer Aided Foresight Integrated Research that organises, stream¬lines and modernises the actual process of foresight research by applying information technology [12] encompassing: a virtual organisation, web platform and neural networks. The following technologies have been investigated to date: laser treatment of hot-work steels [13], casting magnesium alloys [14] and polycrystalline silicon used for photovoltaic purposes [15]; deposition of PVD and CVD coatings, including nanostructural coatings onto a brass substrate [16]; surface manufacture of graded and composite materials including nanostructural materials with the required soft and hard magnetic properties [17] and selected methods of steel thermochemical treatment [18]. The group of high-potential technologies include also PVD and CVD with a broad spectrum of current and future applications, notably in the tool industry, especially for cutting processes.

Efforts to enhance the life of cutting tools on one hand preconditions a constant search for new materials with improved properties characteristic for i.e. sintered tool materials [19-26], on the other hand contributes to increased interest in modern PVD/ CVD coatings. World and domestic papers review shows that deposited coatings contribute to improving strength [27-30], tribological [31-37] and anti-corrosive [38-43] properties as well as higher resistance to wear [44-56] and high temperature [57-61]. A beneficial combination: a substrate - a thin PVD/CVD coating allows to achieve the unique properties of cutting tools, i.e. made of high-speed steels [62-68]. Thin coatings produced from a gaseous phase formed as a result of chemical reactions occurring in a hot substrate [69] enable to produce dense and pure layers, using materials featuring low diffusion coefficients on different substrates, also those with a complex geometry [9,70]. The nanocrystalline [71-73] and graded [70,74-78] coatings deposited in PVD processes occupy a special place amid the coatings discussed. A tool material deposited with a graded coating features a lower friction co-efficient, higher microhardness, resistance to adherence and diffusion wear and to oxidisation. This inclines to analyse such groups of technologies both, in the technical and economic context, hence requires that their long-term development prospects need to be evaluated.

The purpose of this study is a comparative analysis of various, selected technologies of physical and chemical vapour deposition differing in the type of the deposited coatings. The subject of the comparative analysis are the results of investigations into the structure and properties of sintered tool materials with the PVD/CVD coatings applied as well as the value of the individual technologies against the environment and their long-term development prospects with the recommended action strategies and the forecast multi-variant development tracks determined through expert research. The final research results show the strategic position of the analysed technologies against the surface engineering background as well as are a compact compendium of knowledge presented using technology roadmaps and technology information sheets.

2. Interdisciplinary research approach

The research efforts concerning the selected physical and chemical vapour deposition technologies differing in the type of a substrate and chemical composition, as well as the type and number of the layers of the coating deposited are of an interdisciplinary character. The foresight-materials science research method employed origins directly from organisation and management (technology foresight) as well as material engineering (surface engineering). A much broader insight into the concept was, however, required at some stages of the research, hence the following areas of detailed knowledge were also used: computer science embracing: information technology and artificial intelligence (neural networks, Monte Carlo methods); statistics; econometrics; operational studies; construction and operation of machinery; automation and robotisation of industrial processes; strategic, tactical and operation management; quality and environment management; accounting and finance.

The research has been carried out using sintered tool materials based on cemented carbides: (A) WC, Co, (B) WC, TiC, TaC, Co, cermets: (A) Ti(C,N), WC, TiC, TaC, Co, Ni, (B) Ti(C,N), TiC, TaC, WC, Co, Ni, oxide and nitride ceramics and sialon ceramics deposited in PVD and CVD processes with a wide range of monolayer, multilayer, graded coatings resistant to wear. The following eight homogenous groups have been distinguished between from the physical and chemical vapour deposition technologies on sintered tool materials for the purpose of foresight and materials science works carried out under this study by adopting the type of coatings deposited as a criterion of grouping:

(K) The physical vapour deposition of the simple monolayer coatings,

(L) The physical vapour deposition of the complex, classical monolayer coatings,

(M) The physical vapour deposition of the complex, nanocrystalline monolayer coatings,

(N) The physical vapour deposition of the multilayer coatings, number of layers $n<10$,

Table 1. *Tested materials classification scheme*

Coating type			Coating composition	Process	Symbol		Substrate
monolayer	simple		TiN	PVD	(K)		Cemented carbides: A-type: WC, Co B-type: WC, TiC, TaC, Co Cermets: A-type: Ti(C,N), WC, TiC, TaC, Co, Ni B-type: Ti(C,N), TiC, TaC, WC, Co, Ni Al_2O_3+ZrO_2 oxide ceramics Al_2O_3+TiC oxide ceramics Al_2O_3+$SiC_{(w)}$ oxide ceramics Si_3N_4 nitride ceramics SiAlON
	complex	classical	Ti(C,N) (Ti,Al)N	PVD	(L)		
		nanocrystalline	(Ti,Al)N Ti(C,N)	PVD	(M)		
multilayer	n*<10		TiN+TiC+TiN TiN+(Ti,Al,Si)N+TiN TiN+Al_2O_3 TiC+TiN	PVD	(N)		
			Al_2O_3+TiN TiN+TiC/TiN Ti(C,N)+Al_2O_3+TiC Ti(C,N)+Al_2O_3+TiN Ti(C,N)+Al_2O_3+TiN TiN+Al_2O_3+TiN TiC+Ti(C,N)+Al_2O_3+TiN TiN+Al_2O_3+TiN+Al_2O_3+TiN	CVD	(O)		
	n*≥10		TiN+multi(Ti,Al,Si)N+TiN multi(Al,Cr)N	PVD	(P)		
graded	step-graded		Ti(C,N)+ (Ti,Al)N TiN+(Ti,Al,Si)N+TiN TiN+(Ti,Al,Si)N+(Al,Si,Ti)N	PVD	(R)		
	continuous		Ti(B,N) Ti(C,N) (Ti,Zr)N (Al,Ti)N (Ti,Al)N	PVD	(S)		

* n – number of layers

(O) The chemical vapour deposition of the multilayer coatings, number of layers n<10,

(P) The physical vapour deposition of the multilayer coatings, number of layers n≥10,

(R) The physical vapour deposition of the step-graded coatings,

(S) The physical vapour deposition of continuous graded coatings.

A detailed overview and classification of tool materials with their coatings deposited in physical and chemical vapour deposition processes are presented in Table 1.

2.1. Materials science methodology

Surface topography and the structure of the coatings deposited was examined on the transverse fractures with a scanning electron microscope SUPRA 35 by Zeiss with the accelerating voltage of 5-20 kV. A side detector (SE) and InLens detector was used to produce structure images by using Secondary Electrons detection and Back Scattered Electrons detection. Notches were cut on the tested specimens with a diamond disc to obtain a brittle fracture on the tested specimens, and then they were broken and pre-cooled in liquid nitrogen.

The quantitative and qualitative analysis of the tested coatings' chemical composition was made with the EDS scattered X-ray radiation spectroscopy method using an EDS TRIDEX XM4 spectrometer by EDAX incorporated into an electron scanning microscope Zeiss Supra 35. The tests were performed with the accelerating voltage of 20 kV.

Diffraction investigations and the investigations of thin foil structures were performed with a JEM 3010 UHR transmission electron microscope by JEOL, with the accelerating voltage of 300 kV and maximum magnification of 300 000x. Thin foils were made in the longitudinal section, by cutting out approx. 0.5 mm thick inserts from solid specimens, and then discs with the diameter of 3 mm were cut out from them using an ultrasound drill. The discs were initially ground mechanically to the thickness of approx. 90 μm and an approx. 80 μm deep groove was polished. Finally, the preparations were subjected to ion thinning with a device by Gatan company.

The phase composition analysis of substrates and coatings was performed with the X-ray diffraction method with an X´Pert Pro X-ray device by Panalytical with the Bragg-Brentano configuration using the filtered radiation of a cobalt lamp with the rated voltage of 40 kV and the filament current of 30 mA. A 0.05^{o} step and with the pulse counting time of 10 seconds was assumed. As the reflexes of the substrate material and coating are overlapping and considering their intensity making it difficult to analyse the results obtained, the razing-incidence X-ray diffraction method for the primary X-ray beam using a parallel beam collimator before a proportional detector was used in further investigations to obtain more accurate information from the analysed material surface layer. As a diffraction pattern can be recorded with the low incidence angles for the beam onto the specimen surface, diffraction patterns from thin layers can be obtained by increasing the volume of the material taking part in diffusion. The diffraction patterns of graded and multilayer coatings were established for the different incidence angles of the primary beam.

Variations to the chemical concentration of coating components in the direction perpendicular to its surface and concentration changes in the transient zone between the coating and the substrate material were evaluated with tests performed with a GDOES-750 QDP glow discharge optical spectrometer by Leco Instruments. The following working conditions of the spectrometer's Grimm lamp were determined in the tests: inner lamp diameter 4 mm, lamp supply voltage 700 V, lamp current 20 mA, working pressure 100 Pa.

The thickness of the coatings deposited was measured with a "kalotest" method consisting of measuring the characteristic sizes of a crater formed through wear on the surface of the tested specimen with a steel ball with the diameter of 20 mm. A suspension of diamond grains with the diameter of 1 μm was supplied between the rotating ball and the specimen surface. The test time of 120 seconds was set. The wear size was measured through observations with an MEF4A Leica metallographic light microscope. The coating thickness was determined according to the following relationship:

$$g = \frac{D \cdot (D - d)}{4 \cdot R} \cdot 10^3 \tag{1}$$

where:

g – coating thickness, μm,

D – outer crater diameter, mm,

d – inner crater diameter, mm,

R – ball radius, mm.

5 measurements for each of the tested specimens were made to obtain the average thickness values for the measured coatings. In addition, to verify the results obtained, the thickness of the coatings was measured with a scanning electron microscope on the transverse fractures of specimens.

Surface roughness for the polished samples without coatings and with coatings was measured in two mutually perpendicular directions with a Surftec 3+ profilometer by RankTaylor Hobson. The measurement length of $l = 0.8$ mm and the measurement accuracy of ±0.02 μm was assumed. In addition, to confirm the results obtained, surface roughness measurements for the specimens were made with an LSM 5 Exciter confocal microscope by Zeiss. The R_a parameter acc. to PN-EN ISO 4287:2010 was adopted as a value describing surface roughness.

The hardness of the materials tested was determined using the Vickers method. The hardness of the coated substrates made of sintered tool materials was tested using the Vickers method according to PN-EN ISO 6507-1:2007. The microhardness tests of the deposited coatings were conducted with a Future Tech microhardness tester with the dynamic Vickers method. The load of 0.98 N (HV0.1) was used that allows to eliminate, as far as possible, the impact of the substrate on the results obtained. The measurements were made in the periodical loading and unloading mode, where the tester is loading the indenter with the set force, maintains the load for certain time, and then unloads it. Dynamic hardness is determined according to the following formula [20]:

$$DH = \alpha \cdot \frac{P}{D^2} \tag{2}$$

where:

α – a constant expressing the indenter shape impact, for Vickers α = 3.8584,

P – set load, mN,

D – indention depth, μm.

The test allows to observe variations to the plastic deformation and elastic deformation of the tested material, respectively during loading and unloading owing to the precision measuring system recording the depth of the indention formed in the individual phases of the test. The measurements were made by making 6 indentions for each of the tested specimens.

The adherence of the deposited coatings to the tested sintered tools materials was assessed with a scratch – test with REVETEST equipment by CSEM. The method consists of moving a diamond Rockwell C indenter along the specimen surface with a constant speed with the loading force rising proportionally to displacement. The tests were made within the pressing force range of 0-200 N, growing with the speed of (dL/dt) = 100 N/min at the distance of 10 mm.

The L_c critical load at which coating adherence is lost was determined according to the acoustic emission (AE) value registered during a measurement and by observing the scratches formed during a scratch test. The character of the damage was assessed based on observations with a Zeiss Supra 35 scanning electron microscope and with an LSM 5 Exciter confocal microscope by Zeiss.

Abrasive wear resistance tests and the wear factor tests for the tested coatings were performed with the pin-on-disc method with a CSEM High Temperature Tribometer (THT) linked directly to a PC allowing to define the size of load, rotation speed, radius in the specimen, maximum friction coefficient, test duration. A 6 mm wolfram carbide ball was used as a counter-specimen. The tests were made at a room temperature under the following test conditions: pressure force of $FN = 5$ N, movement speed of $v = 0.1$ m/s, radius of $r = 5$ mm.

The functional properties of the deposited coatings were determined with technological cutting tests at a room temperature. The cuttability tests of the tested tool materials without coatings and with the deposited coatings were carried out with a continuous rolling test without the use of the working cooling and lubricating liquids. The tool life of the tested inserts was determined by measuring the wear track width on the tool flank through measuring the VB average wear track width after cutting in a specific time interval. Cutting tests were interrupted when the VB value exceeded the set criterion. For uncoated tools, the test was made to achieve the wear criterion, and the test duration for tools with the deposited coatings was not shorter than for the uncoated tools, thus enabling to compare of VB wear track width after fulfilling the wear criterion by the uncoated specimen. VB measurements were made using a light microscope.

Technological cutting tests for the tested sintered tool materials was carried out under varied conditions and on the varied treated material, corresponding, as much as possible, to the operating requirements concerning the individual groups of substrate materials and also the coatings applied. A model of artificial neural networks was developed to evaluate the effect of coatings properties such as substrate adherence, microhardness, thickness and grain size on the operating life of the coated cutting edges.

2.2. Foresight methodology

In order to determine the strategic position of PVD and CVD technology against materials surface engineering, reference data was used acquired as a result of performing foresight research under the "Foresight of surface properties formation leading technologies of engineering materials and biomaterials FORSURF" project [79]. Nearly 300 independent foreign and domestic experts representing scientific, business and public administration circles who have completed approx. 600 multi-question surveys and held thematic discussions during 7 workshops took part in the FORSURF technology foresight up till now at the different stages of works. Development prospects for about 500 groups of specific technologies were analysed in the initial phase of the research including the evaluation of the state of the art, technology review and strategic analysis with integrated methods. The following scientific and research methods were applied for this purpose: extrapolation of trends, environment scanning, STEEP analysis, SWOT analysis, expert panels, brainstorming, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and static analysis. 14 thematic areas with 10 critical technologies were chosen as a result of the works, being the priority technologies of materials surface engineering with the best development prospects and/or of crucial importance in the industry in the next 20 years. A collection of 140 critical technologies were thoroughly analysed according to three iterations of the Delphi method carried out in consistency with the idea of e-foresight [12] using information technology encompassing a virtual organisation, web platform and neural networks. Neural networks were used in a novel and experimental manner to analyse the crossing influence showing relationships between the analysed trends and events likely to occur in the future within the considered timeframe.

The specific detailed technologies analysed in this chapter were evaluated based on experts opinions using the custom foresight research methodology [11]. A universal scale of relative states being a single-pole scale without zero was used in the research undertaken, where 1 is a minimum rate and 10 an extraordinarily high rate. Homogenous groups should be differentiated between for the technologies analysed in the first place according to the adopted handling procedure in order to subject such technologies to planned experimental and comparative investigations. The individual groups of the technologies were evaluated for their potential representing a realistic objective value of the specific group of technologies and for attractiveness reflecting the subjective perception of a specific technology by its potential users. The results were entered into one of the quarters of the **dendrological matrix of technology value** serving to visualise the objectivised values of the specific separated groups of technologies. A wide-stretching oak is the most promising quarter guaranteeing the future success. A soaring cypress and a rooted dwarf mountain pine may also ensure success provided an appropriate procedure is applied, which is unlikely or impossible for a quaking aspen. **The**

metrological matrix of environment influence presents graphically the results of evaluation for the influence of external positive factors (opportunities) and negative factors (difficulties) on the technologies analysed. Each of the technologies groups assessed by the experts was entered into one of the matrix quarters. Sunny spring illustrates the most favourable external situation ensuring the future success. Rainy autumn, providing a chance for steady progress, corresponds to a neutral environment and hot summer to a stormy environment where the success of a technology is risky but feasible. Frosty winter informs that technology development is difficult or impossible. The results of the expert investigations visualised with the dendrological and meteorological matrix were at the next stage of scientific work entered into the **matrix of strategies for technologies** by means of software based on the previously formulated mathematic relationships [11]. The matrix presents graphically a place of each group of technologies according to its value and environment influence intensity and identifies a recommended action strategy. The **strategic development tracks** were applied onto the technology strategy matrix consisting of sixteen fields reflecting the predicted situation of the given technology if positive, neutral or negative external circumstances occur. The forecast established concerns the time intervals of 2015, 2020, 2025 and 2030 and presents a vision of future events consisting of several variants.

3. Materials science research results

3.1. Substrates structure

Observations with an electron scanning and transmission microscope have revealed that the tested sintered tool materials are characterised by a well densed, compact structure with no pores (Figs. 1, 2). Besides, fracture surface topography for oxide and nitrogen ceramics signifies high brittleness, characteristic for oxided ceramic materials (Fig. 3). The occurrence of the relevant elements in the substrate structure of the tested sintered tool materials was confirmed with the EDS chemical composition analysis (Fig. 4).

The phase composition of the tested substrate materials according to the assumptions confirmed with the tests held with the X-ray qualitative phase analysis method. Fig. 5 presents an X-ray diffraction pattern for the Al_2O_3+TiC ceramics substrate and Fig. 6 shows an X-ray diffraction pattern for sialon ceramics.

Figure 1. *A-type sintered carbide thin foil structure*

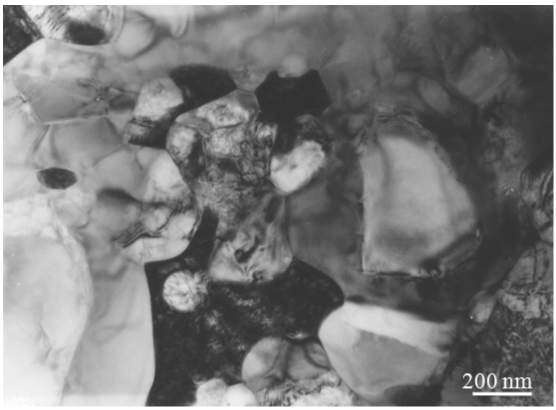


Figure 2. *A-type cermet thin foil structure*

3.2. Structure, chemical and phase composition of coatings

It was found based on the fractographic tests made with a scanning electron microscope that the coatings deposited evenly onto the tested substrate made of cemented carbides, cermet or tool ceramics are characterised by the thickness of 0.8-12.5 μm (Fig. 7, Table 2). All the PVD and CVD coatings deposited onto the substrates made of sintered tool materials are characterised by a structure free of pores, cracks and discontinuities**.** The layer structure of coatings was observed for multilayer coatings and step-graded coatings. The individual layers are characterised by a homogenous thickness within the entire observation area and tight adherence to each other and of the whole multilayer coating to the substrate (Figs. 7-10).

For the coatings deposited with the CVD method onto the substrate made of tool Si_3N_4 nitride ceramics and sialon ceramics with a TiN layer in the surface zone, the observations of the surface morphology have shown that heterogeneities exist related to the occurrence of multiple pores on the substrate and the networks of microcracks characteristic for such process. In case of Ti(C,N)+TiN and TiC+TiN coatings, single minor ball-shaped microparticles can be observed on their surface. When an Al_2O_3 layer is on the substrate surface, however, or if this layer is in the under-the-surface zone (upper TiN layer is very thin), then the particles are shaped polyhedrally (Fig. 11).

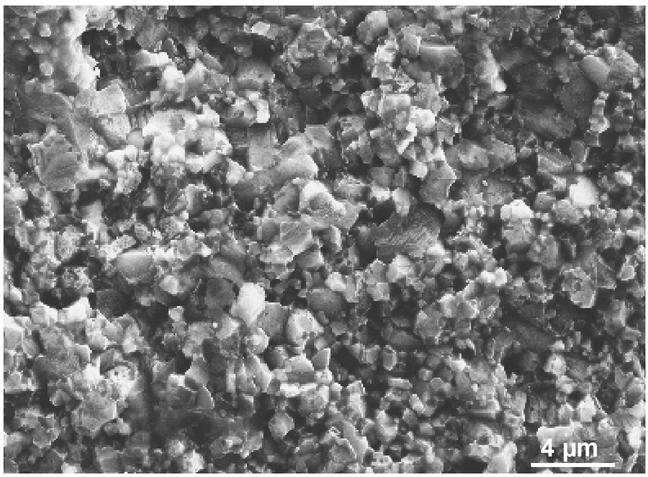


Figure 3. *Fracture surface of the Al_2O_3+TiC oxide ceramics substrate*

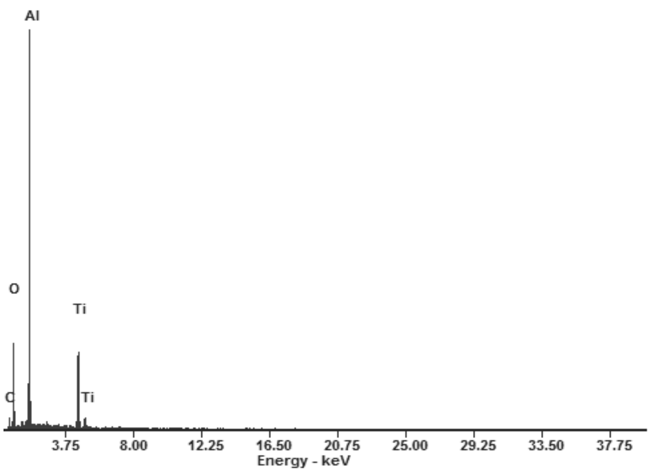


***Figure 4**. The X-ray energy dispersive plot of the area, according to Fig. 3*

The observations of morphology of coatings deposited in the PVD process on the cemented carbides, cermets and oxide ceramics substrate reveal a high heterogeneity related to the occurrence of multiple droplet-shaped microparticles (Figs. 12, 13). The morphological defects observed created when depositing a coating are most probably a result of splashing the titanium droplets removed from a titanium disc against the substrate surface as confirmed by an EDS test from microregions (Fig. 14).

The correct phase composition of the tested coatings was found as a result of thin foils tests with an electron transmission microscope, however, as the TiN and Ti(C,N) phases are isomorphic, the diffraction differentiation of each of the phases is impossible. The structure of step-graded coatings is fine-grained (as signified by, in particular, the clear rings of reflexes on diffraction images). The individual layers cannot be identified with the electrons diffraction method also for coatings containing Ti(C,N), Ti(B,N) and TiN layers due to isomorphism of such phases and the approximate network parameter value. Fig. 15 presents the structure of thin foils made of the Ti(B,N) coating on the sialon substrate and diffraction patterns. A continuous analysis between the coating and the substrate was performed in the spectrometer of the scattered X-ray radiation energy to confirm the occurrence of chemical composition gradient in the (Ti,Al)N, Ti(C,N) coatings (Figs. 16, 17). The character of changes to the concentration of the elements forming the coatings shows their gradient structure.

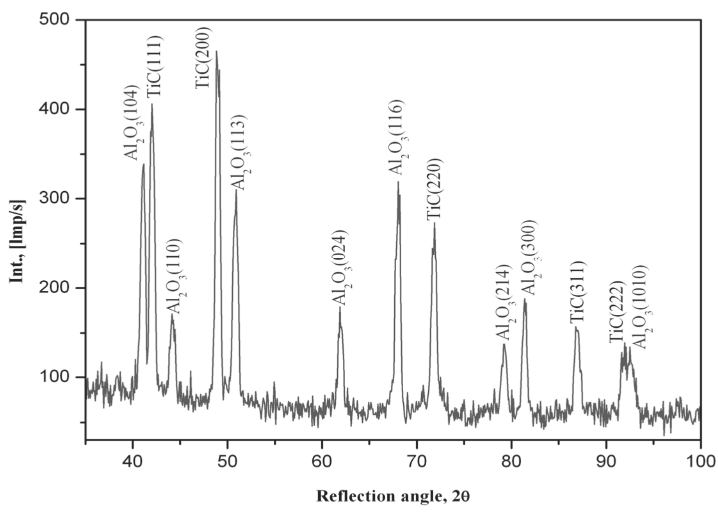


***Figure 5**. The X-ray energy dispersive plot of the Al_2O_3+TiC ceramics substrate (Bragg-Brentano geometry)*

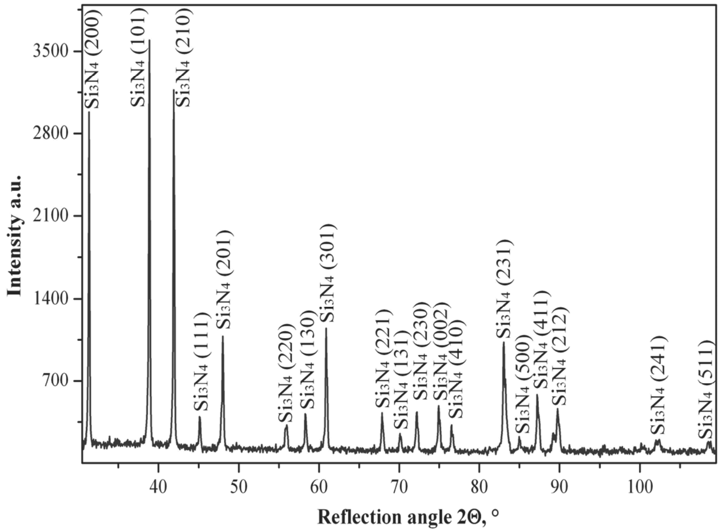


***Figure 6**. The X-ray energy dispersive plot of the sialon ceramics substrate*

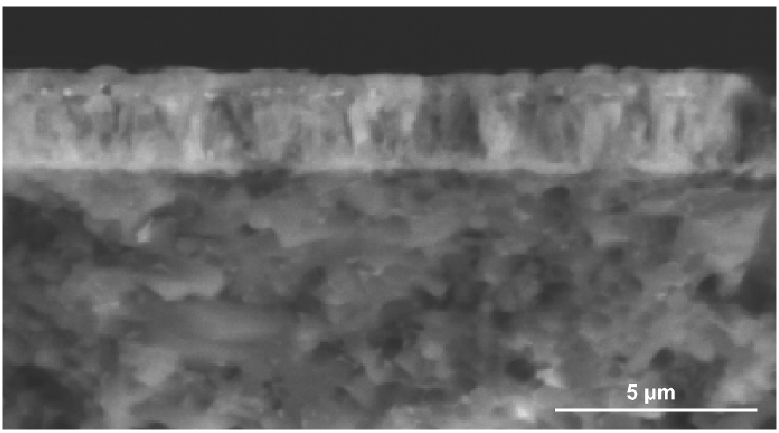


***Figure 7**. Fracture of the Ti(C,N)+TiN multilayer CVD coating deposited onto the Si_3N_4 substrate*

Figs. 18 and 19 illustrate variations to the mass concentration of coatings components and substrate material depending on the numbers of coatings deposited made with the Glow Discharge Optical Emission Spectroscopy GDOES. The investigations only allow to identify quality variations to chemical composition in the chosen microarea of each specimen. The recurrent distribution of elements forming part of the coatings and the substrate was determined.

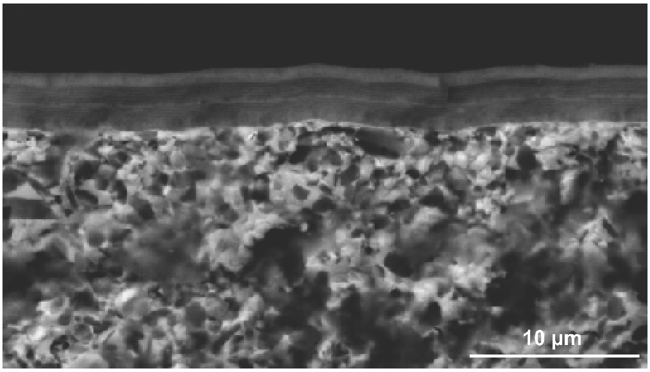


Figure 8. *Fracture surface of the TiN+multiTiAlSiN+TiN multilayer coating deposited onto the cermet substrate*

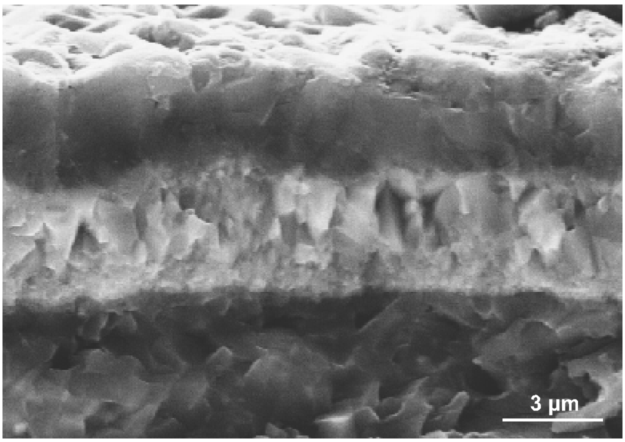


Figure 9. *Fracture surface of the Ti(C,N)+Al_2O_3+TiN multilayer CVD coating deposited onto sialon ceramics*

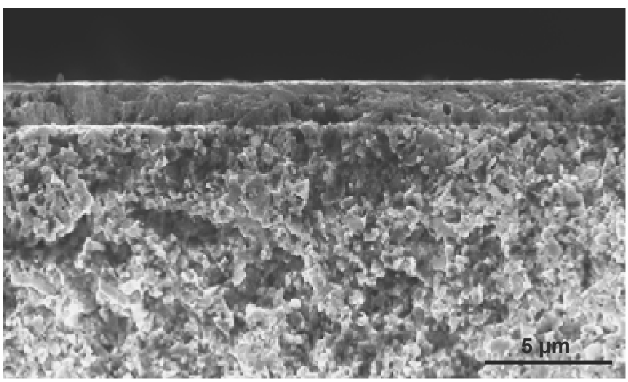


Figure 10. *Fracture surface of the Ti(C,N) monolayer, complex, nanocrystalline coating deposited onto B-type cemented carbides*

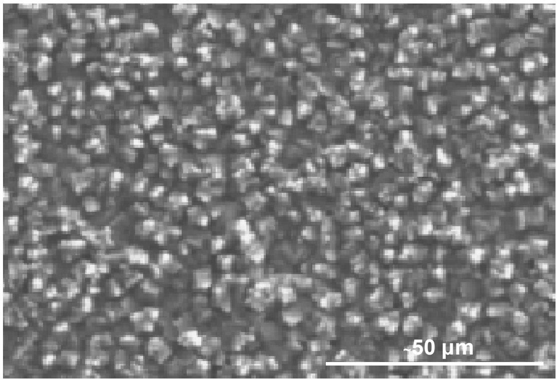


Figure 11. *Surface topography of the* $TiN+Al_2O_3$ *multilayer CVD coating deposited onto the* $Al_2O_3+SiC_{(w)}$ *substrate*

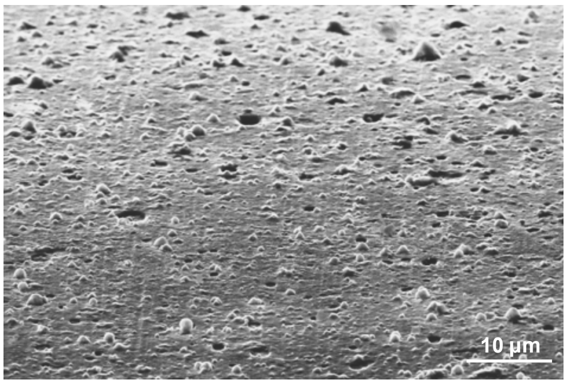


Figure 12. *Topography of the Ti(C,N) graded coating deposited onto the cermet substrate*

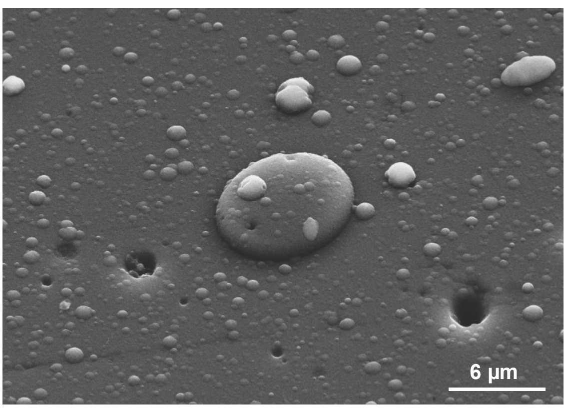
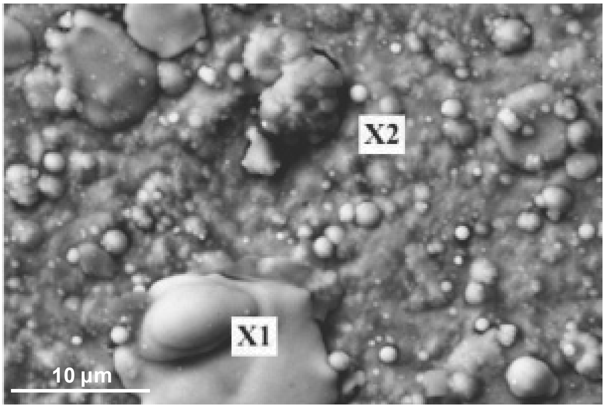
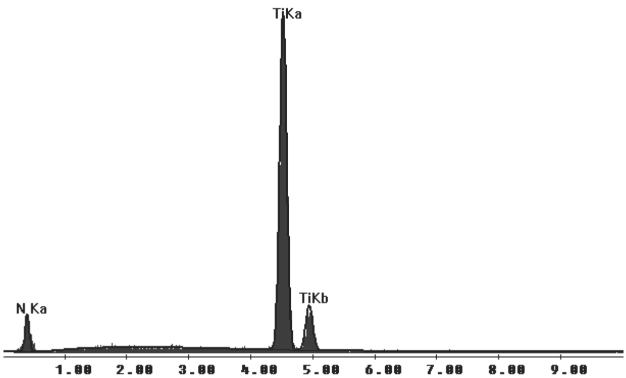


Figure 13. *Topography of the Ti(C,N) (1) continuous graded coating deposited onto sialon ceramics*

a)



b)



c)

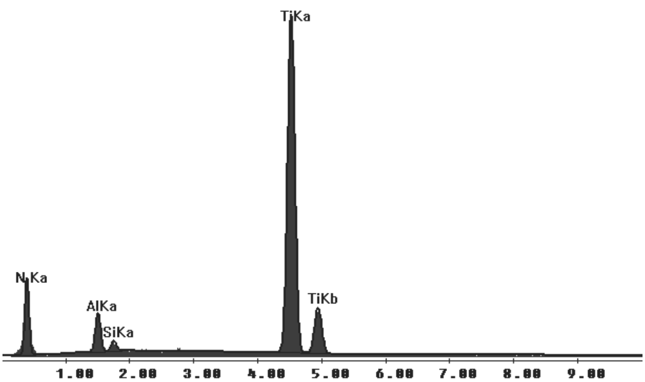
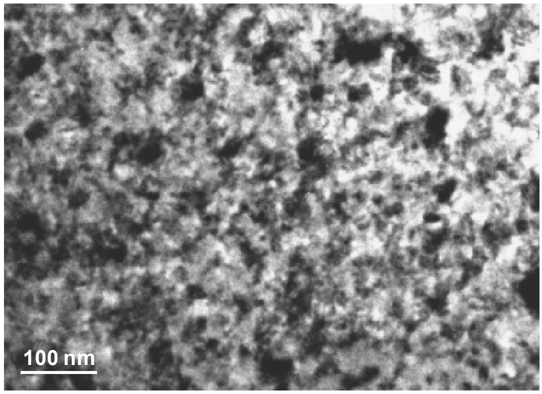
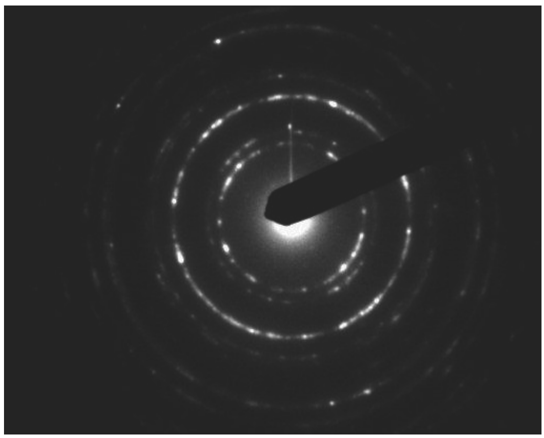


Figure 14. *a) Surface topography and the X-ray energy dispersive plot of the TiN+multi(Ti,Al,Si)N+TiN multilayer coating surface microareas deposited onto the* Si_3N_4 *nitride ceramics – b) X1 area, c) X2 area*

a)



b)



c)

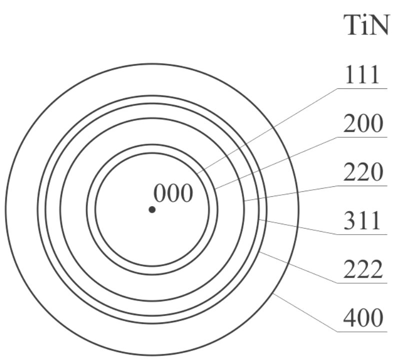


***Figure 15**. The structure of the Ti(B,N)continuous graded coating deposited onto tool sialon ceramics, a) image in light field, b) diffraction pattern from the area as in Fig. a, c) diffraction pattern solution from Fig. b*

It was determined with the X-ray qualitative phase analysis methods that, as assumed, coatings were deposited on the surface of the investigated tool materials containing TiN, TiC and Ti(C,N) type phases for the PVD coatings and TiC, Ti(C,N), Al_2O_3 and TiN phases for the CVD coatings. They could not be distinguished between according to diffraction due to the isomorphism of TiN phases with Ti(B,N), (Ti,Zr)N and (Ti,Al)N phases. The reflexes of phases coming from the substrates were also identified on X-ray diffraction patterns, especially for inserts with the PVD coatings. This results from a small thickness of the coatings applied on the investigated sintered tool materials smaller than the material penetration depth of X-ray radiation beams (Fig. 20).

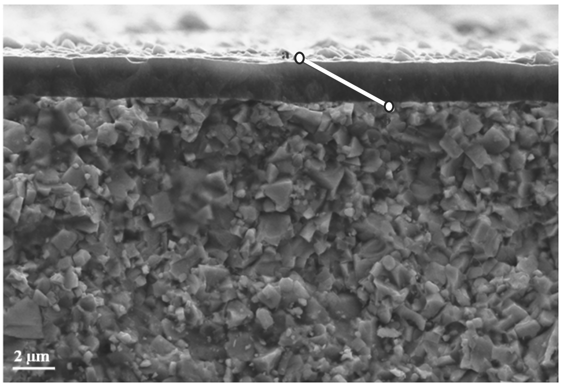


***Figure 16**. Fracture surface of the Ti(C,N) continuous graded coating deposited onto the cemented carbides*

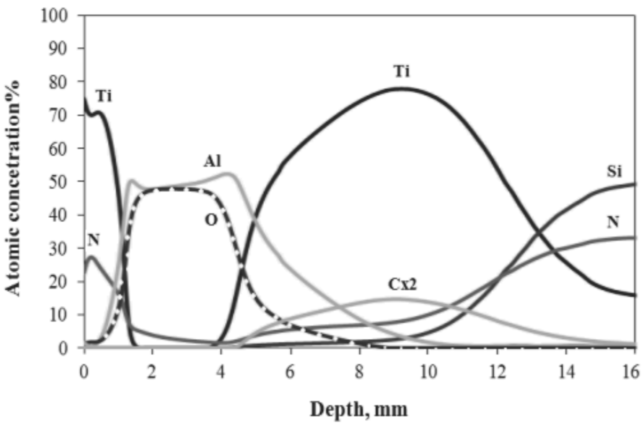


***Figure 18**. Variations to the concentration of components of the Ti(C,N)+Al_2O_3+TiN multilayer CVD coating and the Si_3N_4 nitride ceramics substrate analysed with a GDOES spectrometer*

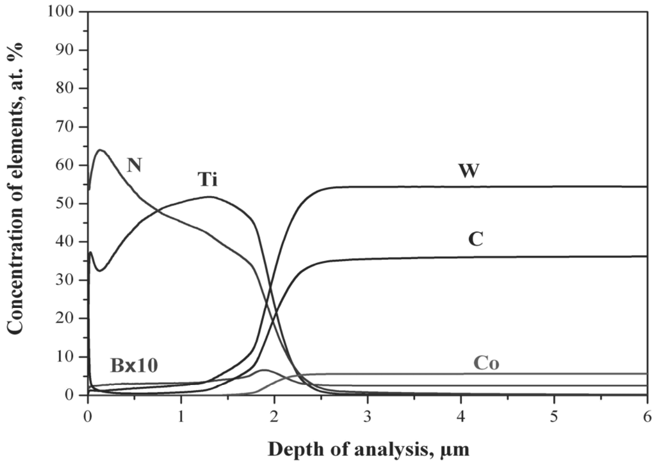


Figure 19. *Variations to the concentration of components of the Ti(B,N) multilayer coating and the substrate material made of A-type cemented carbides with a GDOES spectrometer*

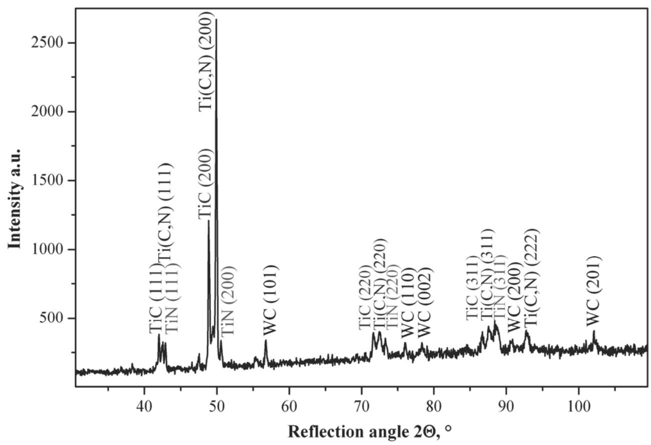
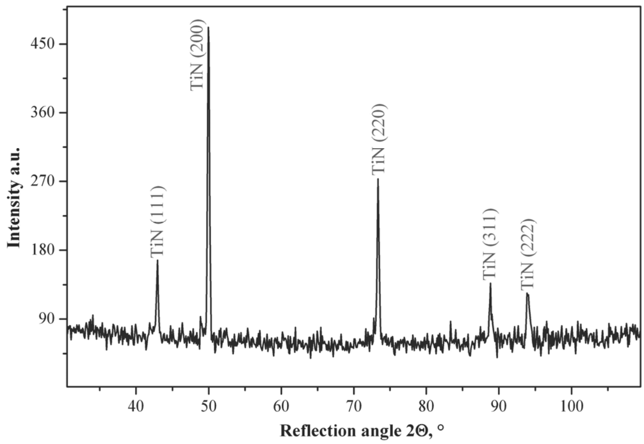


Figure 20. *The X-ray energy dispersive plot of the Ti(C,N)+TiN multilayer CVD coating deposited onto A-type cemented carbides (Bragg-Brentano geometry)*

Reflexes from the thin surface layers were only registered as a result of investigations with the GIXRD – grazing incident X-ray diffraction technique for the low incident angles of the primary X-ray beam (Fig. 21). The lack of reflexes from the phases occurring in the substrates on the diffraction patterns prepared with the GIXRD technique signify that an X-ray beam penetrating the tested coatings did not permeate to the substrate.

a)



b)

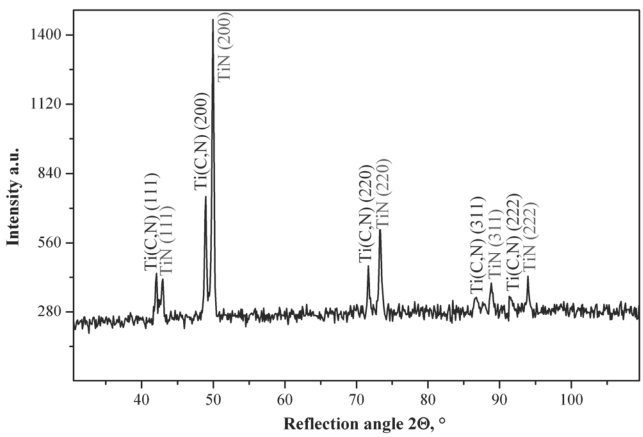


Figure 21. *The X-ray energy dispersive plot of the Ti(C,N)+TiN multilayer CVD coating deposited onto A-type cemented carbides prepared with the grazing incident X-ray diffraction technique (GIXRD): a) α = 0.1°; b) α = 4°*

3.3. Mechanical and functional properties

It was found by measuring the thickness of coatings with the "kalotest" method that that the largest thickness exhibit those of the investigated coatings deposited with the CVD method (up to 12.5 µm for the Ti(C,N)+Al_2O_3+TiC coating deposited onto the B-type cemented carbides substrate). The thickness of coatings deposited with the PVD method is below 1 µm for the majority of simple monolayer coatings and between 1.1 µm to 5.0 µm for multilayer and graded coatings. The majority of the investigated coatings is characterised by the thickness of 2-3 µm, which is an optimum value allowing to use the antiwear properties of the coating while maintaining its good adherence.

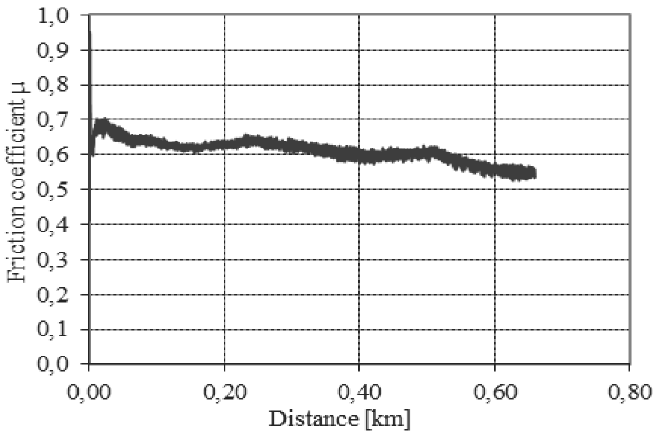


Figure 22. *Interdependence between the friction factor and the friction distance in pin-on-disc test of the TiN+(Ti,Al,Si)N+ (Al,Si,Ti)N step-graded coating deposited onto the* $Al_2O_3+SiC_{(w)}$ *substrate*

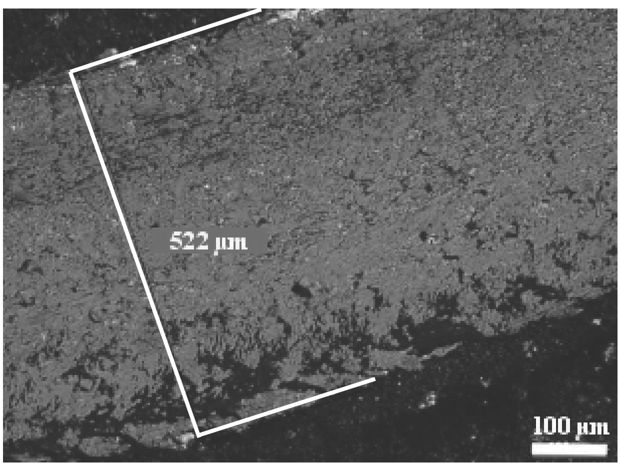


Figure 23. *Measurement of the tribological damage track width on the surface of the TiN+(Ti,Al,Si)N+(Al,Si,Ti)N step-graded coating deposited onto the* $Al_2O_3+SiC_{(w)}$ *substrate*

The roughness tests carried out have shown that the monolayer, complex nanocrystalline coatings (0.12 μm to 0.27 μm) reveal the relatively lowest roughness parameter in terms of materials with the monolayer coatings. For the multilayer coatings, the lowest roughness parameters occur for the CVD coatings except for the coatings containing Al_2O_3, where the parameter is rising due to the polyhedral nature of the coating topography. The lowest roughness parameter values for the graded coatings have been recorded for the Ti(C,N) continuous graded coatings deposited on the "B" type cemented carbides and cermet substrate. It was pointed out that the heterogeneities of the coating surface related to the presence of titanium droplets removed from the disc during the cathode arc evaporation have a significant effect on the roughness of the investigated coatings. Despite such heterogeneities, the roughness parameter values remain at the level enabling effective tool work and do not largely increase the friction factor as confirmed in pin-on-disc tests (Figs. 22, 23).

The investigated coatings are characterised by high microhardness, which is important in terms of applications due to the functions fulfilled connected with, in particular, limited mechanical wear. The largest microhardness for the monolayer coatings is exhibited by the complex monolayer coatings (the 3580 HV and 3340 HV for the (Ti,Al)N coating deposited onto the Al_2O_3+TiC and Al_2O_3+$SiC_{(w)}$ oxide ceramics substrate). The largest percentage growth in microhardness to the substrate material was noted for such materials (respectively, 85% for the (Ti,Al)N monolayer complex coating deposited onto the Al_2O_3+TiC ceramic substrate and 82% for the monolayer (Ti,Al)N complex substrate deposited onto the Al_2O_3+ $SiC_{(w)}$ ceramic substrate). In case of the multilayer coatings, the largest microhardness is exhibited by the TiN+multi(Ti,Al,Si)N+TiN multilayer coatings with more than 10 layers deposited onto the Al_2O_3+ZrO_2, Al_2O_3+TiC and Al_2O_3+ $SiC_{(w)}$ oxide ceramics substrates (respectively, 4050 HV, 3980 HV and 3970 HV). The largest growth of microhardness versus the uncoated materials was noted also for such materials (respectively, 121%, 105% and 116%). The highest microhardness for the graded coatings have the (Ti,Al)N continuous graded coatings on A-type cemented carbides, (Ti,Al)N on the Al_2O_3+TiC oxide ceramics substrate, and (Al,Ti)N on the sialon ceramic substrate (respectively 3327 HV, 3200 HV and 3600 HV). The highest microhardness increase for the graded coatings against the uncoated material was seen for coatings featuring the highest hardness (82% for the (Ti,Al)N coating on the A-type cemented carbides substrate and 77% for the (Al,Ti)N coating on the sialon ceramics substrate) but also for the (Al,Ti)N coating deposited onto the A-type cemented carbides substrate (81% increase).

It was found out that no matter if the tested coatings had been applied as monolayer, multilayer or graded coatings, the hardness of the Ti(C,N) coatings with the presence of metallic TiN and TiC phases show smaller hardness than the (Ti,Al)N or (Ti,Al,Si)N coatings where, both, metallic TiN bonds and covalence AlN bonds exist. If wear resistant coatings are deposited onto the tested substrates, a clear increase in surface layer microhardness is seen, thus contributing to the lower wear intensity of the cutting tools edge made of cemented carbides, cermets, oxide ceramics, nitride ceramics and sialon ceramics during cutting. Fig. 24 presents the highest results for microhardness measurements obtained for the individual groups of coatings.

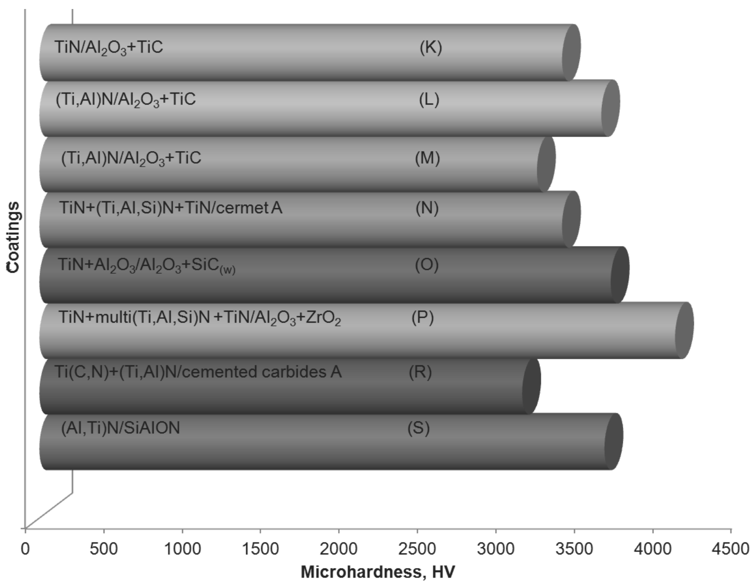


***Figure 24**. The highest results for microhardness measurements obtained for the individual groups of coatings*

The coatings subject to the research are distinctive for their good adherence to the substrate except for the single CVD coatings deposited onto the oxide ceramics substrates, which may be connected with the substrate material being very stable chemically. The best adherence for the monolayer coatings was determined for the classical complex coatings (54-99 N), in addition, very good adherence (over 40 N) was found out also for all the complex monolayer, nanocrystalline coatings. Different L_c critical load values (15 to 131 N) were recorded for the multilayer coatings, multilayer coatings with the number of layers more than 10 are characterised by very good adherence only (over 40 N) (Fig. 25). Different L_c critical load values were recorded for the graded coatings, whereas the highest adherence is exhibited by the (Al,Ti)N and (Ti,Al)N continuous graded coatings on A-type (Al,Ti)N cemented carbides on sialon ceramics. The highest results for critical load obtained for the individual groups of the coatings investigated are shown in Fig. 26.

The coating damages formed as a result of adherence tests with the scratch method were made on the basis of observations in a scanning microscope and confocal microscope (Fig. 27). It was found out as a result of the observations that the damages to the PVD coatings are of the abrasive wear nature, and are also characterised by a high number of single or double coating cracks at the scratch peripheries and delamination inside the scratch leading to coating delamination where it contacts the scratch. An increasing load during the scratch test leads to intensified cracks at the edge peripheries, leading to partial coating delamination. The periodical chipping of coatings can be also observed.

It was stated in an analysis using a model of artificial neural networks that the tool life of the sialon ceramic and cemented carbides tools deposited with the examined coatings depends mainly on the adherence of the coatings to the substrate, and hardness, grain size and grain thickness have a smaller effect on the tool life (Fig. 28).

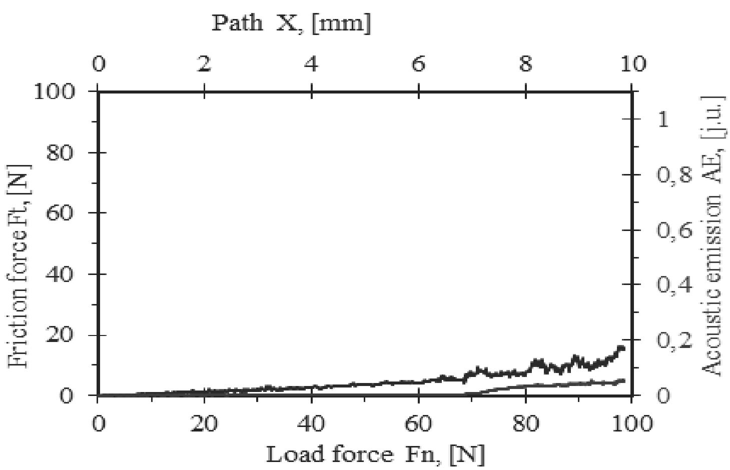


***Figure 25**. Record of scratch test curve for the TiN+multi(Ti,Al,Si)N +TiN multilayer coating deposited onto the Al_2O_3+TiC substrate*

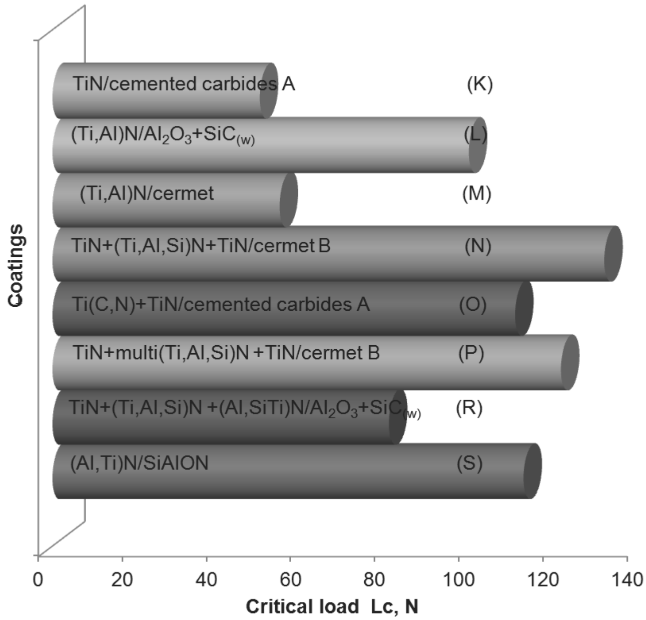
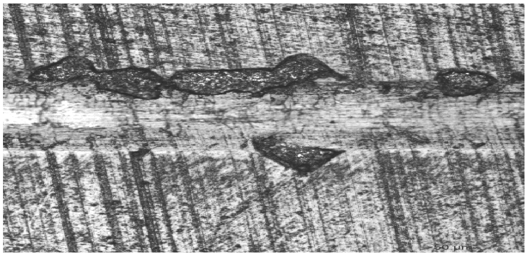


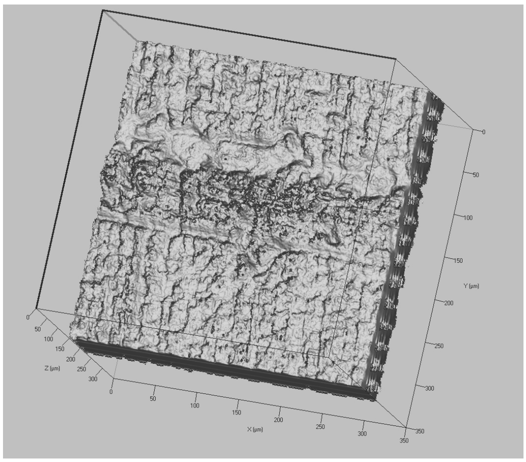
Figure 26*. The highest results for critical load obtained for the individual groups of coatings*

It was stated as a result of the carried out technological cutting tests that the TiN monolayer, simple coating deposited onto the A-type cemented carbides substrate shows the largest relative growth of tool life for the monolayer coatings as compared to uncoated material. It was found out by comparing the average growth of tool life for the specific types of the monolayer coatings that the growth is largest for the monolayer, complex, nanocrystalline coatings (300%). The very high, relative growth of tool life was also achieved for the monolayer, complex, classical coatings (140%). It should be noticed here that the high result of relative tool life growth obtained for the complex monolayer coatings is especially more important that it had been achieved for materials with a ceramic substrate which, regardless the coating applied, feature very high resistance to wear. The highest relative growth of tool life for the multilayer coatings was achieved for the multilayer PVD coatings deposited onto the A-type sintered carbide substrate with the number of layers more than 10: 2700% for the TiN+multi(Ti,Al,Si)N+TiN coating and 2150% for the multi (Al,Cr)N coating. The highest relative growth of tool life was also obtained for the multilayer PVD coatings with more than 10 layers (685%). The highest relative growth of tool life in the group of the graded coatings was achieved for the continuous (Ti,Al)N, (Al,Ti)N and Ti(C,N) graded coatings deposited

a)



b)



c)

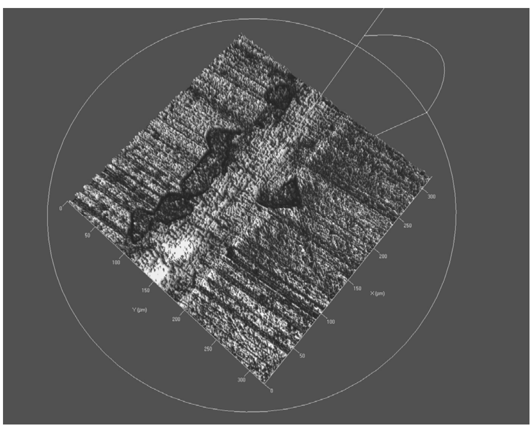


Figure 27*. a) Damage characteristic of the (Ti,Al)N monolayer, complex, nanocrystalline coating deposited onto the B-type cemented carbides substrate in a scratch test curve, b) 3D model, c) surface topography of the damaged coating, viewed with a confocal microscopy*

onto the A-type cemented carbides substrate (respectively, 2900%, 2650% and 2550%). The relative average growth of tool life for the graded coatings was also achieved for the continuous graded coatings (715%). A qualitative relationship was identified between the microhardness results collected and adherence to the substrate and the tested materials' operating properties, in particular the relative growth of wear resistance for the coated material as compared to uncoated material.

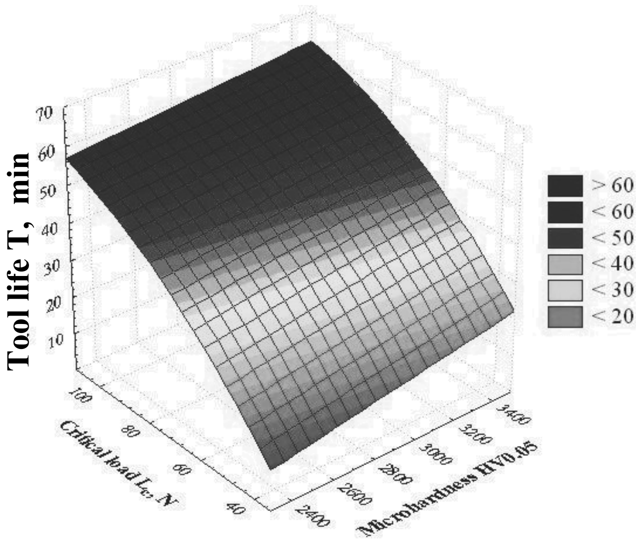


***Figure 28**. Evaluation of the effect of critical load and microhardness of the PVD and CVD coatings on tool life for the cemented carbides tools coated with the PVD and CVD coatings determined with the SSN method for the set coating depth of 2.5 μm and grain size of 9.8 nm*

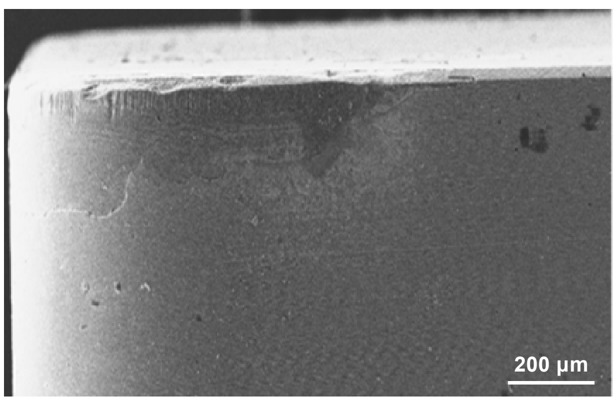


***Figure 29**. Characteristic wear of the tool flank of the Al_2O_3+TiC oxide ceramic insert with the (Ti,Al)N monolayer, complex, nanocrystalline coating deposited*

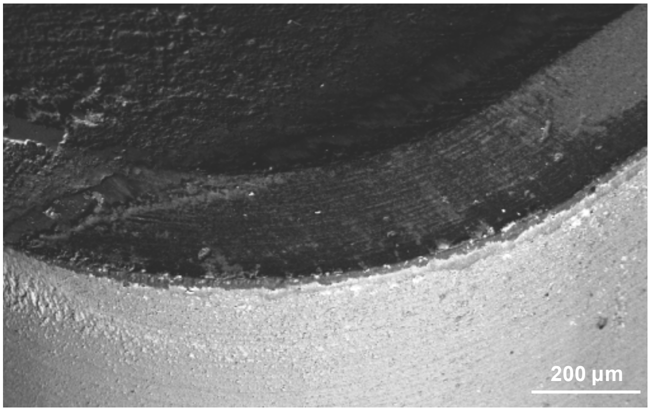


Figure 30. *Characteristic wear of the tool flank of the Si_3N_4 ceramic insert with the Al_2O_3+TiN (2) multilayer CVD coating deposited*

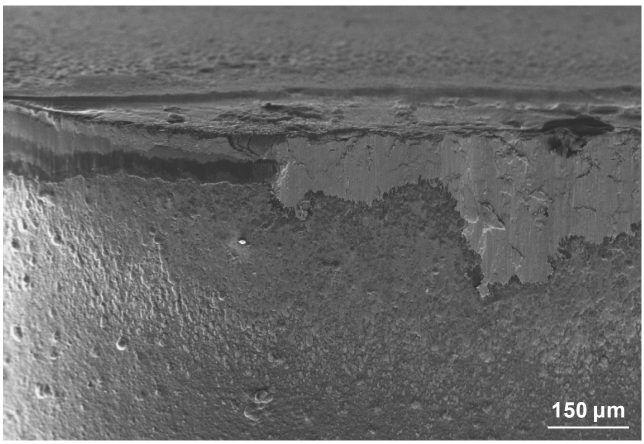


Figure 31. *Characteristic wear of the tool flank of the A-type cemented carbides plate with the Ti(C,N)+Al_2O_3+TiN multilayer CVD coating deposited*

It was found out as a result of examining the topography of a worn tool surface with a scanning electron microscope that the most common types of tribological damages identified for the examined materials are mechanical and abrasive damages to the tool flank, a crater formed at the tool flank, cracks on the tool flank and cutting edge chippings (Figs. 29-31).

Due to the cutting parameters different according to the substrate material used in the technological cutting test, it is not possible to compare the machining quality obtained for the specific groups of coatings, a qualitative relationship can be however pointed out between the mechanical properties results obtained (including microhardness and adherence), tool life and treatment quality measured as the roughness of the workpiece. The high wear resistant of the edge, combined with high coating microhardness and its good adherence to the substrate is accompanied by a relatively low roughness of the cutted material. This should be linked to the better removal of chippings ensured with the coating applied and with the limited formation of buildup on the coated surface of the edge. The detailed results of studies into the structure and properties of sintered tool materials with the PVD and CVD coatings deposited are shown in Table 2.

4. PVD/CVD development perspectives

4.1. PVD/CVD technologies versus surface engineering development

Foresight investigations with the sample size of 198 have revealed that PVD technologies have a strong strategic position among other materials surface engineering technologies. The future role of CVD technology was appraised by independent domestic and foreign experts representing scientific, business and public administration environments. According to 61% of respondents, PVD technologies are in the group of technologies with the best prospects of industrial applications, and CVD technologies according to 46% of respondents. A large group of experts maintain that scientific and research works will be frequently devoted in the nearest 20 years to the concept of physical and chemical vapour deposition. 47% and 35% of the surveyed, respectively, held such a view. Nearly a half of the surveyed (48%) claim that the thematic area of "PVD technologies" is crucial and its importance should be absolutely rising so that an optimistic scenario can come true of the country's development – "Race won" – assuming that the potential available is adequately utilised to fulfil the strategic objectives of development and so that people, statistically, are better off, social attitudes are optimistic and prospects for the coming years bright. 37% of the surveyed persons share such a view with regard to CVD technologies. 51% of the surveyed assert that the significance of PVD technologies in relation to other materials surface engineering technologies will be growing, whereas 47% maintain it will remain on the same level, with only 2% claiming that the importance will diminish over the next 20 years. The anticipated role of CVD technologies was valued somewhat lower by experts. 54% of them point out that the role of CVD technologies in the nearest 20 years in respect of other surface engineering technologies will remain at the current level, while 39% argue it will be growing and 7% think it will be shrinking. The presented results of foresight research elaborated with reference data point to the anticipated role of PVD technologies and a somewhat smaller role of CVD technologies in the development of materials surface engineering in general (mezo scale) and development of Poland's overall economy (macro scale).

4.2. Strategic position of the specified PVD/CVD technologies

The results of the foresight research described in this chapter include the assessment of the potential and attractiveness of the analysed technologies against the micro- and macro-environment performed based on the key experts' opinions expressed in a ten-degree universal scale of relative states and a recommended strategy of managing a relevant technology resulting from the assessment together with the predicted strategic development tracks. The individual technology groups have been evaluated by experts using a ten-degree universal scale of relative states for their: business, economic, humane, natural and system attractiveness as well as for their creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and a result obtained for the individual groups of technologies was entered into the dendrological matrix of technologies value (Fig. 32). An analysis made revealed that the most attractive technology having the greatest potential is the physical vapour deposition of the multilayer coatings with the number of layers of $n \geq 10$ (P), continuous graded coatings (S) and complex, nanocrystalline monolayer coatings (M) that were qualified to the matrix quarter called a wide-stretching oak. The technologies with a high potential and limited attractiveness include the physical vapour deposition of the complex, classical monolayer coatings (L) and multilayer coatings with the number of layers of $n<10$ (N) qualified to the quarter of the matrix called a rooted dwarf mountain pine. The quarter called soaring cypress with highly attractive technologies having a limited potential includes the physical vapour deposition of the step-graded coatings (R). The physical vapour deposition of the simple monolayer coatings (K) and the chemical vapour deposition of multilayer coatings with the number of layers of $n<10$ (O) was classified as the least promising technologies with both, limited potential and attractiveness, hence placed in the least promising quarter of the matrix called a quaking aspen.

***Table 2**. The results of studies into the structure and properties of sintered tool materials with the PVD and CVD coatings deposited*

Process	Coating	Substrate	Thickness, µm	Roughness R_a, µm	Hardness, HV	Hardness, ΔHV, %	Adherence, L_c, N	Cutting parameters	Tool life, min	Δ Tool life increase, %	Machining quality, R_a, µm	Application (ISO 513)
(K) Simple monolayer coatings												
PVD monolayer	TiN	Cemented carbides	2.0	0.59	2000	11	50	D	8	533	2.4	P10-P35
PVD monolayer	TiN	Al_2O_3+ZrO_2	0.8	0.37	2240	22	45	A	11	0	2.4	K01
PVD monolayer	TiN	Al_2O_3+TiC	1.2	0.21	3330	72	47	A	18	33	1.4	K01, H05,
PVD monolayer	TiN	Al_2O_3+$SiC_{(w)}$	0.9	0.30	2760	50	38	B	10	19	1.9	S15, H10
PVD monolayer	TiN	Si_3N_4	0.8	0.34	2255	19.5	20	C	8	0	2.5	K10-K20
(L) Complex, classical monolayer coatings												
PVD monolayer	Ti(C,N)	Cemented carbides	4.0	0.55	2250	18	54	D	17	425	2,1	P25-P45
PVD monolayer	(Ti,Al)N	Al_2O_3+ZrO_2	2.2	0.23	3260	78	82	A	16	45	1.2	K01
PVD monolayer	(Ti,Al)N	Al_2O_3+TiC	2.2	0.07	3580	85	80	A	20	44	1.4	K01, H05,
PVD monolayer	(Ti,Al)N	Al_2O_3+$SiC_{(w)}$	2.8	0.26	3340	82	99	B	11	38	2.4	S15, H10
(M) Complex, nanocrystalline monolayer coatings												
PVD monolayer	(Ti,Al)N	Cemented carbides	2.2	0.14	2755	57	52	F	20	700	1.4	P25-P45
PVD monolayer	Ti(C,N)	Cemented carbides	1.5	0.13	2602	48	44	F	5	100	1.9	P25-P45
PVD monolayer	(Ti,Al)N	Cermet (A)	1.5	0.13	2900	57	54	F	20	680	1.3	P05-P35
PVD monolayer	Ti(C,N)	Cermet (A)	1.5	0.12	2950	59	42	F	8	220	2.0	P05-P35
PVD monolayer	(Ti,Al)N	Al_2O_3+TiC	1.6	0.27	3170	50	53	F	21	68	1.6	K01, H05,
PVD monolayer	Ti(C,N)	Al_2O_3+TiC	1.3	0.23	2850	35	41	F	15	20	1.8	K01, H05,
(N) PVD multilayer coatings, number of layers n<10												
PVD multilayer	TiN+(Ti,Al,Si)N	Cemented carbides	3.5	0.65	3100	72	57	D	20	2000	1.8	P10-P25
PVD multilayer	TiN+(Ti,Al,Si)N	Cemented carbides	3.5	0.64	3190	67	77	D	22	550	1.1	P10-P30
PVD multilayer	TiN+(Ti,Al,Si)N	Cermet (A)	3.5	0.62	3330	33	115	D	43	307	1.3	P05-P25
PVD multilayer	TiN+(Ti,Al,Si)N	Cermet (B)	3.5	0.60	3310	35	131	D	55	323	1.4	P05-P35
PVD multilayer	TiN+TiC+TiN	Cermet (A)	5.0	0,79	3000	20	79	D	35	250	1.5	P10-P40
PVD multilayer	TiN+(Ti,Al,Si)N	Al_2O_3+ZrO_2	1.9	0.43	1900	4	40	A	15	36	1.7	K01
PVD multilayer	TiN+(Ti,Al,Si)N	Al_2O_3+TiC	1.8	0.37	2510	30	40	A	18	30	1.6	K01, H05,
PVD multilayer	TiN+(Ti,Al,Si)N	Al_2O_3+$SiC_{(w)}$	2.5	0.37	2460	34	70	B	11	31	1.4	S15, H10

Process	Coating	Substrate	Thickness, µm	Roughness R_a, µm	Hardness, HV	Hardness, ΔHV, %	Adherence, L_c, N	Cutting parameters	Tool life, min	Δ Tool life increase, %	Machining quality, R_a, µm	Application (ISO 513)
PVD multilayer	TiN+(Ti,Al,Si)N	Si_3N_4	2.0	0.45	2378	26	22	C	8	0	5.1	K10-K20
(O) CVD multilayer coatings, number of layers n<10												
CVD double	TiN+TiC/TiN	Cemented carbides	5.0	0.53	2400	26	57	D	21	525	1.8	P20-P35
CVD multilayer	Ti(C,N)+Al_2O_3+	Cemented carbides	12.5	0.51	2590	36	52	D	19	475	2.1	P10-P35
CVD multilayer	Ti(C,N)+Al_2O_3+	Cemented carbides	8.0	0.60	2300	21	78	D	23	575	2.1	P05-P30
CVD multilayer	Ti(C,N)+	Cemented carbides	8.4	0.63	2315	27	93	E	23	1050	3.7	S05-S15
CVD multilayer	Ti(C,N)+ TiN	Cemented carbides	5.0	0.40	2443	34	110	E	27	1250	4.4	S05-S15
CVD multilayer	Ti(C,N)+TiN	Al_2O_3+ZrO_2	1.5	0.25	2030	10	62	A	13	18	1.9	K01
CVD multilayer	TiN+Al_2O_3	Al_2O_3+ZrO_2	6.0	0.40	3380	84	73	A	17	54	1.9	K01
CVD multilayer	Ti(C,N)+TiN	Al_2O_3+TiC	1.1	0.07	2070	5	15	A	17	22	1.7	K01, H05,
CVD multilayer	TiN+Al_2O_3	Al_2O_3+TiC	5.8	0.29	3440	78	17	A	17	22	1.6	K01, H05,
CVD multilayer	Ti(C,N)+TiN	Al_2O_3+$SiC_{(w)}$	2.6	0.25	2250	22	40	B	9	6	1.8	S15, H10
CVD multilayer	TiN+Al_2O_3	Al_2O_3+$SiC_{(w)}$	7.9	0.30	3640	98	18	B	16	100	2.2	S15, H10
CVD multilayer	Ti(C,N)+TiN	Si_3N_4	4.2	0.15	2268	20.2	52	C	8	0	4.8	K10-K20
CVD multilayer	Ti(C,N)+Al_2O_3+	Si_3N_4	9.5	0.28	2050	9	27	C	8	0	4.6	K10-K20
CVD multilayer	TiC+TiN	Si_3N_4	5.4	0.25	2020	7	67	C	10	25	4.3	K10-K20
CVD multilayer	TiC+Ti(C,N)+Al	Si_3N_4	7.8	0.27	3025	60	32	C	8	0	5.0	K10-K20
CVD multilayer	TiN+Al_2O_3	Si_3N_4	10.0	0.45	3320	76	83	C	16	100	2.3	K10-K20
CVD multilayer	TiN+Al_2O_3+TiN	Si_3N_4	3.8	0.13	2487	31.9	48	C	15	88	3.7	K10-K20
CVD multilayer	Al_2O_3+TiN (1)	Si_3N_4	2.6	0.25	2676	41.9	45	C	16	100	3.8	K10-K20
CVD multilayer	Al_2O_3+TiN (2)	Si_3N_4	1.7	0.23	2775	47.9	27	C	14	75	2.7	K10-K20
CVD multilayer	TiN+Al_2O_3+TiN	Si_3N_4	4.5	0.60	2571	36.3	41	C	20	150	2.6	K10-K20
CVD multilayer	Ti(C,N)+	Sialon ceramics	7.0	0.82	2669	31	43	E	10	0	2.8	S05-S15
CVD multilayer	Ti(C,N)+ TiN	Sialon ceramics	2.8	0.20	2746	35	72	E	15	50	2.1	S05-S15
(P) Multilayer coatings, number of layers $n \geq 10$												
PVD multilayer	TiN+multi(Ti,Al,	Cemented carbides	4.5	0.67	3200	77	60	D	27	2700	1.1	P10-P25
PVD multilayer	TiN+multi(Ti,Al,	Cemented carbides	4.5	0.66	3280	72	90	D	33	825	1.0	P10-P30
PVD multilayer	TiN+multi(Ti,Al,	Cermet (A)	4.5	0.63	3520	40	106	D	43	307	0.7	P05-P25

Process	Coating	Substrate	Thic-kness, µm	Rough-ness R_a, µm	Hard-ness, HV	Hard-ness, ΔHV, %	Adhe-rence, L_c, N	Cutting para-meters	Tool life, min	Δ Tool life in-crease, %	Machi-ning quality, R_a, µm	Appli-cation (ISO 513)
PVD multilayer	TiN+multi(Ti,Al,	Cermet (B)	4.5	0.62	3390	38	120	D	60	352	0.8	P05-P35
PVD multilayer	TiN+multi(Ti,Al,	Al_2O_3+ZrO_2	2.3	0.37	4050	121	76	A	14	27	1.8	K01
PVD multilayer	TiN+multi(Ti,Al,	Al_2O_3+TiC	1.5	0.27	3980	105	71	A	19	42	1.4	K01, H05,
PVD multilayer	TiN+multi(Ti,Al,	Al_2O_3+Si$C_{(w)}$	2.8	0.33	3970	116	58	B	11	38	1.1	S15, H10
PVD multilayer	TiN+multi(Ti,Al,	Si_3N_4	4.0	0.44	3592	91	52	C	10	25	4.1	K10-K20
PVD multi	multi(Al,Cr)N	Cemented carbides	3.8	0.28	2867	57	96	E	45	2150	4.9	S05-S15
PVD multi	muti(Al,Cr)N	Sialon ceramics	4.8	0.31	2230	10	53	E	50	400	3.2	S05-S15
(R) Step-graded coatings												
PVD graded,	Ti(C,N)+	Cemented carbides	2.8	0.31	3076	68	39	E	15	650	4.0	S05-S15
PVD graded	TiN+(Ti,Al,Si)N	Al_2O_3+ZrO_2	2.2	0.40	2060	12	78	A	16	45	1.2	K01
PVD graded	TiN+(Ti,Al,Si)N	Al_2O_3+TiC	2.0	0.24	3040	57	77	A	16	19	2.0	K01, H05,
PVD graded	TiN+(Ti,Al,Si)N	Al_2O_3+Si$C_{(w)}$	2.5	0.32	2370	29	80	B	11	31	1.4	S15, H10
PVD multilayer	TiN+(Ti,Al,Si)N	Si_3N_4	2.5	0.32	2731	44.8	18	C	9	13	5.7	K10-K20
PVD graded,	Ti(C,N)+	Sialon ceramics	1.4	0.30	2786	37	36	E	10	0	2.2	S05-S15
(S) Continuous graded coatings												
PVD graded	Ti(C,N)	Cemented carbides (B)	2.7	0.11	2850	62	64	F	6	140	3.2	P25-P45
PVD graded	(Ti,Al)N	Cemented carbides (B)	2.6	0.14	3000	70	56	F	26	920	3.5	P25-P45
PVD graded	Ti(B,N)	Cemented carbides (A)	1.8	0.29	2951	62	34	E	15	650	4.0	S10-S15
PVD graded	(Ti,Zr)N	Cemented carbides (A)	3.0	0.30	2842	56	40	E	13	550	3.8	S05-S15
PVD graded	Ti(C,N) (1)	Cemented carbides	2.1	0.22	2871	57	49	E	13	550	4.5	S05-S15
PVD graded	Ti(C,N) (2)	Cemented carbides	2.1	0.32	3101	70	77	E	53	2550	4.2	S05-S15
PVD graded	(Al,Ti)N	Cemented carbides	2.5	0.18	3301	81	100	E	55	2650	3.9	S05-S15
PVD graded	(Ti,Al)N	Cemented carbides	3.5	0.39	3327	82	109	E	60	2900	3.5	S05-S15
PVD graded	(Ti,Al)N	Cermet (A)	3.0	0.12	3150	70	63	F	22	780	3.1	P05-P35
PVD graded	Ti(C,N)	Cermet (A)	2.6	0.11	2951	59	60	F	10	280	3.3	P05-P35

Process	Coating	Substrate	Thickness, µm	Roughness R_a, µm	Hardness, HV	Hardness, ΔHV, %	Adherence, L_c, N	Cutting parameters	Tool life, min	Δ Tool life increase, %	Machining quality, R_a, µm	Application (ISO 513)
PVD graded	(Ti,Al)N	Al_2O_3+TiC	3.2	0.24	3201	52	65	F	40	220	2.8	K01, H05,
PVD graded	Ti(C,N)	Al_2O_3+TiC	2.1	0.21	2951	40	55	F	19	52	2.4	K01, H05,
PVD graded	Ti(B,N)	Sialon ceramics	1.3	0.25	2676	31	13	E	10	0	2.2	S05-S15
PVD graded	(Ti,Zr)N	Sialon ceramics	2.3	0.40	2916	43	21	E	11	10	2.0	S05-S15
PVD graded	Ti(C,N) (1)	Sialon ceramics	1.5	0.23	2872	41	25	E	10	0	2.1	S05-S15
PVD graded	Ti(C,N) (2)	Sialon ceramics	1.8	0.38	2843	40	26	E	12	20	2.3	S05-S15
PVD graded	(Al,Ti)N	Sialon ceramics	3.0	0.15	3600	77	112	E	72	620	2.2	S05-S15
PVD graded	(Ti,Al)N	Sialon ceramics	5.0	0.28	2961	46	21	E	12	20	2.1	S05-S15

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Abbreviation	Detail marked substrate kind	Phase composition	
Cemented carbides (A) Cemented carbides (B) Cermet (A) Cermet (B)	A-type cemented carbides B-type cemented carbides A-type cermet B-type cermet	WC, Co WC, TiC, TaC, Co Ti(C,N), WC, TiC, TaC, Co, Ni Ti(C,N), TiC, TaC, WC, Co, Ni	

Cutting parameters:

A – feed of f = 0.15 mm per rev., rolling depth of a_p = 2 mm, cutting speed of v_c = 200 m/min, machined material: grey cast iron

B – feed of f = 0.2 mm per rev., rolling depth of a_p = 2 mm, cutting speed of v_c = 250 m/min, machined material: ductile cast iron

C – feed of f = 0.2 mm per rev., rolling depth of a_p = 2 mm, cutting speed of v_c = 400 m/min. , machined material: grey cast iron EN-GJL-250

D -feed of f = 0.1 mm per rev., rolling depth of a_p = 1 mm, cutting speed of v_c = 250; 315; 400 m/min., non-alloy steel C45E

E – feed of f = 0.2 mm per rev., rolling depth of a_p = 1 mm, cutting speed of v_c = 180 m/min, machined material: grey cast iron

F – feed of f = 0.1 mm per rev., rolling depth of a_p = 1 mm, cutting speed of v_c = 150 m/min, machined material: e.g. grey cast iron

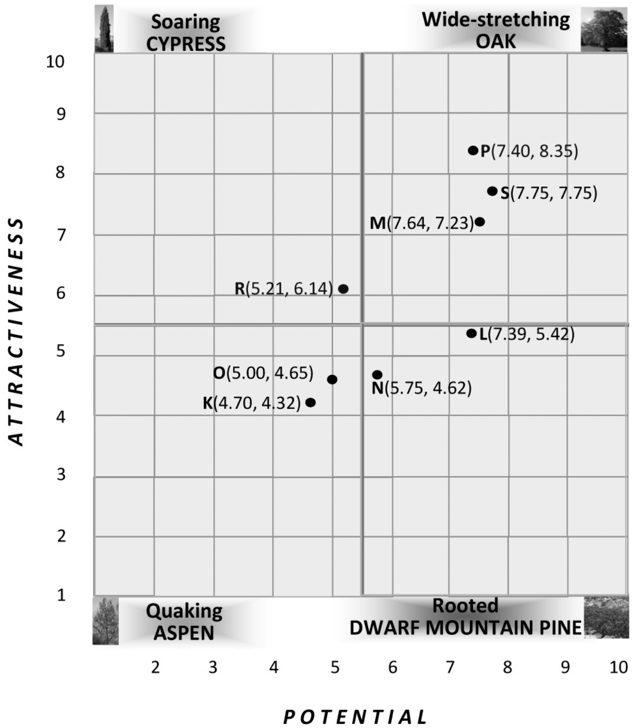

***Figure 32**. The dendrological matrix of technology value prepared for the (K)-(S) physical/chemical vapour deposition onto sintered tool materials*

The positive and negative environment influence on the relevant groups of technologies was evaluated with the meteorological matrix of environment influence. Entered into the matrix were the results of the multi-criteria analysis of the rates acquired when surveying the experts, see Fig. 33. The results of the investigations conducted show that the environment conditions are most supportive to the development of the physical vapour deposition of the continuous graded coatings (S), multilayer coatings with the number of layers of $n \geq 10$ (P) and complex, nanocrystalline monolayer coatings (M) included in the most promising quarter of the matrix called sunny spring. The macro- and micro-environment of such technologies is characterised by few difficulties and numerous opportunities which should help in their further rapid development. The environment for the other investigated groups of technologies is predictable and stable with a neutral character, therefore, no related spectacular opportunities should be expected from it, nor unpredictable difficulties which encourages regular, gradual progress.

At the next stage of research work, the results of the studies presented graphically with the dendrological matrix of technologies value and meteorological matrix environment influence were entered into the technologies strategy matrix (Fig. 34). The matrix is presenting, graphically, the place of the tested physical and chemical vapour deposition technologies onto sintered tool materials according to their value and environment influence intensity, indicating the relevant managing strategy.

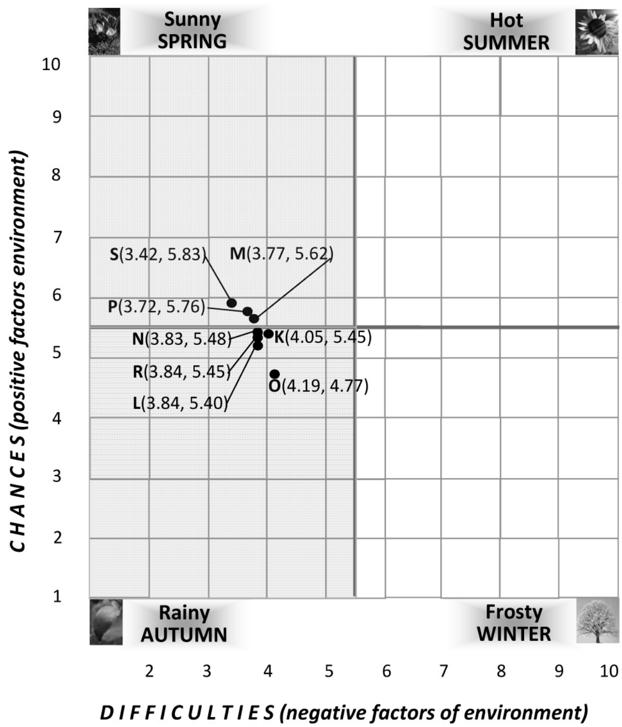

***Figure 33**. Meteorological matrix of environment influence prepared for the (K)-(S) physical/chemical vapour deposition onto sintered tool materials*

Using pre-defined mathematical relationships, the specific numerical values provided in the dendrological and meteorological matrix dimensioned [2x2] were moved to the strategy matrix for technologies dimensioned [4x4]. The circles mark the strategic development prospects of a given group of technologies expressed in numbers.

As regards (P), (S) and (M) technologies awarded 9 points in a ten-degree scale, it is recommended to use an oak in spring strategy guaranteeing the future success related to developing, strengthening and implementing an attractive technology with a large potential in favourable environment conditions. The multilayer coatings with the number of layers of $n \geq 10$ (P), continuous graded coatings (S) and complex, nanocrystalline monolayer coatings (M) deposited in the PVD process onto sintered tool materials are characterised by high microhardness limiting mechanical wear, by good adherence and a large relative tool life increase. Very good functional properties achieved as a result of applying the technology (P) encompassing both, the average values of mechanical properties as well as the best average tool life increase versus a material without the coating are most certainly connected with the using the systems of nanolayers. They allow to differentiate very well the properties in the individual zones of the coating and due to the structure designed in such a way, enable the very good adherence of individual nanolayers and the coating's adherence to the substrate material [19-21]. The technology (S) ensuring a continuous gradient of the structure and of chemical composition, achieved through the continuous change of the concentration of the individual elements forming part of the coating material of the coating surface towards the substrate also allow to achieve good mechanical and functional properties. The continuous gradient applied causes the better relaxation of internal stresses occurring in the coating and eliminates an issue of mutual adherence for individual layers existing in case of the step-graded coatings. [23,69]. The monolayer, complex nanocrystalline coatings deposited in the PVD (M) process are also characterised by very good mechanical properties translating into a high relative increase in tool life accomplished during a technological cutting test. They are also characterised by relatively the lowest roughness.

A strategy of a dwarf mountain pine in autumn recommended for the technologies (L) and (N) assumes that profits are derived from running production in a stable, predictable environment using a solid technology that should be modernised and intensively promoted to improve its attractiveness. The technology (L) evaluated most highly in this group, implemented into sintered tool materials, received 7 out of 10 available points, ensures hard coatings with excellent adherence leading to the highest average tool life characterised for such materials. Very high adherence achieved for materials with the classical, complex coatings deposited may be related to the advantageously selected substrate and coating type combinations resulting in stresses being distributed more advantageously in the zone between the substrate and coating in this group of materials. Development prospects for the technology (N) were found to be moderate (6 points). This reflects the slightly worse properties of the sintered tool materials coated with the multi-layer coatings with the number of layers of $n<10$

(N) as compared to the monolayer, classical, complex coatings (L). The technology (R): physical vapour deposition of the step-graded coatings also offers moderate development prospects (6 points) and for its the cypress in autumn strategy is recommended. The strategy consists in using a stable, predictable environment for production using an attractive technology while strengthening its potential. The issue of mutual adherence of the individual layers of the step-graded coatings needs to be solved in particular.

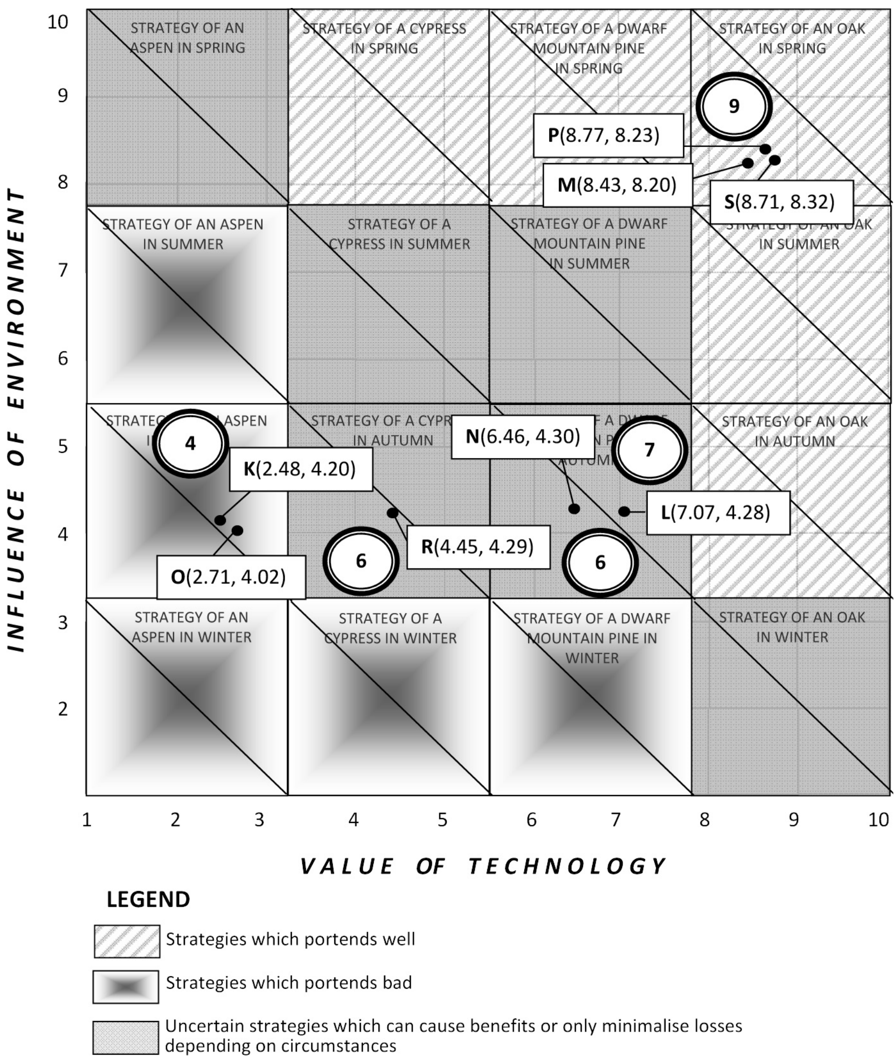

***Figure 34**. The matrix of strategies for technologies prepared for prepared for the (K)-(S) physical/chemical vapour deposition onto sintered tool materials*

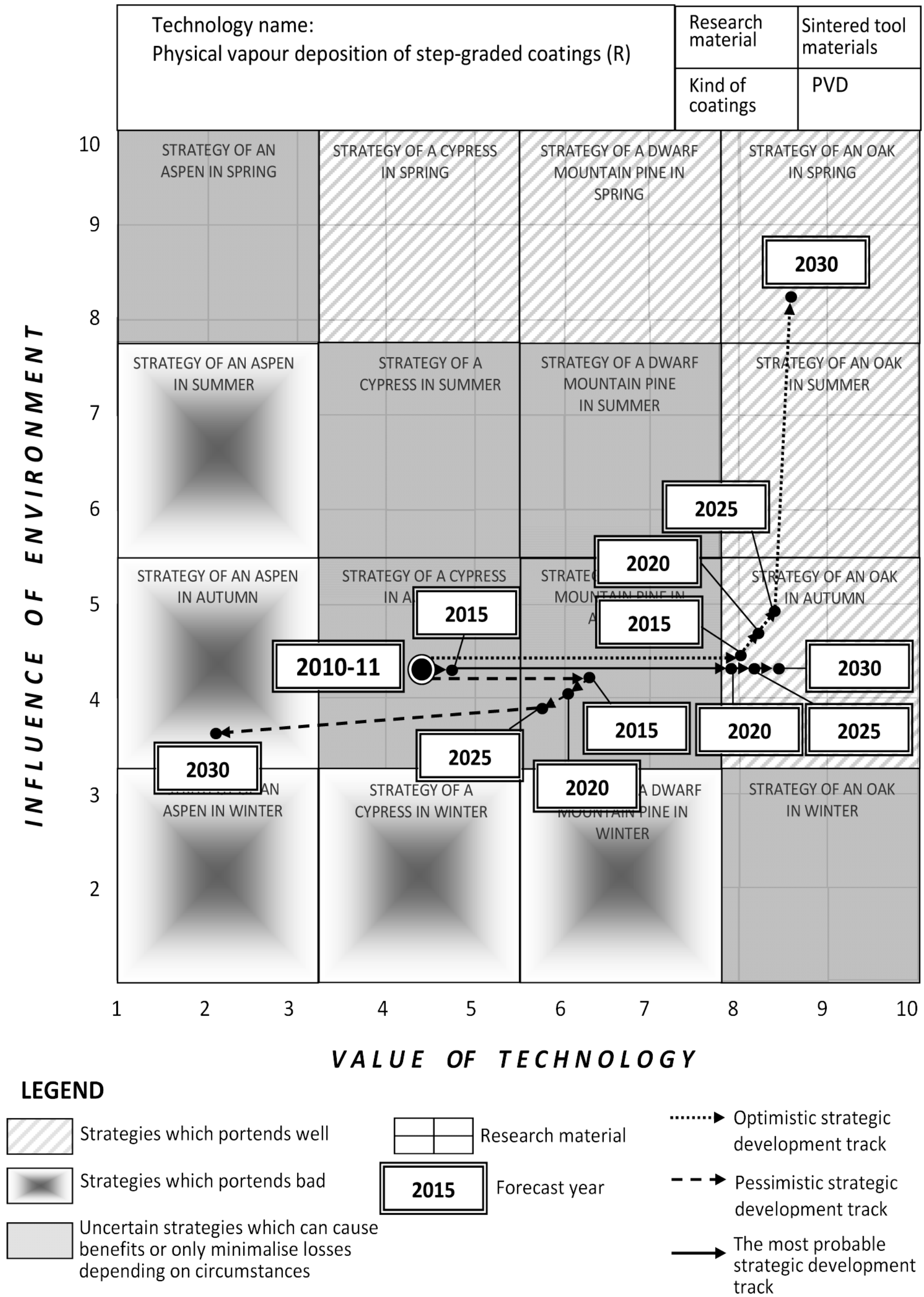

***Figure 35**. The strategic development tracks created for the (R) demonstration technology: the physical vapour deposition of the step-graded coatings*

The physical vapour deposition of the simple monolayer coatings (K) and the chemical vapour deposition of the multilayer coatings with the number of layers of n<10 (O) were found out in the group of technologies featuring the weakest development prospects with their value assessed quite low (4) in the ten-degree scale. It is predicted that in the long-term horizon, the value of such technologies will still be weakening and they will be slowly replaced with ones allowing to deposited coatings with better mechanical and functional properties.

Strategic development tracks for the individual technology groups were established based on the acquired expert opinions. The tracks represent an optimistic, most probable and pessimistic forecast of their development for the relevant time intervals of 2015, 2020, 2025 and 2030. A future success is guarantee for the technologies (P), (S) and (M). It is anticipated that they will be widely used in the nearest 20 years for manufacturing hard tools, especially cutting tools for machining metallic, non-ferrous and hard-to-machine materials for the aviation, automotive and military industry as well as for civil engineering. The macro-environmental factors, especially the overall condition of the global economy will be conditioning the rate of the forecast progress. The technologies (L) and (N) will develop at a somewhat slower rate and the rate of their future success will depend on the range of their individual, specific applications. The future strategic position of the technology (R) will depend on the advancement of works aimed at strengthening their potential. In the optimistic scenario, the technologies may be found out in the best field of the matrix in the future (oak in summer), and in the pessimistic scenario, they may be completely degraded out and forced out by more attractive technologies. The least promising technologies are (K) and (O) and if a sudden breakthrough in their development is not seen through finding new industrial uses, the technologies will most probably be backed out from the market in the nearest 20 years. A graphical example of the strategy matrix for the technologies with strategic development tracks provided in three variants created for the physical vapour deposition of the step-graded coatings (R) is depicted in Fig. 35.

5. Technology Roadmapping

On the basis of the results of the foresight–materials science research concerning critical technologies the Critical Technologies Book including technology roadmaps [80-82] and technology information sheets has been prepared (Fig. 36). The set-up of the custom technology roadmap corresponds to the first quarter of the Cartesian system of coordinates. Three time intervals for the years of: 2010-11 (current situation), 2020 (goals fulfilment methods), 2030 (long-term objectives) are provided on the axis of abscissa. A time horizon for the overall foresight research pursued with its results applied onto the map is 20 years. Seven main layers were applied onto the axis of coordinates of the technology roadmap: time layer, concept layer, product layer, technology layer, spatial layer, staff layer and quantitative layer, made up of more detailed sub-layers. The main technology roadmap layers were hierarchised starting with the top, most general layers determining all-social and economic reasons and causes of the actions taken, through the middle layers characterising a product and its manufacturing technology, to the bottom layers detailing organisational and technical matters concerning the place, contractor and costs.

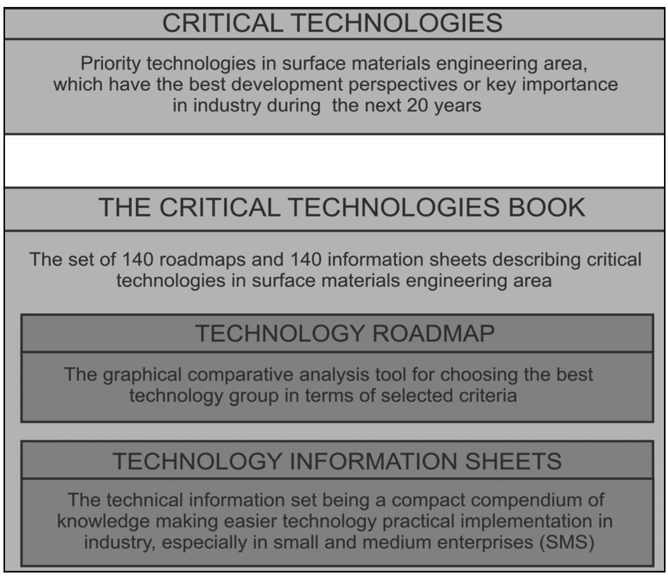

Figure 36*. The Critical Technologies Book content*

The relationships between the individual layers and sub-layers of the technology roadmap are presented with the different types of arrows representing, respectively, cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow.

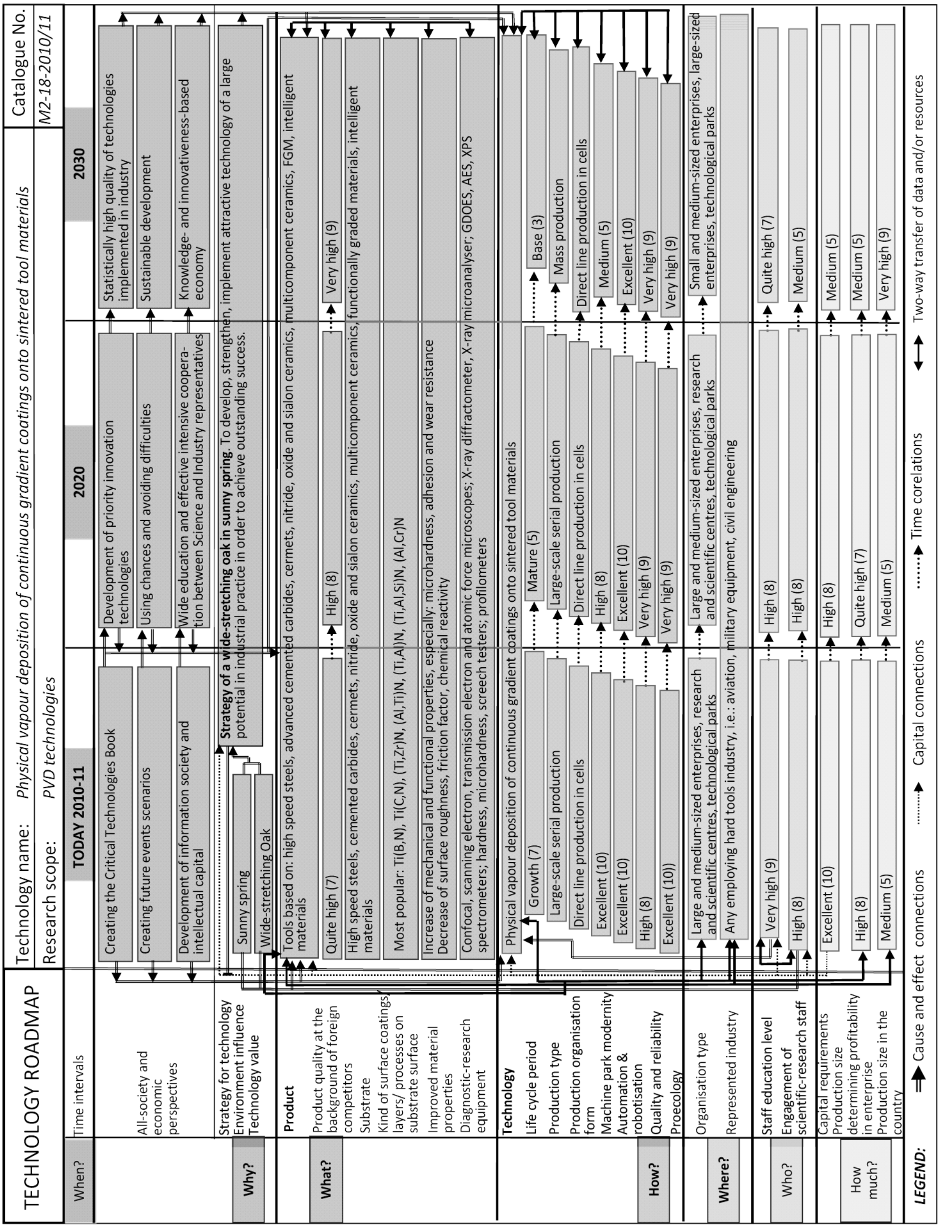

***Figure 37**. An example technology roadmap made for the physical vapour deposition of the continuous gradient coatings onto sintered tool materials*

Table 3. *Selected main source data used for preparation of technology roadmaps for investigated sintered tool materials with deposited coatings*

Technology symbol	Analysed factors																								
	(1)			(2)			(3)			(4)			(5)			(6)			(7)			(8)		
	Time horizon																								
	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c
(K)	6	3	1	9	7	3	7	7	6	10	9	9	8	7	5	8	6	4	8	6	4	6	5	5
(L)	7	5	3	10	8	5	8	9	9	10	9	9	9	7	5	9	7	5	8	7	5	6	7	8
(M)	7	5	3	10	8	5	8	9	9	10	9	9	9	8	7	10	8	5	8	7	5	5	5	8
(N)	7	4	2	9	7	4	9	8	7	9	8	7	9	8	7	10	8	5	8	7	5	6	7	6
(O)	7	4	2	9	7	4	8	9	9	10	9	9	9	8	7	9	7	4	8	7	5	3	4	5
(P)	7	5	3	10	8	5	8	9	9	10	9	9	9	8	7	10	8	5	8	7	5	3	4	5
(R)	7	5	3	9	6	5	8	9	9	10	9	9	9	7	6	10	8	5	8	7	5	4	5	7
(S)	7	5	3	10	8	5	8	9	9	10	9	9	9	8	7	10	8	5	8	7	5	5	5	8

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Technology symbol	Analysed factors	Time horizon
(K) The physical vapour deposition of the simple monolayer coatings (L) The physical vapour deposition of the complex, classical monolayer coatings (M) The physical vapour deposition of the complex, nanocrystalline monolayer coatings (N) The physical vapour deposition of the multilayer coatings, number of layers *n*<10 (O) The physical vapour deposition of the multilayer coatings, number of layers *n*<10 (P) The physical vapour deposition of the multilayer coatings, number of layers *n*≥10 (R) The physical vapour deposition of the step-graded coatings (S) The physical vapour deposition of the continuous graded coatings	(1) Live cycle period (2) Machine park modernity (3) Quality and reliability (4) Proecology (5) Staff education level (6) Capital requirements (7) Production size determining profitability in enterprise (8) Production size in the country	a: 2010-11 years b: 2020 year c: 2030 year

Note: Research results are presented in universal scale of relative state, where:1 is minimal and 10 is excellent level.

Fig. 37 provides a representative technology roadmap prepared for the physical vapour deposition of the continuous graded coatings (S), whereas Table 3 presents an aggregate list containing selected data being an extract from all the technology roadmaps developed under this chapter concerning sintered tool materials with the PVD/CVD coatings deposited. Technology information cards containing technical information very helpful in implementing a specific technology in the industrial practice, especially in small- and medium-size enterprises (SMEs) not having the capital allowing to conduct own research in this field, are detailing and supplementing the technology roadmaps.

6. Summary

This work presents the results of interdisciplinary, experimental and comparative research over sintered tool materials based on cemented carbides, cermets, oxide and nitride ceramics and sialon coated with hard layers coatings in the physical/ chemical vapour deposition processes. Eight homogenous groups of technologies (K)-(S) have been distinguished between for the purpose of the research by adopting physical or chemical vapour deposition onto sintered tool materials as a criterion of grouping. The materials science part of the work included in particular investigations into the structure of sintered tool materials, tests of structure, chemical composition and the phase composition of coatings and the testing of mechanical and functional properties of coatings. A comparative analysis was made for the results of the investigations, with special consideration given to roughness, microhardness, adherence of coatings and tool life increase relative to the material without a coating deposited during a technological cutting tests. A strategic position of the PVD and CVD strategies for the materials surface engineering was identified for foresight research along with a value against the environment of the individual, separated groups of physical and chemical vapour deposition technologies for sintered tool materials (K)-(S). The results of the investigations are presented graphically using a set of matrices. The long-term strategies recommended for implementation with regard to the individual, separate technologies are also presented. Technology roadmaps were prepared at the final stage of works illustrating, in a concise manner, basic information on the technologies analysed. The analysis made has shown that the physical vapour deposition of the multilayer coatings with the number of layers of $n \geq 10$ (P), continuous graded coatings (S) and complex, nanocrystalline monolayer coatings (M) with their success being certain have the best long-term prospects. It is predicted that they will be widely used in the nearest 20 years for producing hard tools, especially cutting tools for machining metallic, non-ferrous and hard-to-machine materials for the aviation, automotive, military industry and for civil engineering. Somewhat slower but regular growth of the physical vapour deposition of the complex, classical monolayer coatings (L), the multilayer coatings with the number of layers of $n<10$ (N) and the step-graded coatings (R) is foreseen, depending on the environment conditions and the individual, specialised uses of such technologies. The least promising technologies include the physical vapour deposition of the simple monolayer coatings (K) and the chemical vapour deposition of the multilayer coatings with the number of layers of $n<10$ (O) to be forced out by other technologies allowing to achieve better mechanical and functional properties. When evaluating the importance of the state-of-art hard abrasive wear coatings deposited in PVD/CVD processes onto sintered tool materials presented in the chapter, their broad scale of future applications in the industry should be emphasised. Hence, they will be very important in the nearest 20 years amongst other technologies of engineer materials surface engineering, which justifies their position in The Critical Technologies Book.

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9 Evaluation of selected steel thermochemical treatment technologies using foresight methods

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Abstract

Purpose: *The purpose of this chapter is to evaluate the development efficiency of classical steel thermochemical treatment. The criterion assumed for dividing the technologies into groups was the thermochemical treatment kind. Three technology groups were selected to realised research, as follows: nitriding, carburising and diffusion boriding.*

Design/methodology/approach: *In the framework of foresight-materials science research: a group of matrices characterising technology strategic position was created, materials science experiments using: light microscope, transmission and scanning electron microscopes, X-ray diffractometer, microhardness tester, work-stands for testing of thermal fatigue resistance and mechanical fatigue strength, abrasion and corrosion resistance were conducted and technology roadmaps were prepared.*

Findings: *The outcarried research pointed out the great industrial importance of nitriding and carburising and good perspectives for these technology groups. However, diffusion boriding is obsolete and will slowly leave the market.*

Research limitations/implications: *Research concerning steel thermochemical treatment constitute a part of a larger research project aimed at identifying, researching, and characterizing the priority innovative technologies in the field of materials surface engineering.*

Practical implications: *Nitriding and carburising with their popularity and good quality-price relation can be recommended for use in small and medium enterprises. Obsolete diffusion boriding is not recommended for that.*

Originality/value: *The value of this chapter is to evaluate the value of thermochemical treatment technologies in the background environment with their future development perspectives determination including the influence of thermochemical treatment on the quality, microstructure and properties of surface layers obtained by thermochemical treatment.*

Keywords: *Manufacturing and processing; Thermochemical treatment; Carburising; Nitriding; Boriding; Foresight; Technology Roadmapping*

This chapter has been also published as:

A.*D. Dobrzańska-Danikiewicz, E. Hajduczek, M. Polok-Rubiniec, M. Przybył, K. Adamaszek, Evaluation of selected steel thermochemical treatment technology using foresight methods, Journal of Achievements in Materials and Manufacturing Engineering 46/2 (2011) 115-146.*

1. Introduction

The European Union's priority strategy set out in the recent years called Europe 2020 assumes that the development of the continent should be intelligent, supportive to social inclusion and sustainable. The sustainable development idea is presented in Fig. 1. In line with the concept, it is necessary to take extensive actions at the European, national and regional level, to support a more effective, competitive and low-emission economy based on knowledge ensuring high employment and social and territorial cohesion. Five quantitative social objectives have been formulated to implement the adopted development strategy that should be brought into life until 2020. The objectives apply, accordingly, to: high employment, higher R&D and innovation investments, mitigation of the adverse climate change effects and the improved utilisation of energy sources, including RES, more widespread education and shrinking poverty and social exclusion. The Cohesion Policy concentrating on a financial aid for the EU's individual regions is promoting enterprises, including SMEs, being innovative, education and information-communicational systems, managing consciously their knowledge as a strategic resource while taking into account the environment influence. It is crucial in this context to focus scientific research in a prioritised manner on the most promising fields and disciplines of science likely to have a large impact on Poland's fast civilisational and economic development based on an information society. It is feasible to put the so-defined objectives and plans into life using the concept of e-foresight [1] and a custom methodology of the Computer-aided Integrated Foresight Research [2, 3] that organises, streamlines and modernises the actual foresight research process. The approach proposed can be implemented practically by developing an information technology including: a virtual organisation, web platform and neural networks.

Figure 1. *Sustainable development*

Thermochemical treatment methods have been long used for producing surface layers on different substrates, including especially metal substrates. They represent one of the most classical methods of formulating the structure and properties of surface in products manufactured using engineering materials [4-7]. The chemical composition and structure of the alloy surface layer is changing, hence the properties of the treated pieces change in such case due to temperature variations and the chemical effect of the medium. This causes the intended diffusion change of the surface layer chemical composition and improves the relevant useful properties of whole parts. Despite the fact that most of the technical issues relating to the technologies have been investigated long ago, some of them continue to be used commonly in the industrial practice [8-33]. This obviously inclines to analyse this group of technologies both in technical and economic terms, thus requiring to assess their development efficiency.

The purpose of this work is to compare the efficiency of the various selected structure and properties formulation technologies accomplished through thermochemical treatment for the selected engineering materials, using the harmonised chosen knowledge and technology management methods [1, 2], in order to develop technology foresight in this area while taking into account the results of thorough material science studies justifying the development preferences of the analysed technologies. The surface layers tests of the selected machine steels, hot-work tool steels and high-speed steels were carried out in this work to achieve the set goal. The steels were subjected to, respectively, nitriding, carburising, diffusion boriding and the impact of such operations was identified in particular on some useful properties of the products treated in this manner. Considering the myriad of research alternatives available, the materials and technologies mentioned above were selected, first and foremost due to a broad range of heat treatment temperatures preceding thermal and chemical treatment, starting with almost the lowest possible austenisation temperatures for machine steels to the highest ones used for high-speed steels, and secondly due to the fact that thermochemical treatment is used after quenching and tempering as for nitriding and directly after or during quenching as for carburising or boriding. The results of some earlier internal tests [34-45] being performed for many years at the Institute of Engineering Materials and Biomaterials of the Silesian University of Technology were employed in order to demonstrate the possibility of shapening the structure and properties of the selected steel grades using thermochemical treatment methods.

2. Research scope and subject matter

The research conducted is of an interdisciplinary character, and the research methodology employed is primarily concerned with technology foresight [46, 47] being part of the field of science known as organisation and management and surface engineering forming part of the widely-understood materials science. Methods originating from artificial intelligence, statistics, information technology, machine construction and operation, strategic and operational management have also been applied at some stages of the research. The key methodological assumptions of the research are illustrated graphically in Fig. 2.

2.1. Foresight methodology

According to the handling procedure accepted [2, 48, 49], homogenous groups should be distinguished between in the first place for the technologies assessed in order to subject them to the planned research of an experimental and comparative character. The dendrological matrix of technology value is used to determine the objectivised values of the relevant separated technologies or groups thereof, and the meteorological matrix of environment influence for determining the degree of the positive and negative environment influence on the specific technologies. The methodological structure of the both matrices refers to portfolio methods commonly known in management sciences, and most of all the BCG matrix [50]. Their unparalleled popularity derives from reference to simple associations and intuitive reasoning becoming an inspiration when elaborating methodological assumptions for the dendrological and meteorological matrix [2]. A ten-degree universal scale of relative states presented in Table 1 was used to assess the individual groups of technologies for their value and environment influence degree.

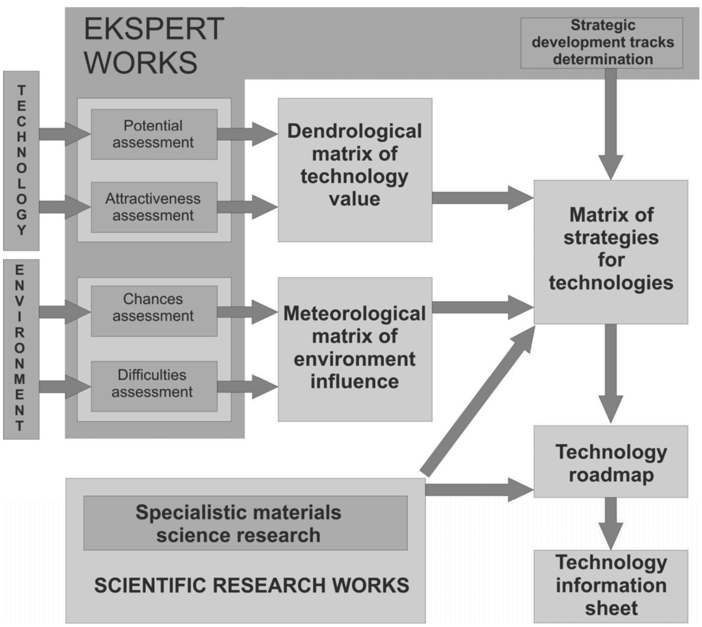

Figure 2. *Methodology of interdisciplinary foresight-materials science research*

The dendrological matrix of technology value presents assessment results for the relevant technology groups according to the potential being the actual objective value of the specific technology and attractiveness reflecting the subjective perception of the relevant technology by potential users. Depending on the potential value and attractiveness level determined in an expert assessment, each of the analysed technologies is placed into one of the following matrix quarters:

- Quaking Aspen – a weak technology with limited potential and attractiveness with the future success uncertain or impossible,
- Soaring Cypress – a technology with limited potential but high attractiveness with the future success possible,
- Rooted Dwarf Mountain Pine – a technology with limited attractiveness but high potential with the future success possible,
- Wide-stretching Oak – a technology characterised by high potential and attractiveness guaranteeing future success.

Table 1. *Universal scale of relative state [2]*

NUMBER	Class discriminant	LEVEL	
			perfection
10	0.95	EXCELLENT	
9	0.85	VERY HIGH	
8	0.75	HIGH	normality
7	0.65	QUITE HIGH	
6	0.55	MODERATE	
5	0.45	MEDIUM	
4	0.35	QUITE LOW	mediocrity
3	0.25	LOW	
2	0.15	VERY LOW	
1	0.05	MINIMAL	

The meteorological matrix of environment influence illustrates graphically the results of influence of external circumstances on the relevant group of technology grouped by the difficulties with negative influence and the opportunities with positive influence on the analysed technologies. Depending on the influence degree of positive and negative environment factors determined in an expert assessment, each of the analysed technologies is placed into one of the following matrix quarters:

- Frosty Winter – the environment produces many difficulties and few chances, thus the success is difficult or impossible,
- Hot Summer – the environment produces many chances and many difficulties, thus the success of the technology in given circumstances is highly risky, but possible,

- Rainy Autumn – the environment is neutral with few difficulties and chances for steady progress,
- Sunny Spring – the environment is friendly with many chances and few difficulties guaranteeing the future success.

The results of expert studies visualised with a dendrological and meteorological matrix were applied in the next stage of research works applied onto the **technology strategies matrix** consisting of sixteen fields corresponding to the individual variants from the set of combinations of technology types with environment types. Mathematic relationships were formulated after entering the concepts of the relative technology value V_n and the relative value of environment influence E_n and a computer programme based on them was created enabling to transfer the specific numerical values from the dendrological and meteorological matrix dimensioned [2x2] to the strategy matrix for technologies dimensioned [4x4] [2]. The matrix of strategies for technologies presents graphically the place of technology including its value and environment influence degree and indicates an action strategy to be adopted with reference to the specific technology considering the factors analysed earlier. The **strategic development tracks** were applied onto the technology strategy matrix consisting of sixteen fields reflecting the predicted situation of the given technology if positive, neutral or negative external circumstances occur. The forecast established concerns the time intervals of 2015, 2020, 2025 and 2030 and presents a vision of future events consisting of few variants.

2.2. Tests material

The materials science investigations were carried out with the selected steel grades with their chemical composition as provided in Table 2. 18CrMnTi4-4 steel is intended for carburising, 38CrAlMo6-10 is machine steel for nitriding, 37CrMoB10-4 is low-alloy hot-work tool steel, X37CrMoV5-1 and X40CrMoV5-1 are Cr-Mo hot-work tool steels of 5-1 type characterised by high resistance to cyclic temperature variations. 40CrWMoVB17-11-16 is multi-component steel and HS6-5-2 and HS12-0-2+C are high-speed steels. The steels were alloyed with a conventional method in electric-arc furnaces and X37CrMoV5-1(vac), X40CrMoV5-1(vac) and 40CrWMoVB17-11-16 steels were remelted in vacuum in an electric-arc furnace at the pressure of approx. 1 Pa, and X40CrMoV5-1(es) steel was subject to electroslag remelting. Vacuum remelting and electroslag remelting were applied to produce steel with a higher structural homogeneity and a limited fraction of non-metallic inclusions as compared to the ones remelted conventionally.

Table 2. *Chemical composition of the tested steels*

Steel type	Concentration of elements, %										
	C	Mn	Si	P	S	Cr	Mo	V	W	Ni	Others
18CrMnTi4-4	0.17	1.10	0.23	0.030	0.040	0.93					Ti 0.09
38CrAlMo6-10	0.40	0.43	0.36	0.015	0.007	1.48	0.26			0.12	Al 0.94
37CrMoB10-4	0.38	1.39	0.23	0.014	0.014	2.43	0.42	0.10		0.17	B 0.003
X37CrMoV5-1	0.44	0.49	0.97	0.016	0.012	5.06	1.28	0.50		0.09	
X37CrMoV5-1(vac)	0.38	0.43	0.81	0.015	0.014	5.41	1.35	0.44		0.23	
X40CrMoV5-1(vac)	0.45	0.42	1.04	0.018	0.020	5.10	1.38	1.02		0.12	
X40CrMoV5-1(es)	0.41	0.34	0.78	0.024	0.005	5.56	1.08	1.27	0.10	0.11	
40CrWMoVB17-11-16	0.40	0.40	0.29	0.017	0.020	4.30	1.67	1.48	2.80	0.28	Co 1.68 B 0.05
HS12-0-2+C	1.06	0.31	0.28	0.030	0.023	4.41	0.64	2.56	11.20	0.22	
HS6-5-2	0.88	0.26	0.37	0.030	0.021	3.90	4.90	1.88	6.20		

The tested steels underwent heat treatment in the conditions given in Table 3 and thermochemical treatment. High-temperature thermochemical treatment operations, i.e. carburising and boriding are conducted prior to heat treatment, and low-temperature operations, i.e. nitriding and its variants are carried out after heat treatment. The gas nitriding of the specimens treated thermally was carried out in a retort in an atmosphere of partially dissociated ammonia, at a temperature of 540°C and 570°C for 0.5 to 8 hrs. Some of the specimens were nitrided in an atmosphere containing 50% NH_3 + 50% N_2, and 25% NH_3 + 75% N_2. Plasma nitriding was performed in VHT equipment in the atmosphere of a gas mixture composed in 90% N_2 + 10% H_2. The specimens were nitrided for 3 h at a temperature of 550°C, pressure of 300 Pa and voltage of 1250 V.

Twist drills with the diameter of 5 mm were made of the same HS6-5-2 steel cast with the hot-rolling method. The drills were heat treated, ground and subjected to selected thermochemical treatment methods, in particular to:

- passivation at a temperature of 540°C for 2 hours in a retort into which distilled water was added drop by drop,
- selective nitriding, i.e. first oxidising in water vapour at a temperature of 540°C for 30 min., and then nitriding in an atmosphere of partially dissociated ammonia at a temperature of 520°C for 30 min.,

- gaseous sulphonitriding in an atmosphere of ammonia with the addition of sulphur vapour, and next vacuum nitriding (with decreased pressure in a retort),
- oxynitriding in a fluidised bed at a temperature of 550°C for 25 min. in an atmosphere of water vapour and partially dissociated ammonia,
- plasma nitriding at a temperature of 505°C for 15 min. in an atmosphere of N_2+H_2 at a pressure of approx. 270 Pa.

Table 3. *Heat treatment conditions for the tested steels*

Steel type	Temperature, °C	
	Austenitisation	Tempering
18CrMnTi4-4	840	160-300
38CrAlMo6-10	920	500-600
37CrMoB10-4	890	500-600
X37CrMoV5-1	970-1030	500-600
X37CrMoV5-1(vac)	970-1030	500-600
X40CrMoV5-1(vac)	1000-1060	500-600
X40CrMoV5-1(es)	1000-1060	500-600
40CrWMoVB17-11-16	1090-1150	500-650
HS12-0-2+C	1160-1220	510-630
HS6-5-2	1190-1250	510-630

The specimens and the toothed gears made of 18CrMnTi4-4 steel were carburised at a temperature of 880°C in an endothermic atmosphere with the addition of 4% of methane. Carbonitriding was carried out in the conditions given, by adding 3 and 6% of ammonia to the carburising atmosphere. After annealing for 2.5 to 17 hrs, as a result of which a 0.2 to 1.65 mm thick layer is produced, the specimens were cooled to the temperature of 840°C and quenched directly in oil, and then tempered for 2 h between 160 to 300°C.

The diffusion boriding of the specimens was performed in powder containing 15% B_4C, 83.6% Al_2O_3, 0.7% NH_4Cl and 0.7% NaF, in heat-resisting steel containers, at a temperature of 950, 1000 and 1030°C for 2 to 12 hours. The containers were cooled in air after boriding at 950°C and 1000°C, and next the X40CrMoV5-1 steel specimens were removed and quenched from 1030°C and tempered at 600°C. After boriding at a temperature of 1030°C, the containers were cooled with a stream of a water suspension in air which enabled to quench the specimens immediately from the boriding temperature, and then the specimens were tempered twice at 600°C.

2.3. Materials science methodology

Structural tests using the methods of light metallography, transmission and scanning electron microscopy and an X-ray structure analysis were carried out to determine the impact of thermochemical treatment conditions and of some functional properties tests on the structure of the tested steels surface layers and core.

Metallographic tests were undertaken with MEF4A light microscopes by Leica with a Leica-Qwin, MeF image analysis system by Reichert and Neophot 2 by Carl Zeiss Jena with the magnification range of 10 to 1000. Some of the structure tests with the magnification of up to 3000 times were made with JXA-50A scanning electron microscopes by JEOL, DSM-940 scanning electron microscopes by Opton and SUPRA 35 by Zeiss, with the accelerating voltage of 20 kV, using back scattered electrons (BSE) and secondary electrons (SE) detection.

The phase composition of the specimens diffusion layers and core was examined with an X-ray qualitative and quantitative phase analysis method using DRON 2,0 and X'Pert diffractometers by Philips. Textures were also examined with a reflection technique with a Siemens-Halske Kristalloflex-4 diffractometer. The penetration depth of X-rays in the conditions applied was estimated at approx. 0.03 to 0.04 mm. For this reason, to determine the phase composition of the thick surface (borided and carburised) layers, diffraction patterns were made after grinding off the subsequent 0.03 mm thick layers from the specimens until the diffusion layer has been removed completely.

The structure of the steel diffusion layers and core was tested by observing thin foils in Tesla BS 540 and JEOL 200CX transmission electron microscopes with the accelerating voltage of 100 to 200 kV. The thin foils made of approx. 0.3 mm thick layers were prepared by cutting off plates from the specimens surface. Next, discs were cut out from such plates with the diameter of approx. 3 mm which were then thinned out mechanically to approx. 0.1 mm. The final electrolytic polishing was performed with a jet method in an electrolyte composed of 20 cm^3 H_2SO_4 and 80 cm^3 CH_3OH. The thin foils made with the heat treated steels and with the carburised layer were polished electrolytically in a reagent containing 50 g CrO_3 and 490 cm^3 H_3PO_4. Some of the thin foils were subjected to ion thinning in a Gatan device. The phase composition and the relative orientation of phases with the matrix was determined with the selected area electron diffraction method.

An X-ray microanalysis with the energy dispersive spectroscopy (EDS) method and wavelength dispersive spectroscopy (WDS) method by means of JXA-50A apparatuses by JEOL, SEMQ by ARL and SUPRA 35 by Zeiss at the accelerating voltage of 10 to 20 kV was undertaken to identify the distribution of elements in the surface layer of the thermochemical treated specimens. The qualitative analyses of surface and linear elements distribution were made and quantitative analyses in the selected points on the tested sections of surface layers were made.

Variations in the concentration of elements in the nitrided layer and in the substrate were determined also based on tests in the glow discharge optical emission spectroscopy (GDOES) GDS-750 QDP by Leco Instruments. Variations in the concentration of carbon in the carbonised and carbonitrided layers were investigated with LECO equipment, by analysing the chips taken every 0.1 mm from the surface layer to the core.

Dilatometric tests with a differential Adamel dilatometer and with DI-4 and Linceis absolute dilatometers were made to calculate the linear expansion factor for borides and to identify the phase transition temperature in the carbonised layer.

Some mechanical properties of the heat treated steels were determined during the tests. The Rockwell method at the C scale was used to measure the hardness of specimens for the tested tool and high-speed steels after heat treatment and machine steels for carburising and the total load applied was 1471 N. At least 30 measurements for each condition were taken. Impact strength at room temperature and at higher temperature was investigated with a Charpy pendulum machine using 10 specimens for each test variant. Tensile strength and bending strength tests were made with an Instron 1195 tensile testing machine fitted with a high-temperature attachment using 6 specimens for each test variant. The fatigue strength tests of steel with carburised layers were carried out with a PWY tensile testing machine by Schenck according to a neutral and ripple cycle, at a frequency of 16.66 Hz and with the agreed number of cycles $N_G = 10^7$. The results of the strength properties tests were developed statistically by calculating the average value and the average value confidence interval for the confidence level of $\alpha = 0.05$.

Measurements were made with an attachment fitted to an MeF microscope and with a DUH 202 ultra-microhardness tester by Schimadzu on lateral fractures with the Vickers method by applying the load of 0.49 and 0.98 N in order to identify microhardness distribution for the section of diffusion layers. The average hardness value was calculated each time with 5 to 10 measurements carried out within the same distance from the specimen surface.

Thermal fatigue resistance tests were performed with devices enabling direct and indirect specimens heating. A device was used to test heat treated steels enabling the induction heating of the surface layer of the rotating disc being cooled in water. This method ensures a short thermal cycle. The maximum temperature of the specimens surface was approx. 600°C. The number of thermal cycles during the tests was 3000 or 5000. Indirect heating was used for testing resistance to cyclic temperature steel variations with surface layers, as the induction heating method cannot be used in this case due to the different physiochemical properties of the surface layer and the core. Indirect heating was accomplished by contacting cyclically the specimen with a cooper insert heated to approx. 900°C and cooling was accomplished with a water jet. One cycle within 600-20°C lasted 12 s. As the specimens tested in the device with direct-contact heating were subjected to the corrosive effect of air and water during thermal cycles, therefore, some of the thermal fatigue resistance tests were made with a device where the specimens were heated by means of radiation and convection in an electric furnace and were cooled in water. The specimens, lifted and dropped cyclically, can be placed in air-tight containers securing them against corrosion during thermal cycles. A thermal cycle within the range of 600-80°C set within the distance of approx. 0.1 mm from the solid specimen's heated surface lasts approx. 140 s with the furnace temperature of approx. 860°C. Considering a long thermal cycle of the solid specimens placed in the containers, cylindrical specimens with a smaller thermal capacity were also used. The solid specimens tested without the safety containers were subjected to 100 to 2500 thermal cycles, and the cylindrical specimens placed in the containers underwent between 2500 to 25000 thermal cycles. The thermal fatigue resistance analyses for the specimens were undertaken based on the measurements of depth and density of cracks (the average number of surface cracks formed at the distance of 1 mm). The cracks were measured on the fractures made in the plane perpendicular to the specimens surface. Approx. 50 measurements were taken in 3 specimens for each test variant. The measurements results were established on a statistical basis.

The specimens after the thermal fatigue test, without using the safety containers, were etched in a 40% aqueous water solution of hydrochloric acid in order to remove mineral salts depositing on their surface. The specimens, after etching and drying, underwent gravimetric tests on an analytical WA-31 scale with the measuring accuracy of 0.1 mg to determine mass variations during cyclical temperature variations.

Abrasive wear resistance tests with the pin-on-disc method were carried out with a CSEM THT – High Temperature Tribometer – at a room temperature and in 500°C. A 6 mm Al_2O_3 corundum ball was used as a counter-specimen. The fixed ball was pressed with the force of 7 N against a disc rotating in the horizontal plane at a speed of 50 cm/s during a pin-on-disc test carried out with 1000 and 7500 revolutions with the specimens having their layer nitrided and after being heat treated. The width of the formed wear tracks was measured with a light microscope after the test and the average volume of the material removed during tribological wear was calculated.

Fatigue strength tests for Z_{gj} tooth root were made at a test stand comprised of two transmission gears with the first gear being a test object and the other being a closing gear. The load applied onto the gears was changed by twisting alternately the clutch discs seated on the independent parts of a torsion shaft.

The cutting properties of the heat treated and thermochemically treated high-speed HS6-5-2 steel drills were tested with constant cutting parameters. 15 mm deep blind openings were bored for this purpose in flat bars made of normalised C55 steel with the hardness of 200-220 HBW, with the cutting speed of 28.7 m/min and the shift of 0.18 mm per revolution. Cooling with the capacity of approx. 5 l/min using an emulsion made of 10% emulsifiable E oil and water was applied for boring. The wear equal to VB_{max} = 0.5 mm at the flank surface was adopted as a blade blunting criterion. 15 cutting ability tests were carried out for drills from each batch, and the results were elaborated in a statistical manner.

2.4. Technology roadmaps and technology information sheets

The results of the experimental and comparative research made provide source data for creating **technology roadmaps** [51-53]. The set-up of the custom technology roadmap corresponds to the first quarter of the Cartesian system of coordinates. Three time intervals for the years: 2010-11, 2020 and 2030 are provided on the axis of abscissa, and the time horizon for all the results of the research applied onto the map is 20 years. Seven main layers were applied onto the axis of coordinates of the technology roadmap answering subsequently to more and more detailed questions: When? Why? What? How? Where? Who? How much? The main technology roadmap layers hierarchised starting with the top, most general layers determining all-social and economic reasons and causes of the actions taken, through the middle layers characterising products and their manufacturing technology, to the bottom layers detailing organisational and technical matters concerning the place, contractor and costs. The middle layers of the technology roadmap are subject to two types of influence – pull from the top layers and push from the bottom layers. The relationships between the individual layers and sub-layers of the technology roadmap are presented with the different types of arrows representing, respectively, cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow.

The technology roadmaps prepared with a custom concept are a very convenient tool for a comparative analysis enabling to select the best technologies according to the criterion chosen. Besides, their undisputed advantage is flexibility and, if needed, additional sub-layers can be added or expanded for the maps according to the circumstances of the industry, size of enterprise, scale of the company's business or an entrepreneur's individual expectations.

Technology information sheets, containing technical information very helpful in implementing a specific technology in the industrial practice, especially in SMEs not having the capital allowing to conduct own research, are detailing and supplementing the technology roadmaps. The technology information sheets provide, in particular, a description of the technological process progress and a characteristic of a physiochemical phenomenon accompanying the technological processes, the advantages and disadvantages of the relevant technology, the most prospective detailed technologies and substitute / alternative technologies. A technology information sheet also contains the types of a coating / surface layer that may be deposited or the processes occurring at the substrate surface, as well as the specific properties of coatings / surface layers / substrate surfaces as a result of technological processes. A special heed was paid also to the general physiochemical conditions of technological process implementation, substrate material preparation methods, research instrument type / kind and possible specific accessories. Besides, the research results acquired with an expert research method have allowed to provide the following details in the developed sheets determined with a universal scale of relative states: the impact of technology application on the predicted and expected material properties, the efficiency of preventing the consequences of wear, industry section acc. to the PKD classification having the highest technology applicability, the applicability of computer modelling and steering methods and the development prospects of the individual analysed technologies. In addition, each technology information sheet provides a general or example diagram of the considered production process and a three-part list of the recommended references.

3. Technologies value and their strategic development directions

The results of the foresight research described in this chapter includes the assessment of potential and the attractiveness of the analysed technologies against the micro- and macro-environment performed based on the key experts' opinions expressed in a ten-degree universal scale of relative states and a recommended strategy of managing a relevant technology resulting from the assessment together with the predicted strategic development tracks. The three homogenous groups have been separated from the analysed technologies in order to carry out experimental and comparative works including, respectively:

(A) nitriding and its variants,

(B) carburising and carbonitriding,

(C) diffusion boriding.

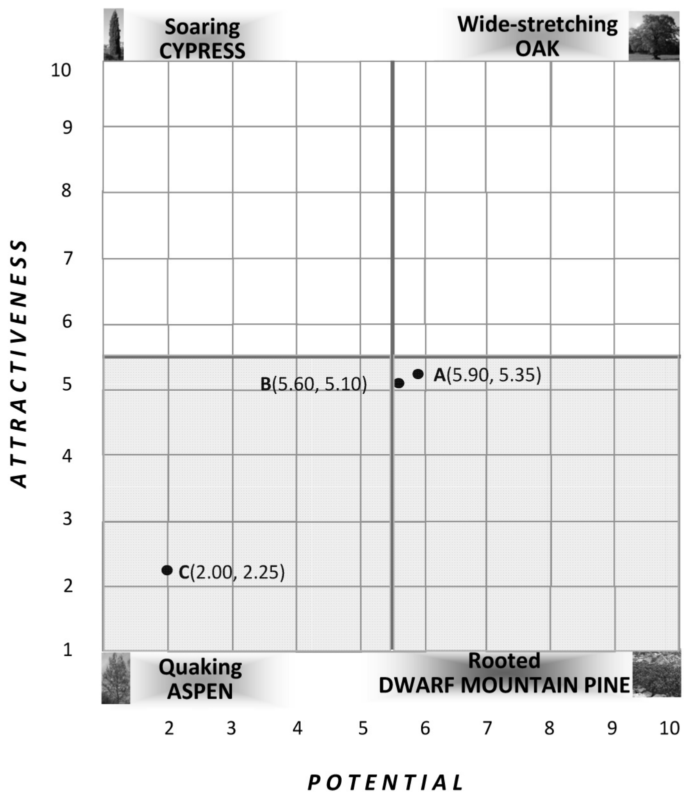

Figure 3. *The dendrological matrix of technology value for the following thermochemical technologies: (A) nitriding and its variants, (B) carburising and carbonitriding, (C) diffusion boriding*

The individual technology groups have been evaluated by experts using a ten-degree universal scale of relative states for their: business, economic, humane, natural and system attractiveness as well as for their: creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and the result received for the individual groups of technologies was entered into the dendrological matrix of technologies value (Fig. 3). The analysis showed that a group of (A) technologies including nitriding and its variants and (B), including carburising and carbonitriding in high temperature, were classified to the quarter called Rooted Dwarf Mountain Pine representing solid, proven technologies with high potential, characterised by limited attractiveness. Diffusion boriding (C) was classified to the least promising matrix quarter referred to as Quaking Aspen representing technologies with limited potential and small attractiveness.

The positive and negative environment influence on the relevant groups of technologies was evaluated with the meteorological matrix of environment influence. The results of the multi-criteria analysis were entered into the matrix evaluated in the experts process, as shown in Fig. 4. The results of the studies made show that in the case of all the tested group of technologies, the environment is predictable and stable with a neutral character. Therefore, no related spectacular opportunities should be expected from it, nor unpredictable difficulties that are definitely not supportive to the development of the technology groups in question. Very similar results (3.26, 5.25) were obtained for the technology group (A) and the technology group (B), receiving the value of (3.11, 4.91), and the technology group (C) with (4.70, 3.33) ranked lower, meaning fewer opportunities and more difficulties in the future.

At the next stage of research works, the research results presented graphically with the dendrological matrix of technology value and the meteorological matrix of environment influence were entered into the technologies strategy matrix (Fig. 5). The matrix is presenting, graphically, the place of the individual technology groups of steel thermo-chemical processing with regard to their value and the environment influence degree, indicating the relevant managing strategies. Using the pre-defined mathematical relationships, the specific numerical values provided in the dendrological and meteorological matrix dimensioned [2x2] were moved to the strategy matrix for technologies dimensioned [4x4]. For the group of technologies (A) and (B), it is recommended to use a strategy of a dwarf mountain pine in autumn that recommends deriving profits from production implementation in a stable, predictable environment using a solid technology that should be modernised and promoted intensively to strengthen its attractiveness. As regards the technology group (C), the strategy of an aspen in autumn should be applied that recommends the withdrawal of a technology from a market not providing new opportunities.

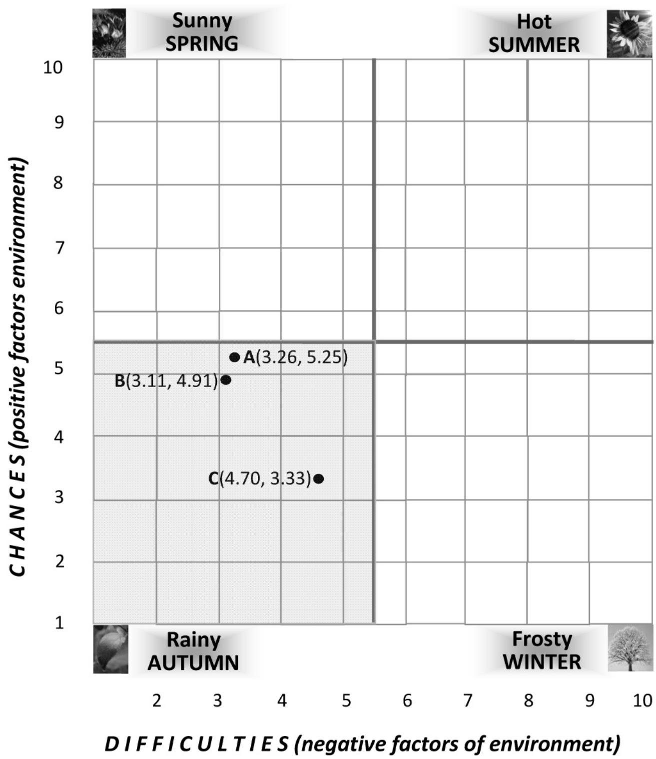

Figure 4. *The meteorological matrix of environment influence for the following thermochemical technologies: (A) nitriding and its variants, (B) carburising and carbonitriding, (C) diffusion boriding*

Strategic development tracks for the individual technology groups were established based on the acquired expert opinions. The tracks represent an optimistic, most probable and pessimistic forecast of their development for the relevant time intervals: 2015, 2020, 2025 and 2030. A graphical example of the strategy matrix for the technologies with strategic development tracks provided in three variants created for nitriding and its variants is shown in Fig. 6. The most probable strategic development track for this technology group assumes that neutral environment conditions will be maintained to be slowly, insignificantly improving in the next years. The technology value should also increase slightly as forecast, which is connected with the strengthening potential of technology with attractiveness maintaining at the existing level. The optimistic strategic development track assumes more dynamic, positive changes taking place in the environment related to the decreasing number of external difficulties accompanied by the strengthening technology potential. This, on the other hand,

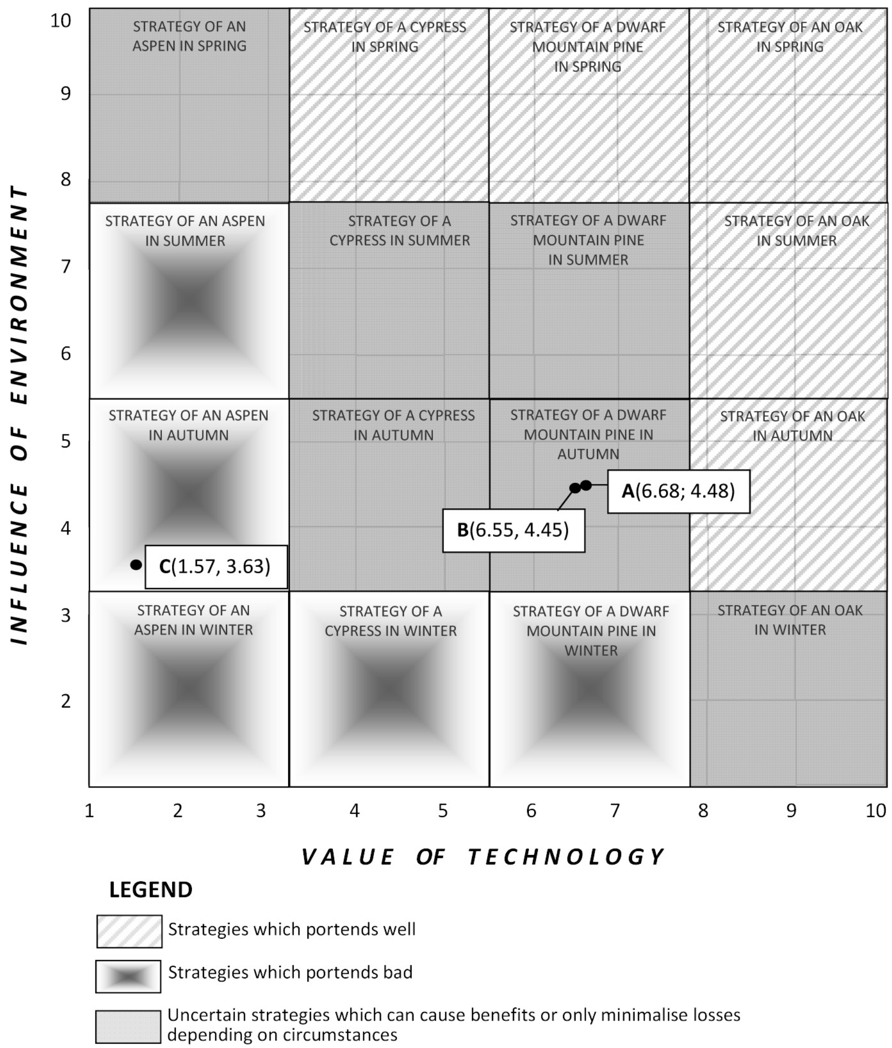

***Figure 5.** The matrix of strategies for technologies prepared for selected thermochemical technologies, as follows: (A) nitriding and its variants, (B) carburising and carbonitriding, (C) diffusion boriding*

allows to move the technology group (A) from the strategy field of dwarf mountain pine in autumn to the strategy field of dwarf mountain pine in spring. This means that the key objective should be strengthening, modernising, automating, computerising and promoting technologies with high potential based on good economic conditions at the market. The optimistic variant of events is mainly related to the vision of fast development of the most prospective (ion, glowing) plasma nitriding and hybrid technologies with nitriding (e.g. in connection with the technology of physical deposition from gaseous phase – PVD) with the development of nitriding at the existing level with decreased pressure and the smaller importance of conventional gas nitriding. The development of nitriding and its variants must be also accompanied by an improved ecological aspect of the discussed technologies to minimise the harmful substances emitted to the environment.

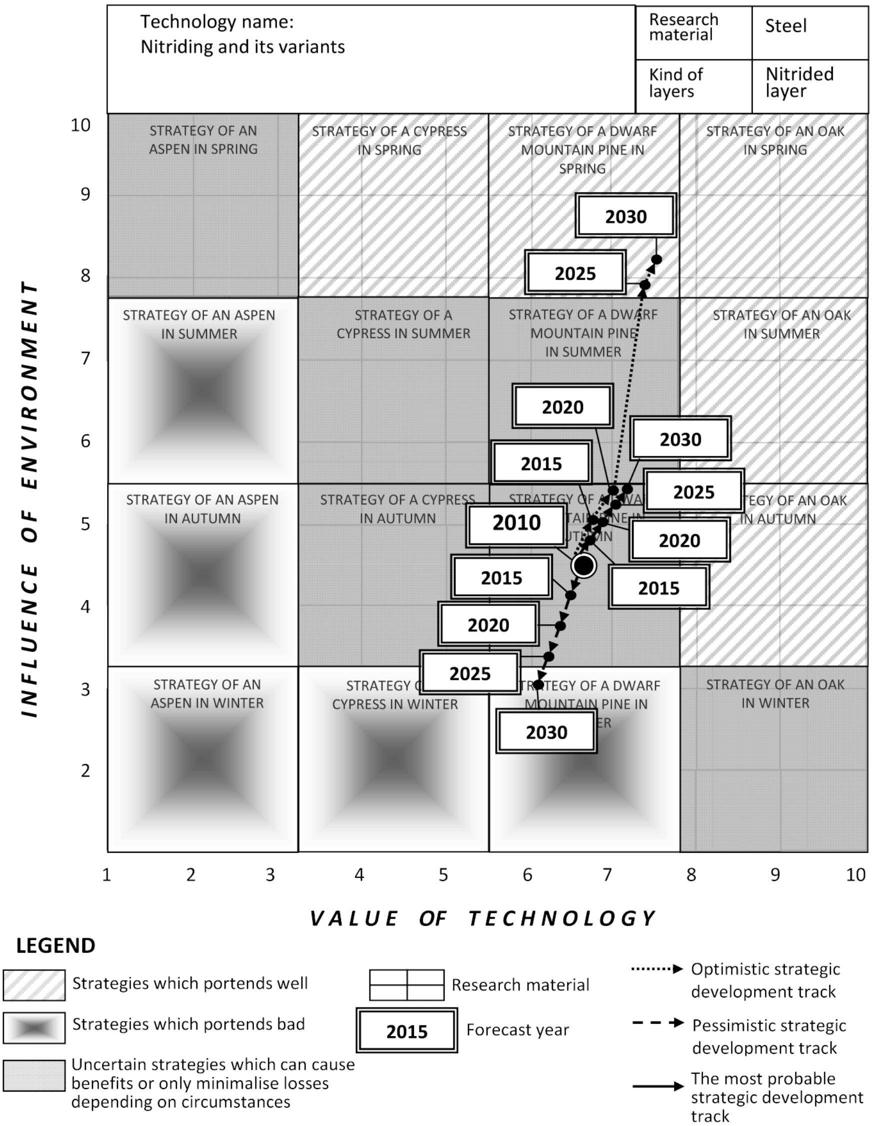

Figure 6. *The strategic development tracks created for the (A) demonstration technology group: nitriding and its variants*

The analysis made has shown that that the development forecast of the technology group (B) including carburising and carbonitriding is very similar to the development forecast of the technology (A), reaching slightly smaller, very similar values. An optimistic development variant of this technology group is conditioned by the strengthening and growing importance of the most promising technologies such as: glowing carbonitriding, carburising in a controlled atmosphere of natural gas and the carbonitriding variants enabling direct quenching and low-temperature tempering of the treated parts.

Table 4. *The strategic development tracks of selected thermochemical treatment. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic, (MP) – the most probable*

No	Technology name	Steady state 2010	Type of strategic development tracks	Years			
				2015	2020	2025	2030
1.	Nitriding and its variants	Strategy of a dwarf mountain pine in autumn A (6.7, 4.5)	(O)	(6.8, 4.9)	(7.0, 5.4)	(7.5, 7.9)	(7.7, 8.3)
			(P)	(6.5, 4.1)	(6.4, 3.8)	(6.2, 3.4)	(6.1, 3.1)
			(MP)	(6.8, 4.7)	(6.9, 4.9)	(7.1, 5.2)	(7.3, 5.4)
2.	Carburising and carbonitriding	Strategy of a dwarf mountain pine in autumn B (6.6, 4.5)	(O)	(6.7, 4.7)	(6.8, 5.1)	(7.2, 7.8)	(7.4, 8.1)
			(P)	(6.4, 3.9)	(6.2, 3.6)	(6.0, 3.2)	(5.8, 2.8)
			(MP)	(6.7, 4.5)	(6.8, 4.6)	(6.9, 4.8)	(7.1, 5.1)
3.	Diffusion boriding	Strategy of an aspen in autumn C (1.6, 3.6)	(O)	(1.8, 4.0)	(2.0, 4.3)	(2.3, 5.6)	(2.6, 5.9)
			(P)	(1.5, 3.1)	(1.4, 2.5)	(1.3, 2.1)	(1.2, 1.7)
			(MP)	(1.5, 3.5)	(1.4, 3.4)	(1.3, 3.4)	(1.2, 3.3)

The weakest development prospects are exhibited by the technology group (C) including diffusion boriding with its significance most likely declining within the nearest 20 years. This stems from limited effectiveness, high costs and the unfavourable environmental impact of the existing boriding technologies. The pessimistic variant of events assumes that already in 2015 the technology group (C) moves from the field of matrix corresponding to the strategy of aspen in winter, where it is recommended to withdraw a weak technology from the market with difficulties predominant. An optimistic variant provides gradual, moderate improvement in the value of the technology group (C) with more favourable conditions of the environment over the next 20 years, which would allow to move it in 2025 to the matrix field corresponding to the strategy of aspen in summer. The strategy allows to run a risk and to make an attempt to exploit the new emerging external circumstances. This, however, is possible only if there is a major breakthrough by finding a new, wide range of industrial applications and a spectacular improvement in the currently used solutions, especially for environmental protection.

The numerical values resulting from all the research performed for the three analysed group of technologies are presented in Table 4.

4. Research results concerning the structure and properties of thermochemically treated steels

4.1. Structure and properties of surface layers of steel after nitriding

Nitriding and its variants are very popular methods of thermochemical treatment for machine and tool steels. They are performed as the final operation after prior quenching and high tempering at a temperature slightly higher than the assumed nitriding temperature. A layer of nitrides is forming at the surface of the tested steels as a result of gas nitriding carried out in an atmosphere of partially dissociated ammonia at a temperature of 540°C. The hardness of the layer is up to approx. 1500 HV 0.05 and the layer is transiting into the diffusion zone to the core. The hardness of the zone depends on the chemical composition and on the steel heat treatment conditions (Fig. 7). The largest thickness for the continuous zone of nitrides and for the entire diffusion zone was obtained on 38CrAlMo6-10 machine steel for nitriding (Fig. 8) whereas high-speed HS6-5-2 and HS12-0-2+C (Fig. 7) steels exhibit the highest surface hardness. If the nitriding temperature of high-speed steels is raised to 570°C, the thickness of the hardened layer is higher by approx. 20-25%. The continuous zone of nitrides by the steel surface contains most of all a phase with the $Fe_{2-3}N$ lattice structure and also phases with CrN and Mo_2N lattices in individual steels. Such nitrides also occur as precipitates distributed at the boundaries of grains in the diffusion zone of a nitrided layer (Fig. 9). The dispersive precipitates of $Fe_{16}N_2$ nitride are also produced in martensite in this layer and the precipitates maintain their privileged crystallographic orientation with the matrix (Fig. 10).

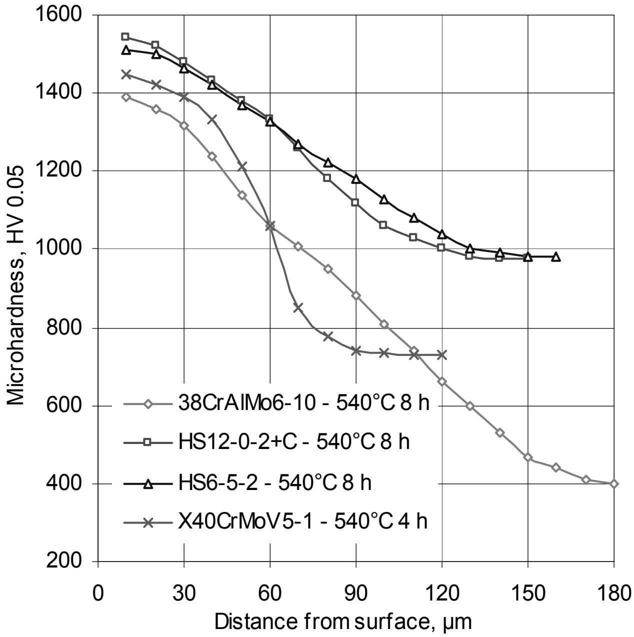

Figure 7. *Microhardness distribution in the surface layer of the selected steels subject to quenching and tempering gas nitriding at 540°C*

Figure 8. *Surface layer microstructure of 38CrAlMo6-10 steel quenched from 920°C, tempered at 575°C and gas-nitrided at 570°C for 8 hrs in an ammonia atmosphere*

The continuous layers of hard brittle nitrides produced at the steel surface reduce the strength and ductility of steel. Nitriding in the atmosphere of an ammonia and technical nitrogen mixture has a favourable effect on maintaining ductility at high strength and slightly limited surface hardness. This is because the continuous zone of nitrides is produced on the surface to a limited extent only. If 50% of inert N_2 molecular nitrogen is added to the nitriding atmosphere, the hardness and thickness of the hardened layer is lowered, and this becomes clear after nitriding for a short time. Hardness variations in the surface layer are similar to those achieved after nitriding in an atmosphere of 100% NH_3 for a twice shorter time (Fig. 11). After nitriding in an atmosphere containing 25% NH_3 and 75% N_2, a diffusion zone is created only at the steel surface (Fig. 12), even after long-term nitriding. The thickness of surface layers and their hardness and abrasion resistance is lower, however.

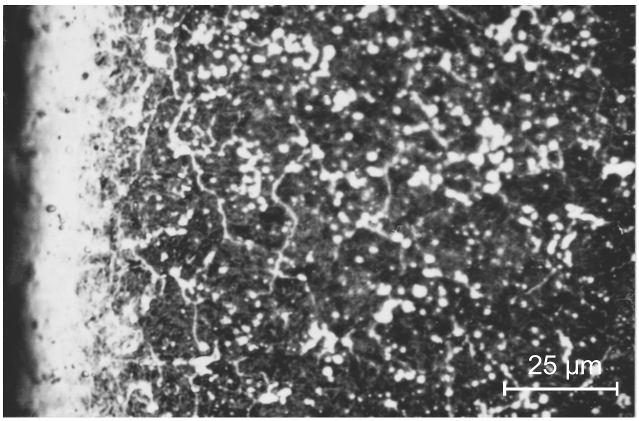

Figure 9. *Surface layer microstructure of HS6-5-2 steel quenched, tempered and gas-nitrided at 570°C for 4 hrs in an ammonia atmosphere*

If a surface layer with high hardness is formed, this causes the bending strength of the tested steels to change with impact load and static load. Impact strength is usually significantly reduced, especially the impact strength of the steel featuring small crack resistance, and static bending strength increases if layer thickness is low as compared to part dimensions. The tested 4 mm thick specimens made of high-speed HS6-5-2 steel quenched from 1230°C and tempered once at 550°C have their bending strength R_g of approx. 2460 MPa. The strength increases by approx. 20% after tempering again at the same temperature. This is certainly a result of dispersion alloy carbides being released in the martensite formed as a result of retained austenite transformation during steel cooling after the first tempering. Steel nitriding at 540°C in an atmosphere of 100% NH_3 is reducing R_g resistance to approx. 1640 MPa (Fig. 13). R_g rises slightly under the tested conditions only after nitriding the high-speed HS6-5-2 steel for 0.5 h in an atmosphere containing 25% NH_3 and 75% N_2. The plastic properties of steel can

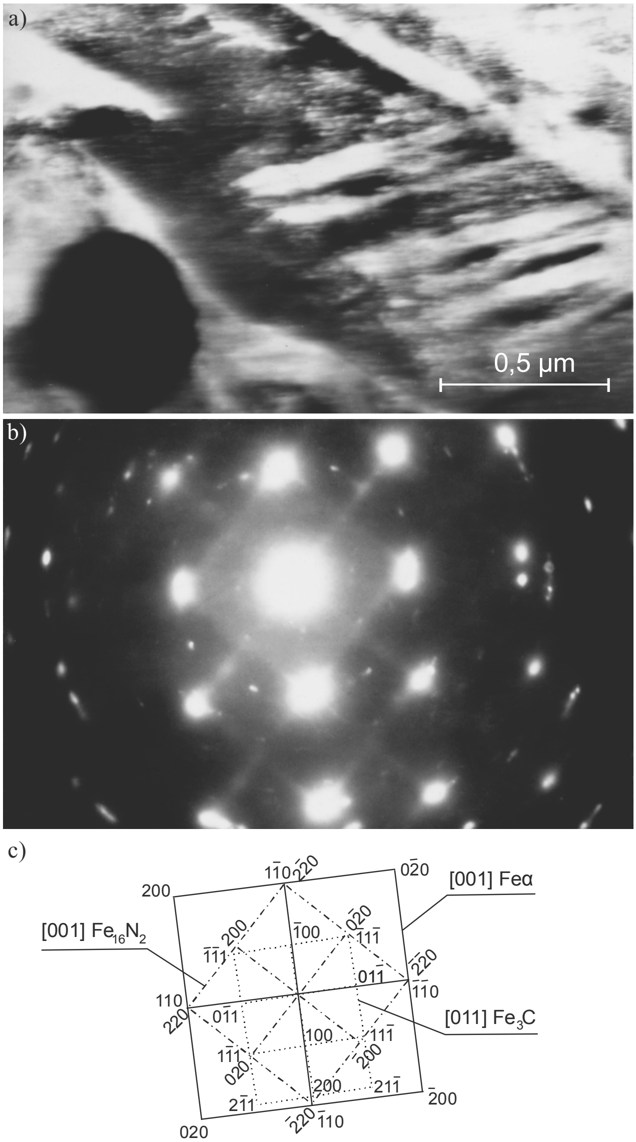

Figure 10. *Surface layer microstructure of HS6-5-2 steel quenched, tempered and gas-nitrided at 540°C for 3 hrs; thin foil, a – bright field image, b – diffraction pattern from the area as in Fig. a, c – diffraction pattern solution from Fig. b*

be characterised to some extent by the total deflection value f_g that decreases from 1.95 mm for the heat treated steels to 0.84 mm after nitriding the specimens at 540°C for 4 hrs (Fig. 13). The plastic deflection value f_{pl} for the heat treated high-speed steel specimens is very low – 0.05 mm, and becomes practically immeasurable after nitriding.

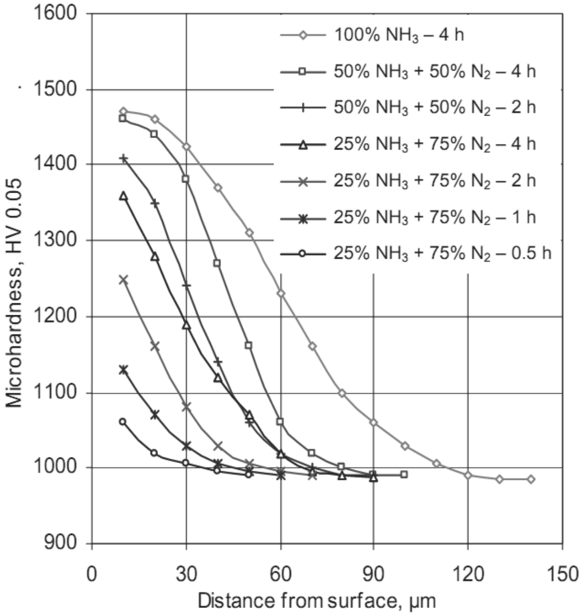

Figure 11. *Microhardness distribution in the surface layer of HS6-5-2 steel quenched from 1230°C, tempered at 550°C and gas-nitrided at 540°C in an atmosphere of ammonia with addition of nitrogen*

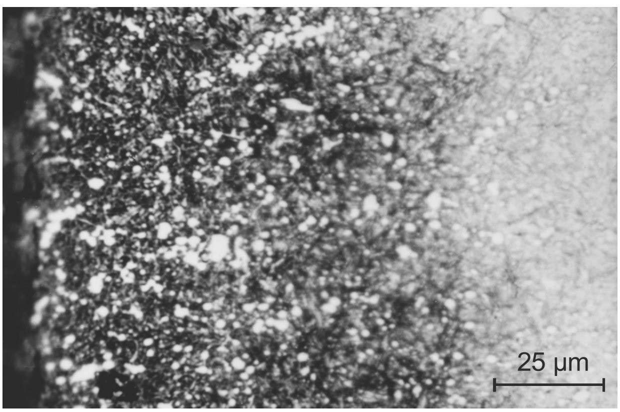

Figure 12. *Surface layer microstructure of HS6-5-2 steel gas-nitrided at 540°C for 4 hrs in an atmosphere containing 25% NH_3 and 75% N_2*

The structure of the hot-work X40CrMoV5-1 tool steel surface layer after gas nitriding in an atmosphere of dissociated ammonia at a temperature of 570°C consists of a continuous zone of alloy nitrides and of a diffusion zone lying underneath (Fig. 14). The zone of continuous nitrides with the hardness of approx. 1340 HV 0.05 consists of ε-$Fe_{2\text{-}3}N$ and CrN phase.

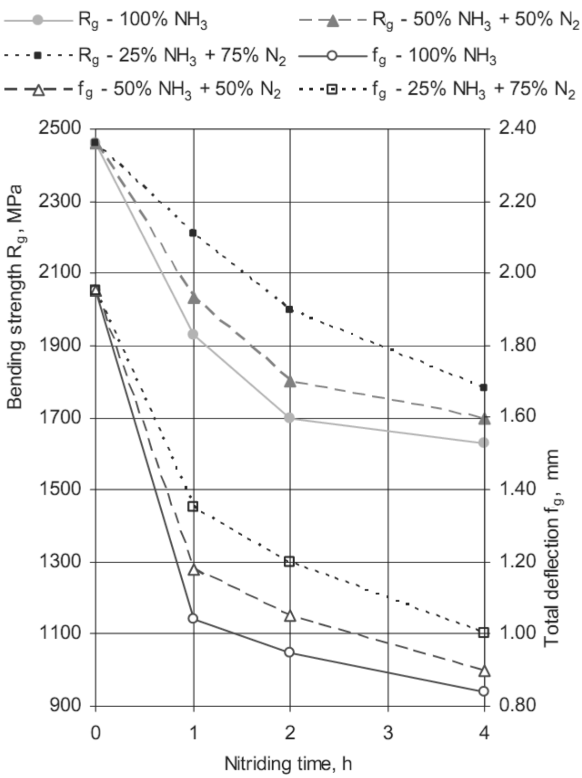

Figure 13. *Influence of nitriding conditions on the properties identified in the static bending test of the specimens made of HS6-5-2 steel quenched, tempered and gas-nitrided at 540°C*

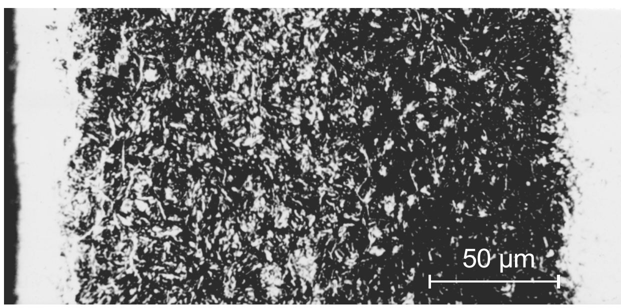

Figure 14. *Microstructure of the X40CrMoV5-1 steel surface layer gas-nitrided at 570°C for 6 hrs*

The nitrogen concentration and the layer hardness are declining constantly in the diffusion zone and reach the value of approx. 550 HV 0.05 in the core (Figs. 14, 15). The diffusion zone has a tempered martensite structure with the dispersion precipitates of carbides and nitrides of $Fe_{16}N_2$ type and the grainy nitrides of CrN and $Fe_{2-3}N$ type. In the core of the nitrided specimens, X40CrMoV5-1 steel has a tempered martensite structure with the M_7C_3, M_4C_3 and M_3C dispersion alloy carbides. The structure is formed during heat treatment including quenching from 1030°C and tempering at 600°C.

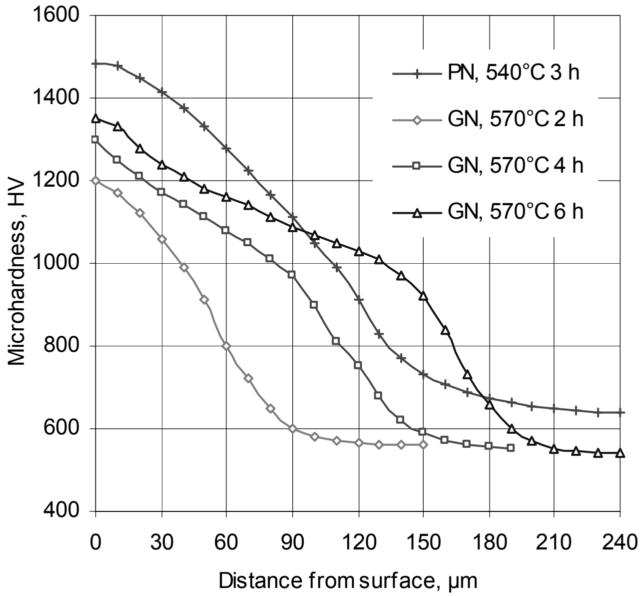

Figure 15. *Microhardness distribution in the surface layer of gas-nitrided X40CrMoV5-1 steel (GN) and plasma-nitrided X37CrMoV5-1 steel (PN)*

Some functional properties of the nitrided layer strongly depend upon its thickness and the fraction of structure components. Hence, it may be more advantageous to perform nitriding with a method allowing greater control over the structure of the produced surface layer as compared to normal gas nitriding. The plasma nitriding technology (also known as ion nitriding, plasma-ion nitriding or glow-discharge nitriding) progressing using glow discharge, has been dynamically entering the global industry and is successfully replacing the traditional process. This technology is so successful because of the following advantages distinctive for the plasma nitriding process as compared to traditional technologies [4-7]:

- the four basic types of nitrided layers structures can be achieved in a controlled manner: a diffusion zone only, a diffusion and iron nitride zone γ'-Fe_4N, a diffusion and iron carbonitride zone ε-$Fe_{2\text{-}3}(C,N)_{1\text{-}x}$ and a diffusion zone with the surface layer of ε + γ' components; this allows to choose the type of the nitrided layer structure for the specific operating conditions of a given part,

- parts with complicated shapes can be treated,
- a shorter process duration as a charge is heated faster to the treatment temperature and the faster activation of the environment and the treated charge surface,
- controllable increase of dimensions for the parts subject to treatment,
- considerable electricity savings, a batch alone is heated only and no heat-resisting retorts are required, etc.; energy consumption with a specific charge represents 30-40% as compared to gas nitriding,
- the need of using ammonia as a reactive atmosphere is eliminated.

The plasma nitriding of X37CrMoV5-1 steel can be provided as an example. It is revealed with the metallographic observations of the microstructure of steel plasma-nitrided at a temperature of 550°C for 3 hrs that the nitrided layer is characterised by its homogenous, compact and zonal structure. The thickness of the layer that is plasma-nitrided in such conditions is 148 μm. Nitride precipitates are present at the boundaries of steel grains (Fig. 16) that were identified with an X-ray phase analysis method as ε-Fe_3N and γ'-Fe_4N phases. Feα reflections coming from martensite were also recorded (Fig. 17), with martensite being the matrix of the surface layer and the substrate.

A GDOES analysis performed for hot-work heat treated and plasma nitrided X37CrMoV5-1 tool steel (Fig. 18) shows, apart from the elements present in steel, i.e. Fe, Si, C, Mo, V, Mn, Mo, also the presence of nitrogen introduced into the surface layer in nitriding.

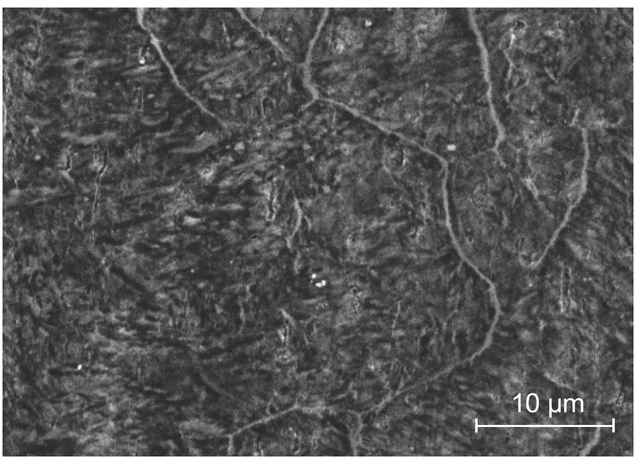

Figure 16. *Microstructure of the plasma-nitrided surface layer of X37CrMoV5-1 steel, SE image*

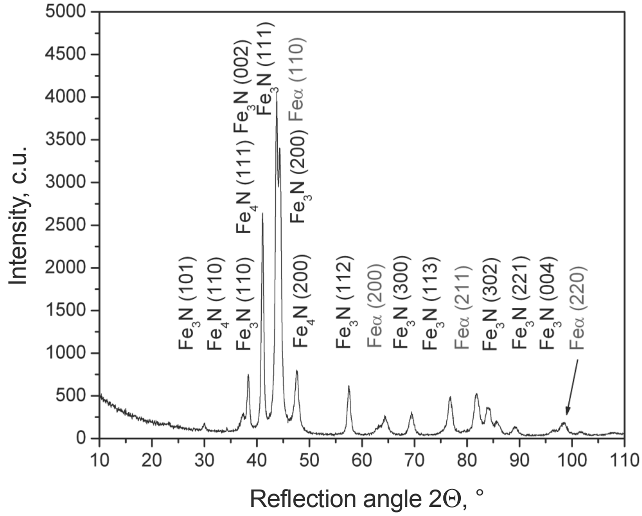

Figure 17. *X-ray diffraction pattern of the plasma-nitrided surface layer of X37CrMoV5-1 steel*

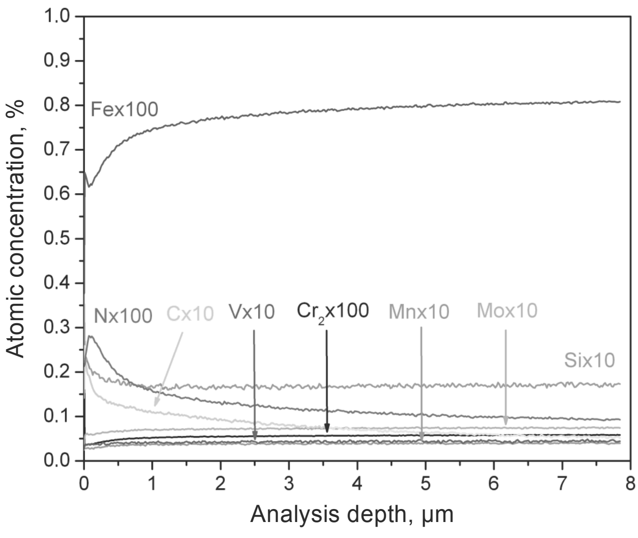

Figure 18. *Variations in the concentration of elements in plasma-nitrided X37CrMoV5-1 steel analysed with a GDOES spectrometer*

The maximum hardness of the plasma-nitrided layer is approx. 1480 HV 0.1. As distance from the surface increases, the micro-hardness of the tested nitrided layer is slowly decreasing to approx. 610 HV 0.1, which is adequate for the core (Fig. 15).

The tested hot-work heat treated X37CrMoV5-1 tool steel specimens prepared for depositing a plasma-nitrided layer exhibit the roughness of R_a = 0.008 μm. Roughness after plasma nitriding rises to 0.08 μm, i.e. typical for this process [54, 55]. An abrasive resistance test with the pin-on-disc was carried out to create a functional and operating characteristic of the tested plasma-nitrided layer. As the tested layers are intended for work at higher temperatures, the test was made at a room temperature and a temperature increased to 500°C. The friction coefficient variations tests during a test for heat treated steel and for a plasma-nitrided layer allow to conclude that the highest friction coefficient of approx. 0.5 to 0.7, respectively, for 20°C and 500°C, is exhibited in the tested conditions by the heat treated steel (Fig. 19a). If temperature is raised to 500°C, both for heat treated steel and for plasma-nitrided steel, the friction coefficient grows. This is a consequence of higher width and depth of the wear track at an increased temperature and an increased volume of the material worn. The high friction coefficient values for this steel may relate to its relatively low hardness. If a nitride layer is produced on this substrate, the friction coefficient falls to some 0.4 to 0.6 for 20°C and 500°C (Fig. 19b) along with smaller friction width (Fig. 20). The width of the wear tracks is correlated with their depth, thus the volumetric wear of the material removed during the abrasion resistance test was calculated on such basis. The largest material loss was identified for heat treated steel and, e.g. wear at 500°C after 7500 revolutions is 1.31 mm^3, whereas the volumetric wear of steel with a nitrided layer represents 1.03 mm^3 in such conditions (Fig. 21).

One may conclude based on the tests performed that the highest material wear in the conditions of a pin-on-disc test is seen at a temperature of 500°C. As a result of the plasma nitriding of X37CrMoV5-1 hot-work tool steel, the abrasion resistance of the steel improves substantially as compared to the heat treated steel. A nitrided layer improves the anti-wear properties mainly by reducing a friction coefficient. The layers nitrided in the conditions making it impossible to create a continuous zone of nitrides at the steel surface may also represent a substrate for multizonal hybrid layers [32, 33, 45, 56, 57] with the better functional properties than those achieved after thermochemical treatment with the methods used to date.

Nitriding is one of thermochemical treatment methods enhancing the wear-resistant of many tools, including those exposed to cyclical temperature variations. The substrate of the layers produced through thermochemical treatment should therefore be resistant to thermal fatigue. It was found as a result of the tests that the steels austenitised at a temperature ensuring the fine-grain structure of primary austenite and relatively high hardness after quenching and double

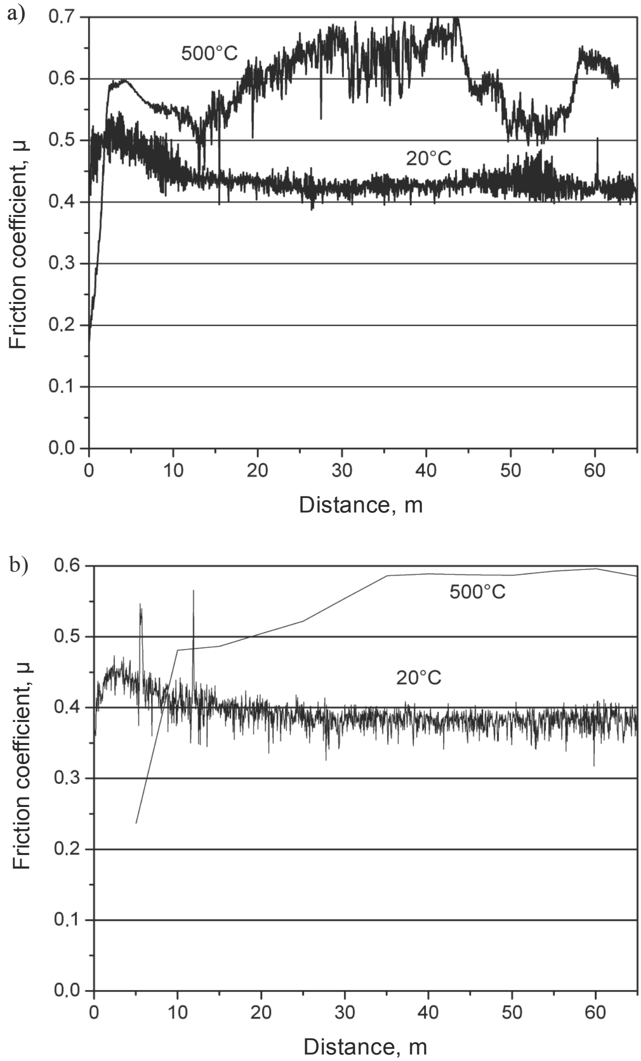

Figure 19. *Variation curve of the friction coefficient according to the friction path length for a) thermal treated and b) plasma-nitrided X37CrMoV5-1 steel tested at a temperature 20°C and 500°C for 1000 revolutions*

tempering at 600°C exhibit the highest resistance to thermal fatigue. The smallest depth of cracks with their density only slightly increased, are seen for X37CrMoV5-1, X40CrMoV5-1 and 40CrWMoVB17-11-16 steels tempered at 600°C (Fig. 22), i.e. at a temperature of approx. 50 to 100°C higher than this ensuring the secondary hardness effect. If tempering temperature is increased to 650°C, steel hardness is reduced and resistance to thermal fatigue deteriorated.

Low-alloy 38CrMoVB10-4 steel features much lower resistance to thermal fatigue as compared to the steels described above. This results from a high thermal expansion factor, low hardness and a large fraction of non-metallic inclusions. The cracks caused by thermal cycles nucleate at the surface of specimens and are distributed perpendicular to the core direction. The boundaries of primary austenite grains and non-metallic inclusions are the initiators of such cracks most frequently. The cracks are propagated mainly along the boundaries of primary austenite grains (Fig. 23) thus chipping off steel particles from the specimens surface.

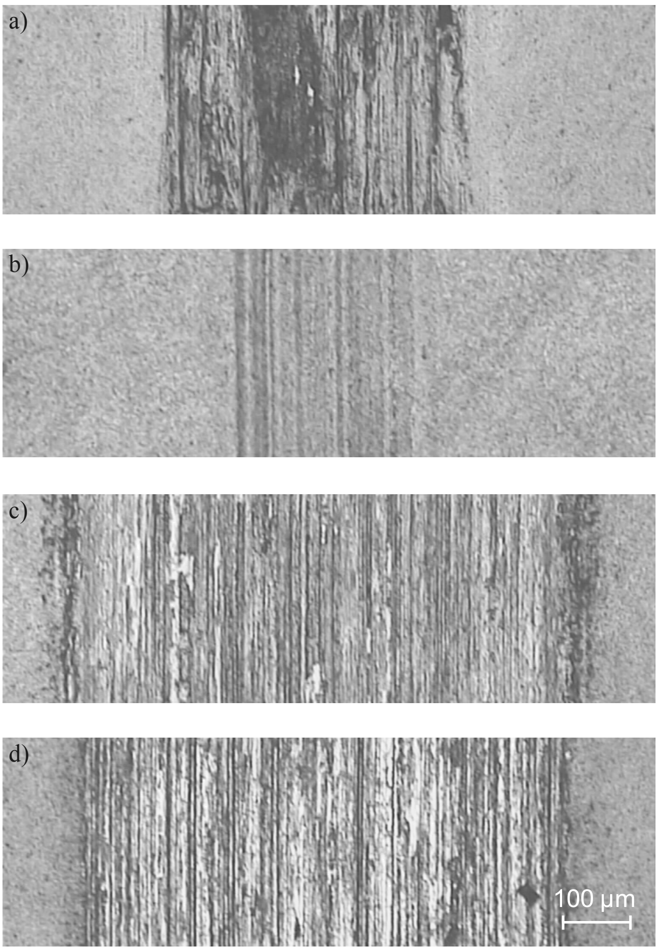

Figure 20. *Wear tracks formed in a pin-on-disc test on X37CrMoV5-1 steel: a) and c) heat treated steel, b) and d) plasma-nitrided steel; a test for 7500 revolutions at a temperature of a) and b) 20°C, and c) and d) 500°C*

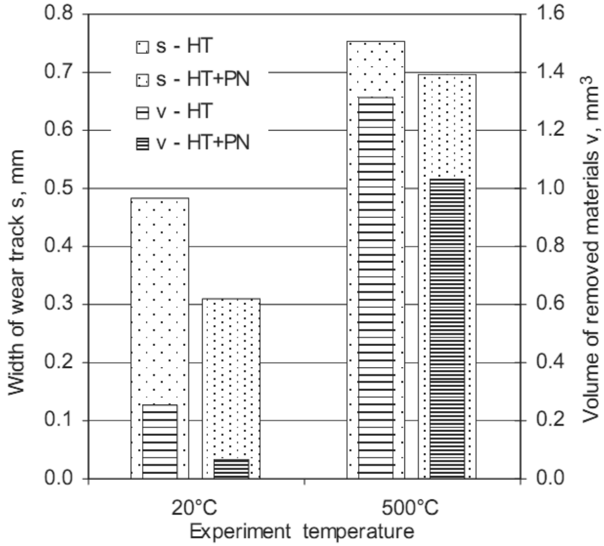

Figure 21. *Width of wear tracks and the volume of the removed material for heat treated (HT) and plasma-nitrided X37CrMoV5-1 steel (PN), subject to a pin-on-disc test (after 7500 revolutions)*

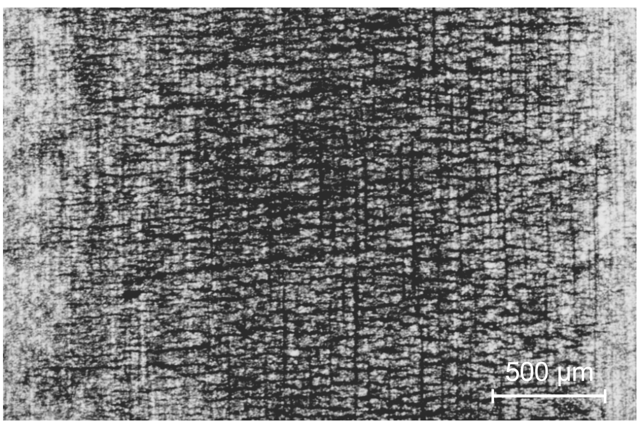

Figure 22. *Cracks at the surface of the X40CrMoV5-1(vac) steel specimen quenched from 1030°C and tempered at 600°C, formed during 5000 thermal cycles within 600-100°C with induction heating*

Thermal cycles cause structure changes in a steel surface layer. It was found that the steels quenched and tempered in the conditions ensuring maximum resistance to cracking at cyclical temperature variations show a structure of tempered martensite with dispersion allow carbides, and in particular for: Cr-Mo steels of 5-1 type – M_7C_3, M_4C_3 and M_3C (Fig. 24), in

40CrWMoVB17-11-16 steel – M_4C_3, M_2C and M_3C, and in 37CrMoB10-4 steel – M_3C. Cyclical heating and cooling during a thermal fatigue test causes martensite to decompose further with the intensity rising along with a higher maximum temperature of a cycle. Alloy cementite is partially dissolved in the tested high-alloy steels and there are more precipitates of more stable carbides, i.e. MC and M_2C type, and the coagulation of M_3C precipitates is experienced for low-alloy 37CrMoB10-4 steel. Steel matrix recovery is taking place along with phase transitions and the growth of carbides. The structural changes caused by cyclical temperature variations reduce the hardness of the surface layer and support the propagation of cracks.

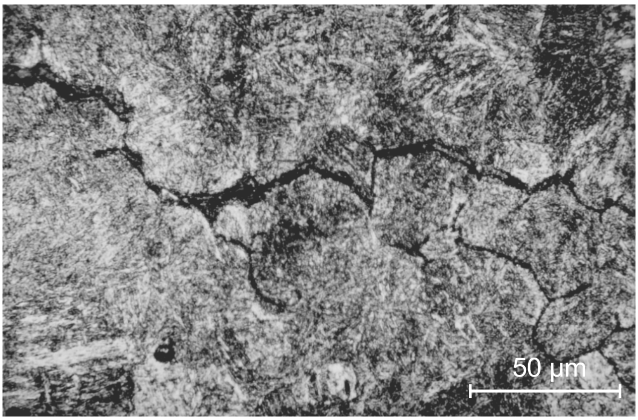

Figure 23. *Microstructure within approx. 0.5 mm from the 38CrMoVB10-4 steel specimen quenched from 890°C and tempered at 500°C, after 3000 thermal cycles within 600-100°C*

Figure 24. *Microstructure of X37CrMoV5-1 steel quenched from 1030°C and tempered at 600°C; thin foil*

The studies made reveal that X40CrMoV5-1 steel is most resistant to the formation of a surface cracks lattice during thermal cycles within 700-100°C. 40CrWMoVB17-11-16 steel shows cracks that are somewhat less deeper after thermal cycles at 600-100°C. The steel, however, contains a considerable concentration allow elements (Table 2) reducing susceptibility to diffusion boriding. As it has been confirmed that a smaller portion of non-metallic inclusions leads to enhanced resistance of steel to cyclic temperature variations, hence further studies after thermochemical treatment were carried out on the specimens made of X40CrMoV5-1 steel after vacuum or electroslag remelting.

A nitrided layer accelerates the nucleation and propagation of cracks during cyclic temperature variations within 600-20°C performed with direct-contact heating at a rate of approx. 85°C/s and cooling in water at a rate of approx. 180°C/s. The depth of cracks is approx. 0.12 mm for steel with a nitrided approx. 0.23 mm thick layer subjected to 25000 thermal cycles. Surface layer hardness in the nitrided specimens declines during thermal cycles because the continuous zone of nitrides is dissolving and because nitride is diffusing deep inside the steel. Diffusion layer hardness is lowering in such conditions causing also the coherent precipitates of $Fe_{16}N_2$ phase to fade. Mass losses for the nitrided specimens and the specimens subjected to thermal fatigue tests with the corrosive impact of air and water are much smaller than for the heat treated specimens. The corrosive destruction processes are present within the surface cracks and the depth increases along with more thermal cycles. Some of the thermal fatigue resistance tests were carried out with a device where specimens were separated from the activity of the atmosphere and the cooling medium by placing them in air-tight heat-resisting steel capsules. Studies into the effect of thermal cycles with the heating rate of approx. 8.5°C/s and cooling speed of approx. 15.5°C/s within 600-100°C on the progress of surface layer cracks point out that cracks in the nitrided specimens are nucleated and propagated much slower than in the borided specimens and only dozen or so % faster than in those quenched and tempered.

4.2. Structure and properties of surface layers of 18CrMnTi4-4 machine steel subject to carburising and carbonitriding

The surface layer of steel after carburising or carbonitriding should contain near its surface approx. 0.8% of C. This was ensured for the tested 18CrMnTi4-4 machine steel through thermochemical treatment in the conditions stated in sub-chapter 2. The depth largely of the surface layer depends on NH_3 addition in the atmosphere in carbonitriding – it increases from approx. 0.51 mm after carburising to 0.65 mm after carbonitriding with the addition of 6% NH_3. This permits, in particular, to produce a surface layer with the required thickness during carbonitriding by approx. 20% faster than during carburising.

The structure of the 18CrMnTi4-4 steel surface layer after carburising at a temperature of 880°C and quenching is composed of partially twinned lath martensite with a high dislocation density (Fig. 25) and of retained austenite with its volume fraction by the surface of approx. 22%. If ammonia is added to the carburising atmosphere, major structure changes are seen in the surface layer. The changes include most of all a retained austenite fraction increased to approx. 39% after carbonitriding with an addition of 6% NH_3 in the atmosphere. This stems from the impact of nitrogen on the decreased initial and final temperature of a martensitic transformation in 18CrMnTi4-4 steel after thermochemical treatment. M_s temperature in the surface layer of carburised steel is approx. 160°C, and it drops to approx. 130°C after carbonitriding with 6% of NH_3 added, and M_f temperature in both cases is below 0°C. The fraction of partially twinned lath martensite in martensite in the surface layer of steel following carbonitriding is going up (Fig. 26), and some martensite grains are characterised by a lath morphology following carbonitriding in an atmosphere with 6% NH_3 added. The dispersion precipitates of cementite occur in the carburised and carbonitrided layer in some areas of martensite. The precipitates are formed during martensite self-tempering. A concentration of carbon in the surface layers is decreasing as the distance from the surface is rising. This, in turn, is mildly reducing hardness to approx. 380 HV 1 in the core (Fig. 27).

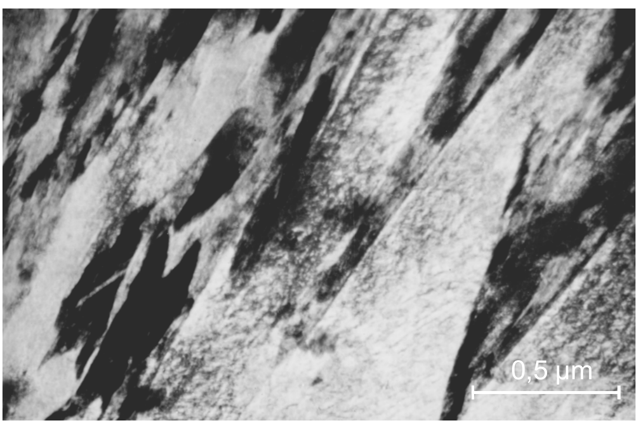

Figure 25. *Microstructure of the surface layer of 18CrMnTi4-4 steel after carburising at a temperature of 880°C and quenching from 840°C, thin foil*

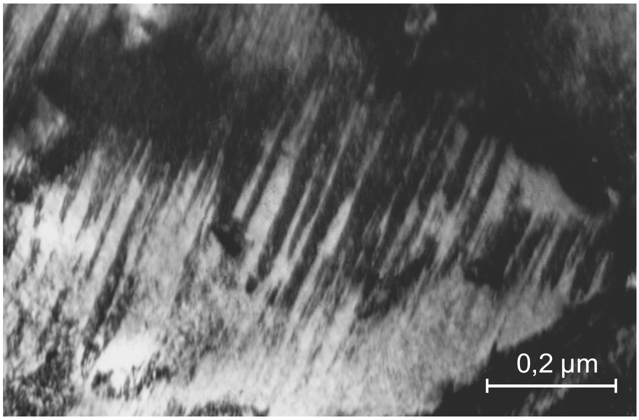

Figure 26. *Microstructure of the surface layer of 18CrMnTi4-4 steel after carbonitriding with addition of 6% of NH_3 at a temperature of 880°C and quenching from 840°C, thin foil*

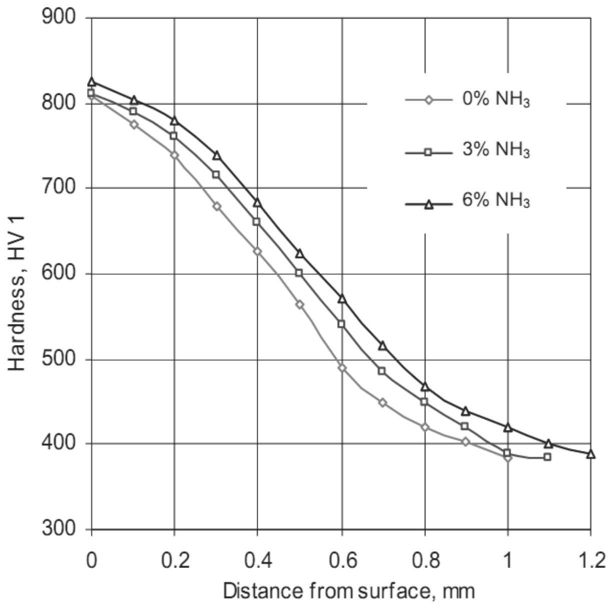

Figure 27. *Hardness distribution in a surface layer of 18CrMnTi4-4 steel after carburising and carbonitriding with addition of 3 and 6% of NH_3, at temperature of 880°C for 5 hrs and quenching from 840°C*

The final properties of carburised or carbonitrided steel are achieved in quenching and low tempering. As a tempering temperature increases, a concentration of carbon in martensite lowers stemming from cementite precipitation and retained austenite transition. Cementite precipitates are formed predominantly at the boundaries of laths and microtwins in martensite (Fig. 28).

Such processes are developing intensively after tempering at a temperature of above. 200°C thus considerably reducing surface layer hardness. Carbonitrided layers are more resistant to tempering however, i.e. structural changes and hardness reduction occur in them after tempering at a slightly higher temperature as compared to carburised layers.

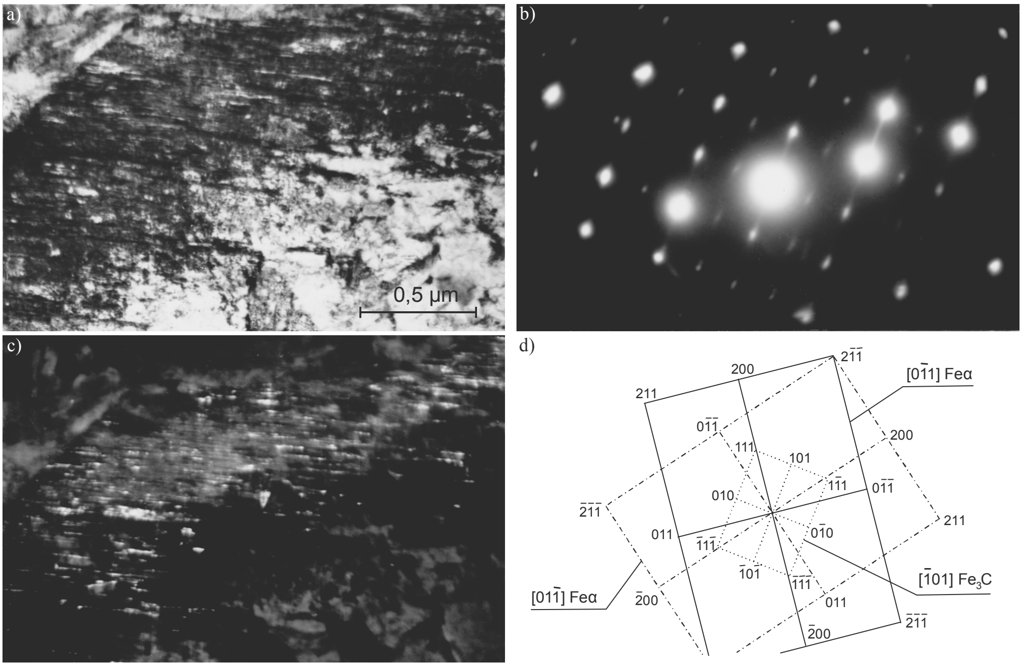

Figure 28. *Surface layer microstructure of 18CrMnTi4-4 steel after carburising at temperature of 880°C, quenching from 840°C and tempering at 300°C, thin foil, a – bright field image, b – diffraction pattern from the area as in Fig. a, c – dark field image from 111 M_3C reflection, d – diffraction pattern solution from Fig. b*

The tests results for 18CrMnTi4-4 steel specimens with a surface layer produced in carburising or carbonitriding show that fatigue strength Z_{gj} has improved and the best properties are ensured by layers approx. 0.4 mm thick containing approx. 27% of retained austenite at the surface. Thicker layers do not ensure improved functional properties important for the strength of toothed gears, similar as a tempering temperature increased above 200°C causing a decreased fraction of retained austenite and the progressing cementite precipitation from martensite.

The surface layers formed during carburising and carbonitriding contribute to improved fatigue strength properties Z_{gj} and the strength of toothed gears. A prerequisite for achieving the most advantageous effect of such thermochemical treatment is identifying the impact of multiple factors on the functional properties of specific parts, including in particular atmosphere composition, carburising temperature, quenching and tempering temperature, which are decisive for layer thickness and structure (especially for a fraction of retained austenite) and properties.

4.3. Cutting ability of high-speed HS6-5-2 steel drills subject to thermochemical treatment

The improvement of operating properties of cutting tools with thermochemical treatment has been known from the scientific and technical literature [4, 6]. The low-temperature methods of such treatment are used most often for high-speed steels, carried out for pre-heat treated tools. The tools are usually subjected to nitriding in different atmospheres; oxynitriding, passivation and more and more often to ion implantation. The numerous studies have shown that the most advantageous operating properties for nitrided cutting tools are ensured by a surface layer with a tempered martensite structure with the dispersion precipitates of $Fe_{16}N_2$, γ'-Fe_4N, ε-$Fe_{2-3}N$ nitrides being arranged uniformly in the matrix. The formation of a continuous zone of nitrides and carbonitrides of ε-$Fe_{2-3}(C,N)$ type at the surface of cutting tools should be prevented during such thermochemical treatment as it increases blade brittleness. Different nitriding methods are used for producing the appropriate surface layer of the tool, especially in the conditions of a limited nitride potential, e.g. in an ammonia atmosphere with addition of nitrogen, nitriding with prior oxidising or plasma nitriding [4, 7, 24, 39]. The life of blades in tools also ameliorates due to a thin layer of spinels being formed on the surface, e.g. Fe_3O_4. The tools are passivated for this purpose in a water vapour atmosphere or oxynitrided. This is done by introducing a water vapour into the nitriding atmosphere when saturating the surface layer with nitride or after finishing this process. The results of works [4, 30, 58] show that oxynitriding is a very effective method of improving the life of tool blades. Considering the different technologies, types of tools and conditions of operating tests used, the results of such tests do not provide a clear answer concerning the efficiency of different thermochemical treatment methods on the life of tools.

To determine, by comparison, the impact of the selected thermochemical treatment methods on the cutting ability of some tools, tests were carried out on twist drills made of the same

HS6-5-2 steel melting. The drills were heat treated in one batch and subjected to the selected thermochemical treatment processes (see sub-chapter 2). The thermochemical treatment applied has an important effect on the life of high-speed HS6-5-2 steel drills (Fig. 29). The drills made of heat treated steel not subjected to any additional thermochemical treatment are wearing fastest in the tested conditions. The average number of openings bored with such drills is 69, and the blade useful life until next sharpening is approx. 3.2 min. Wearing is done by abrasion from the flank and margin side. Scratches and grooves are formed on the flank face, whereas the chamfered corner is worn out only slightly. Local chipping at the cutting blades occurs when using the drills, both, near the margin and near the chamfered corner. The blades of the plasma-nitrided drills show smaller wear from the flank face as compared to the plasma-nitrided drills, but are more brittle.

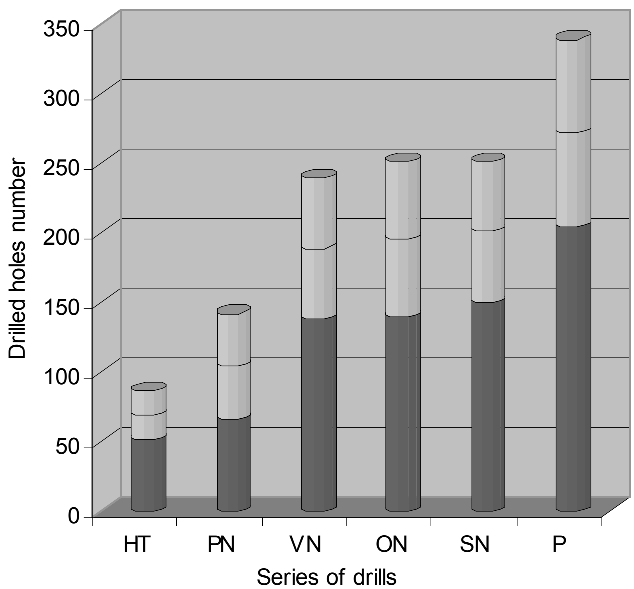

Figure 29. *Cutting ability tests results for drills made of HS6-5-2 steel that was heat treated (HT) and plasma nitrided (PN), sulphonitrided and vacuum nitrided (VN), oxynitrided in fluidised bed (ON), nitrided selectively (SN) and passivated in water vapour (P)*

Important statistical differences were found between the life of the heat treated drills and the drills subjected to other thermochemical treatment methods. The drills that are sulphonitrided and then vacuum nitrided, oxynitrided in a fluidised bed and nitrided selectively feature similar durability (Fig. 29). The average number of openings bored with the drills subject to the above types of thermochemical treatment is, respectively, ca. 189, 196 and 201, and the blade life, respectively, approx. 8.6, 8.9 and 9.2 min. The blade life indices given for the thermochemically treated drills do not differ much, but their life build-up is approx. 170 to 190% against the heat treated drills. The blades of vacuum nitrided, selectively nitrided and oxynitrided drills show the similar symptoms of wear such as material losses at the cutting blade, especially close to the margin and the chamfered corner and also the wear of the surface layer at the flank and face surface. Besides, the sulphonitrided and vacuum nitrided drills show minor blade chipping along the cutting blade and chamfered corner.

The passivation of drills in a water vapour atmosphere ensures much longer blade life with the average number of openings bored with such drills being about 272 (Fig. 29). This corresponds to the blade life of approx. 12.4 min. If we take into consideration the average number of openings, passivation ensures the life increase of approx. 290% for the drills as compared to those heat treated. The relative difference between the average number of openings drilled with such drills and the first blade blunting represents approx. 35-39%. Cutting blades become blunt when using passivated drills, especially near the margin and chamfered corner and the oxides layer on the flank and on the face is being destroyed.

The tests carried out show that the presence of an oxides layer on the surface of 5 mm drills has a very positive effect on their life. The largest number of openings until the first blade blunting was bored with the tools having their oxides surface layer, i.e. passivated, nitrided selectively and oxynitrided. The beneficial effect of passivation on the improved life of a drill blade shows that it is reasonable to use such treatment after sharpening the tool each time and allows to implement this technology at some industrial plants, especially when we realise that the process does not require costly equipment.

4.4. Structure and properties of surface layers of X40CrMoV5-1 steels after boriding

Diffusion boriding is used to a limited extent, however, research is still held to modify and optimise the technological process conditions and improve the quality and the properties for the surface layers produced [59-65]. Boriding is considerably improving the strength of some parts of hot-working machines and tools. Tool steel highly resistant to cyclic temperature variations and with a high austenitisation temperature was chosen as the test material enabling direct quenching from a boriding temperature. A dual-zone layer of borides is forming at the X40CrMoV5-1 steel surface subject to diffusion boriding, i.e. an outer FeB zone with multiple pores and an Fe_2B zone bordering the substrate Fe_2B (Fig. 30). The total thickness of the borides layer is increasing as the boriding time is extending. However, an increase in FeB zone thickness is smaller than the total layer thickness (Fig. 31). Thickness growth for the FeB borides zone is also significantly reduced if a boriding temperature rises above 1000°C. The limited fraction of the FeB phase causes the smaller density of pores and cracks in the surface layer that are forming when cooling the specimens from the boriding temperature. Stresses are produced at the interphase boundary of FeB and Fe_2B borides. The borides are characterised by their high hardness and high elasticity modulus values and different thermal expansion coefficients. The stresses cause cracks perpendicular and parallel to the surface. The cracks perpendicular to the surface are also formed due to the different properties of borides and matrix. During cooling, however, it is possible to partially relax the structural stresses at the interphase boundary of Fe_2B borides with the matrix by deforming austenite or ferrite plastically. The density of surface cracks is rising in heat treatment performed after boriding so that the steel is endowed with its required strength parameters.

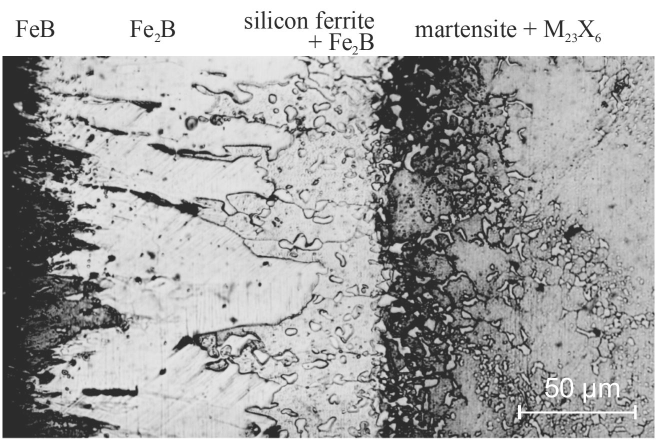

Figure 30. *Elongation of FeB and Fe_2B boride grains in the surface layer of X40CrMoV5-1 steel borided at 1030°C for 6 hrs*

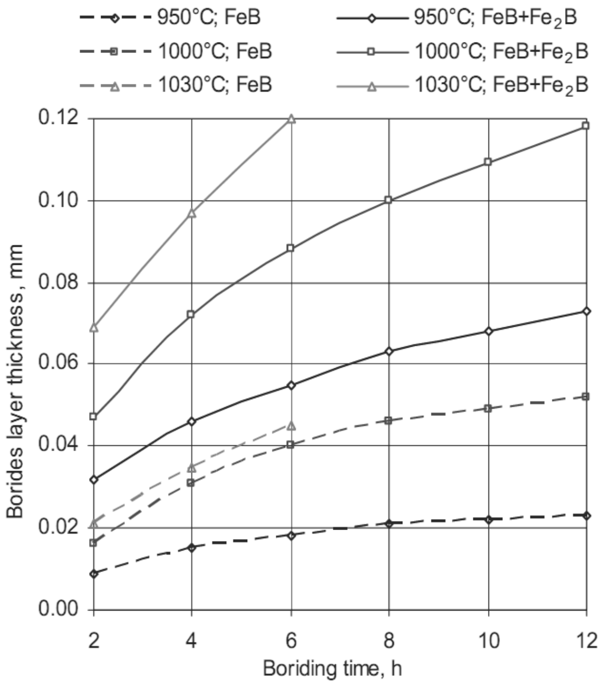

Figure 31. *Effect of boriding temperature and duration on borides layer thickness on X40CrMoV5-1 steel*

Advantageous conditions are achieved when boriding X40CrMoV5-1 steel at a temperature of 1030°C that is equal to the optimum austenitisation temperature. Considering the high hardenability of the tested steel, the hardening operation can be performed when cooling from boriding temperature. It permits to shorten heat treatment duration, reduce the thickness of the FeB phase zone that is usually porous and decrease the density of surface layer cracks. Hardness and resistance to the wear of the borided layer is enhanced at the same time. The hardness of FeB borides measured on the specimens having compact surface layers with sufficient thickness is ca. 1900-2000 HV 0.05. The values, for the significance level of $\alpha = 0.05$, are substantially higher than the hardness of Fe_2B borides of approx. 1800-1950 HV 0.05 (Fig. 32).

FeB and Fe_2B borides have an elongated shape in the direction perpendicular to the surface, i.e. according to the boron concentration gradient diffusing to steel during thermochemical treatment. The orientation of such crystals' phases, and also a relative intensity of X-ray radiation deflected from the individual families of lattice planes different than the intensity for powder preparations imply that boride textures exist in the surface layer. This fact was confirmed with an X-ray texture analysis method by showing that both FeB borides and Fe_2B borides have their axial structure of $\langle 001 \rangle$ type. The formation degree of this texture increases along with a higher temperature and longer boriding time. The structure tests of thin foils in a transmission electron microscope indicate that dislocations and other lattice defects occur in borides, probably packing errors created as the diffusion layer grows. Dislocations in some Fe_2B grains are distributed in the planes {100} (Fig. 33).

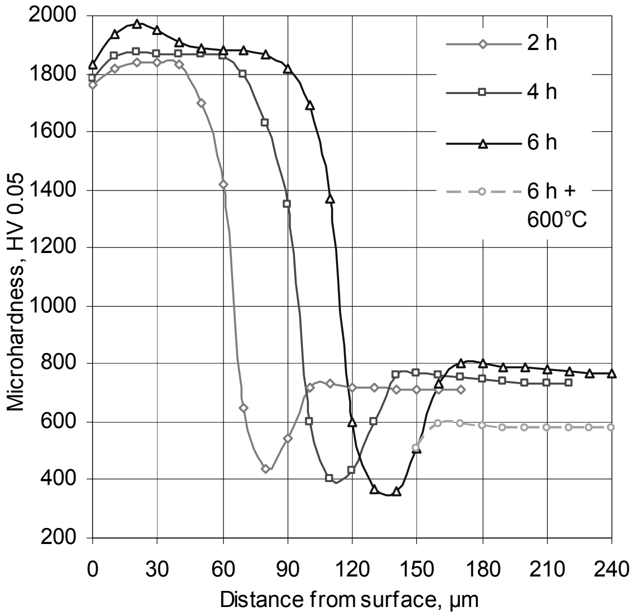

Figure 32. *Microhardness distribution in the surface layer of X40CrMoV5-1 steel borided at temperature of 1030°C for 2 to 6 hrs and directly quenched and tempered twice at 600°C*

Small, elongated areas exist between Fe_2B phase grains with hardness much smaller than the hardness of borides (Fig. 30). These are ferrite grains probably rich in silicon dislodged from the borides layer. The areas show a limited concentration of boron and chromium and other alloy elements in steel, and the carbon concentration does not vary much compared to this existing in Fe_2B borides. The largest concentration of boron in the borided layer occurs, as expected, near the surface, i.e. in a FeB zone. A lower concentration of boron in this zone occurs in the place of cracks and pores by the surface. The boron concentration in the Fe_2B phase

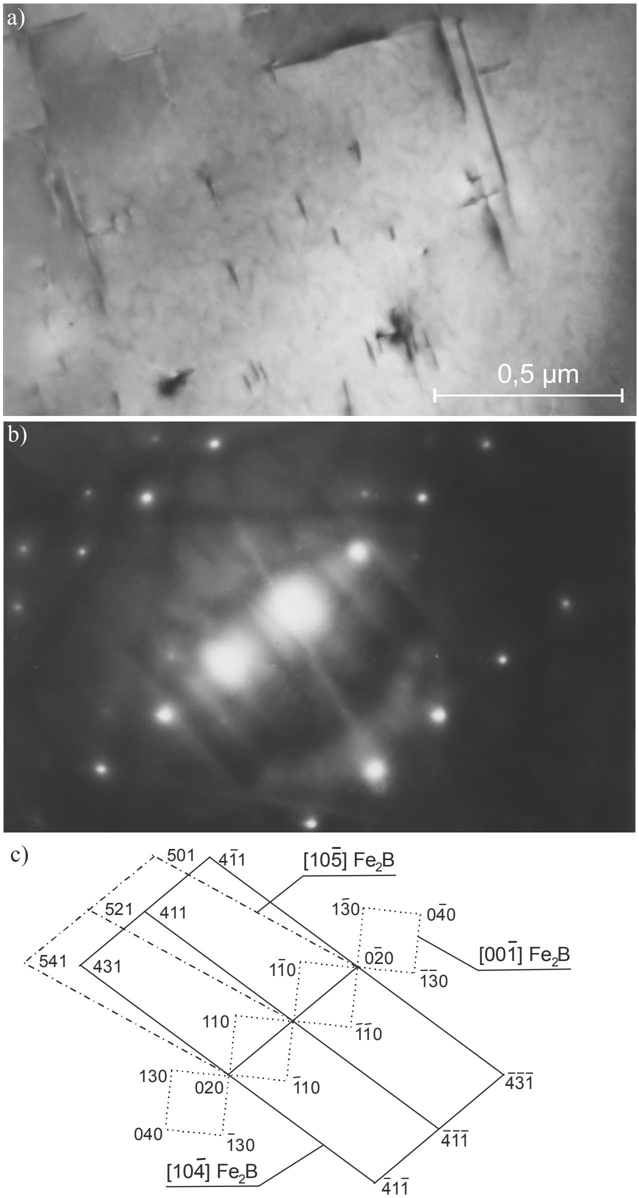

Figure 33. *Dislocation in a Fe_2B boride grain within approx. 0.08 mm from the surface of X40CrMoV5-1 steel borided at 1030°C for 6 hrs; thin foil, a – bright field image, b – diffraction pattern from the area as in Fig. a, c – diffraction pattern solution from Fig. b*

zone maintains at a constant level, and it is sharply reduced underneath the borides layer. Chromium and other carbide-forming elements introduced into steel, i.e. Mo, V and Mn are partly dissolving in Fe_2B borides. The large solubility of chromium in Fe_2B borides is a result of chromium and boron forming an isomorphic Cr_2B phase with Fe_2B with similar lattice parameters. Carbide-forming alloy elements occur in the tested steel in a concentration that is too small to produce separate phases with boron. Silicon and carbon do not dissolve in borides, however, hence the elements are dislodged deep inside the steel.

A silicon ferrite zone is situated immediately underneath the borides layer with the grainy precipitates of Fe_2B, and $M_{23}X_6$ exists slightly deeper (Figs. 30, 34). The silicone concentration increased to approx. 3.5-4.0% exists in the silicone ferrite areas. The quantitative microanalysis method revealed that the grainy precipitates of Fe_2B phase contain, apart from boron, approx. 80% Fe, 8% Cr, 1.5% V and 1% Mo. The zone hardness immediately beneath the borides layer is approx. 340-450 HV 0.05, i.e. much less than the hardness of the specimens core (Fig. 32).

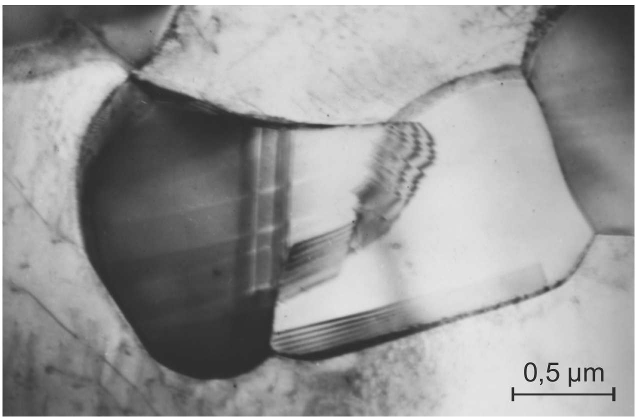

Figure 34. *Microstructure within approx. 0.15 mm from the X40CrMoV5-1 steel surface borided at 1030°C for 4 hrs; thin foil*

Another martensite zone occurs underneath silicone ferrite with retained austenite and multiple, grainy precipitates of a phase with its lattice structure appropriate for $Cr_{23}C_6$ carbide (Fig. 30, 34). These are boron carbides that can be assigned an $M_{23}X_6$ formula where M – Fe, Cr, Mo, V and Mn, and X – C and B. It was found with the X-ray quantitative microanalysis method that $Fe_{17.6}Cr_{4.3}V_{0.7}Mo_{0.3}Mn_{0.1}C_{3.5}B_{2.5}$ formula is corresponding to one of the analysed boron carbides. Hardness in the martensite zone with grainy precipitates $M_{23}(C,B)_6$ is about 600 to 800 HV 0.05 depending on the boriding temperature and the cooling method after boriding. Transition to the core with the hardness of approx. 760 HV 0.05 in steel borided at 1030°C and quenched directly (Fig. 32) occurs only outside that zone. The width of silicone ferrite zones from Fe_2B precipitates and of martensite zones with $M_{23}X_6$ precipitates extends as temperature is rising and boriding time is extending (Fig. 31). Apart from the phases mentioned, a small fraction of VC and M_7C_3 carbide precipitates (Fig. 30) not dissolved in austenite during boriding is present across the section of the entire diffusion zone.

When austenitising steel that was borided in prior at 950°C, the concentration of alloy and carbon elements is partially homogenised underneath a borides layer, thus contributing to small growth in hardness after quenching by approx. 30 HV 0.05 in the silicon ferrite zone and to growth in core hardness to approx. 760 HV 0.05. The tested steel, that in prior was borided and quenched, is quenched without causing structural changes in the borides layer. However, retained austenite is transformed in the matrix and M_7C_3 and M_4C_3 carbides are precipitated from lath martensite and the recovery of a solid solution begins. This leads to the smaller matrix hardness of approx. 560 to 580 HV 0.05 (Fig. 32).

If specimens with surface layers are cyclically heated and cooled, much faster nucleation and cracks development is seen than in the case of heat treated specimens. This is caused by the structural stresses formed during thermal cycles between the diffusion surface layer and the steel matrix with different physiochemical properties. A deformation occurring as a result of such stresses can be compensated by a plastic deformation of the matrix or by the cracking of the borides layer. Stresses and deformations in the surface layer are even greater in case of rapid temperature variations in a thermal cycle. It was concluded as a result of the tests carried out in equipment with direct-contact heating that cracks are nucleated in the heat treated specimens after approx. 20000 when heating at a rate of approx. 85°C/s and water cooling at a rate of approx. 180°C/s within 600-20°C. On the other hand, the density of surface cracks in the borided specimens is much higher already after 5000 thermal cycles. Some of the cracks in the borided specimens are initiated by cracks in the surface layer after thermochemical treatment. The lattice of surface cracks is becoming more and more evident as the thickness of borides layer and the number thermal cycles grow due to the higher depth, density and width of cracks (Fig. 35). Micro-cracks are growing fastest in borides zone along the boundaries of grains and through the crystals of such phases. The rate of cracks growth is declining in the silicone ferrite zone as stresses may relax in the plastic matrix. Once the crack face passes silicone ferrite to the martensite zone tempered with a large fraction of $M_{23}X_6$ phase, the intensity of corrosion destruction of steel is accelerated.

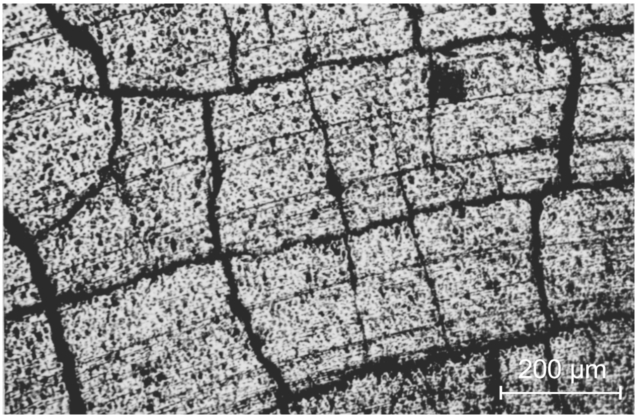

Figure 35. *Cracks lattice at the surface of the X40CrMoV5-1 steel specimen borided at a temperature of 950°C for 8 hrs, quenched from 1030°C and tempered twice at 600°C, formed during 25000 thermal cycles within 600-20°C*

The tests of influence of thermal cycles with the heating rate of approx. 8.5°C/s and cooling rate of approx. 15.5°C/s within the range of 600-100°C on the progress of surface layers cracks in the samples isolated from the activity of atmosphere and cooling medium and placed in air-tight heat-resisting steel capsules reveal that the first cracks in the heat treated specimens are formed after approx. 5000 cycles. The number of cracks in borided samples, however, rises significantly already after 2500 cycles. The depth of maximum cracks in steel with a thick borides layer is rising insignificantly to approx. 10000 thermal cycles due to a broad silicone ferrite zone. More thermal cycles causes the propagation of cracks to the tempered martensite zone with the grainy precipitates of $M_{23}X_6$ phase having smaller cracking resistance. The tests of solid specimens heated through radiation and convection at a rate of approx. 4°C/s and cooled in water at a rate of approx. 47.5°C/s within 600-80°C enable to identify the portion of corrosion processes in the destruction of the steel surface layer. The specimens without surface layers are intensively destroyed by corrosion. In this case the surface is oxidising and an oxides layer is cracking and chipping during cooling. The fusion-borided specimens corrode much slower and higher wear resistance should also be expected until hard borides are preserved in the surface layer. This ensures enhanced resistance for many hot-work tools subjected to boriding as compared to those heat treated. Diffusion boriding has not been used broadly in industrial manufacturing due to technological reasons, however.

5. Technology Roadmapping

The results of foresight research and the results of traditional materials science research have set a basis for creating a series of roadmaps for the technology groups considered. Table 5 provides a representative technology roadmap prepared for carburising and carbonitriding, and an aggregate list containing data provided in all the roadmaps produced for this chapter for the selected steel thermochemical treatment technology groups presented in Table 6. Technology information sheets were also prepared for the research undertaken that detail out and supplement the technology roadmaps. The sheets present technical information being very helpful for the implementation of the technology in the industrial practice, especially in SMEs not having the capital allowing to conduct own research in this field. Table 7 presents selected data stated in the technology information sheets concerning the specific analysed groups of steel thermochemical treatment technologies concerning the impact of technology application on the predicted and expected material properties, the efficiency of preventing the consequences of wear, industry section acc. to the PKD classification having the highest technology applicability. The level of the individual analysis factors has been expressed in relative values using a universal scale of relative states presented in 2.1. of the chapter (Table 1).

6. Summary

Thermochemical treatment methods, especially the different variants of carburising and nitriding, are still being extensively applied to produce surface layers ensuring the enhanced functional properties of machine and tool parts. This mainly relates to surface layers with their thickness between several decimals of mm to over 1 mm, with the mild transition of properties from the part surface to the part core. Investigations are still being conducted aimed at improving the functional properties of the thermochemically treated parts by modifying the conditions during diffusion saturation (temperature variations, saturating medium composition, activating factors) and during heat treatment prior to nitriding or after carburising.

The foresight-materials science research undertaken as part of the work, being part of a wider project aimed at selecting the priority, innovative material surface engineering technologies [66-70], has revealed the stable and certain development outlooks for nitriding as well as

Table 5. *An example technology roadmap made for carburising and carbonitriding*

TECHNOLOGY ROADMAP		Technology name: *Carburising and carbonitriding* Research scope: *Thermochemical technologies*		Catalogue No. *M4-11-2010/11*
When?	Time intervals	TODAY 2010-11	2020	2030
	All-society and economic perspectives	Creating the Critical Technologies Book	Development of priority innovation technologies	Statistically high quality of technologies implemented in industry
		Creating future events scenarios	Using chances and avoiding difficulties	Sustainable development
		Development of information society and intellectual capital	Wide education and effective intensive coopera-tion between Science and Industry representatives	Knowledge- and innovativeness-based economy
Why?	Strategy for technology	**Strategy of a dwarf mountain pine in autumn.** "To milk the cow" as long as possible strengthening technology attractiveness. To profit the realization of production in a sable, practicable environment using solid technology which should be modernized and intensively promoted in order to strengthen its attractiveness		
	Environment influence	Rainy autumn		
	Technology value	Rooted dwarf mountain pine		
What?	**Product**	Toothed gears, worms, pinions, crankshafts and camshaft, piston pins, piston rings, shafts of large rolling bearings, pins of slide bearings, sleeves of diesel engine cylinders for tracks and others		
	Product quality at the background of foreign competitors	Moderate (6)	Moderate (6)	Medium (5)
	Substrate	Low-carbon non-alloy and alloy steels for carburising, e.g.: C10E, C15R, 17Cr3, 12CrMo4, 14NiCrMo13-4, alloy steels for carburising for rolling bearings, e.g.: 20Cr3, 13MoCrNi42-16-14, alloy machine steels with C contents between 0.07 to 0,24%		
	Kind of surface coatings/ layers/ processes on substrate surface	Diffusion carburised layer hardened by quenching and low-temperature tempering		
	Improved material properties	Hardness, resistance to strain wear, adhesive wear and abrasive wear, resistance to pitting and spalling, contact strength (to fretting and scuffing), erosion strength		
	Diagnostic-research equipment	Light microscope, hardness and microhardness tester or optional: transmission and scanning electron microscopes, X-ray diffractometer, work-stand for testing of thermo-mechanical fatigue strength, abrasion and corrosion resistance		
How?	**Technology**	Carburising and carbonitriding		
	Life cycle period	Late mature (4)	Base (3)	Base (3)
	Production type	Serial and mass production	Serial and mass production	Serial and mass production
	Production organisation form	Non-direct-line production at line and in cells	Non-direct-line p. at line, synch. direct-line p..	Non-direct-line p., synch. & aut.. direct-line p
	Machine park modernity	Medium (5)	Quite low (4)	Low (3)
	Automation & robotisation	Moderate (6)	Medium (5)	Medium (5)
	Quality and reliability	Moderate (6)	Medium (5)	Medium (5)
	Proecology	Quite low (4)	Quite low (4)	Medium (5)
Where?	Organisation type	Small-, medium- and large sized enterprises, microenterprises, R&S centres	Large-sized as well as small- and medium-sized enterprises, microenterprises	Large-sized as well as small- and medium-sized enterprises, microenterprises
	Represented industry	Automotive, machine (for mining sector, agriculture, motor industry), aviation and electrical ones		
Who?	Staff education level	Quite low (4)	Quite low (4)	Quite low (4)
	Engagement of scientific-research staff	Quite low (4)	Quite low (4)	Quite low (4)
How much?	Capital requirements	Moderate (6)	Moderate (6)	Medium (5)
	Production size determining profitability in enterprise	High (8)	Quite high (7)	Moderate (6)
	Production size in the country	Very high (9)	High (8)	Moderate (6)

LEGEND: ⟹ Cause and effect connections; ········► Capital connections; ·······► Time corelations; ◄──► Two-way transfer of data and/or resources carburising and carbonitriding. The technology groups referred to above are characterised by high potential and a neutral environment enabling slow but steady progress. In the future, after having eliminated the difficulties originating from the environment, it may become even more supportive. Such development of events, however, is conditioned, in case of nitriding, by the swift development of the most prospective of its variant, i.e. plasma nitriding (ion, glowing nitriding) and hybrid technologies with nitriding (e.g. in combination with Physical Vapour Deposition – PVD) with the growth of low-pressure nitriding maintaining at the current level and the declining importance of conventional gas nitriding. The optimistic variant of carburising and carbonitriding development requires that such technologies strengthen and grow as: carburising in a controlled atmosphere and variants of carbonitriding, enabling direct quenching and low-temperature tempering of the treated parts. The development of nitriding and carburising and carbonitriding must be also accompanied by an improved ecological aspect of the discussed technologies to minimise the harmful substances emitted to the environment. The weakest development prospects are exhibited by diffusion boriding with its significance most likely declining within the nearest 20 years. This stems from limited effectiveness, high costs and the unfavourable environmental impact of the existing boriding technologies. Another scenario is only possible if there is a major breakthrough by finding a new, wide range of industrial applications and a spectacular improvement in the currently used solutions, especially for environmental protection.

Table 6. *Selected main source data used for preparation of technology roadmaps for selected groups of thermochemical treatment, as follows: (A) nitriding and its variants, (B) carburising and carbonitriding, (C) diffusion boriding*

No.	Analysed factor		Time interval	Analysed technology		
				A	B	C
1.	All-society and economic perspectives	1st trend	2010	Creating the Critical Technologies Book		
			2020	Development of priority innovation technologies		
			2030	Statistically high quality of technologies implemented in industry		
		2nd trend	2010	Creating future events scenarios		
			2020	Using chances and avoiding difficulties		
			2030	Sustainable development		

<table>
<tr><td rowspan="2">No.</td><td rowspan="2" colspan="2">Analysed factor</td><td rowspan="2">Time interval</td><td colspan="3">Analysed technology</td></tr>
<tr><td>A</td><td>B</td><td>C</td></tr>
<tr><td rowspan="3"></td><td rowspan="3"></td><td rowspan="3">3rd trend</td><td>2010</td><td colspan="3">Development of information society and intellectual capital</td></tr>
<tr><td>2020</td><td colspan="3">Wide education and effective intensive cooperation between Science and Industry representatives</td></tr>
<tr><td>2030</td><td colspan="3">Knowledge-based economy</td></tr>
<tr><td>2.</td><td colspan="2">Strategy for technology (the most probable one)</td><td>2010-2030</td><td>Strategy of a dwarf mountain pine in autumn</td><td>Strategy of a dwarf mountain pine in autumn</td><td>Strategy of an aspen in autumn</td></tr>
<tr><td>3.</td><td colspan="2">Environment influence</td><td>2010-2030</td><td colspan="3">Rainy autumn</td></tr>
<tr><td>4.</td><td colspan="2">Technology value</td><td>2010</td><td>Rooted dwarf mountain pine</td><td>Rooted dwarf mountain pine</td><td>Quaking aspen</td></tr>
<tr><td>5.</td><td colspan="2">Product</td><td>2010-2030</td><td>Forging dies, drawing dies, mouldings for plastics, parts of injection moulders and extruding machines, crankshafts, shafts, piston rings and pins, parts of engines and pumps, cutting tools (milling cutters, drills, screw taps), tools for precision die cutting, medical technology tools, parts of worm gears, of electromagnetic clutches, part of sensor technology and others</td><td>Toothed gears, worms, pinions, crankshafts and camshaft, piston pins, piston rings, shafts of large rolling bearings, pins of slide bearings, sleeves of diesel engine cylinders for tracks and others</td><td>Mining / drilling tools, working parts of road machines and special machines, including caterpillars, casting equipment, parts of pumps and valves, tools not subjected to dynamic loads (drawing dies, plugs for drawing pipes), parts of weapon, parts of clothing industry machines and others</td></tr>
<tr><td rowspan="3">6.</td><td rowspan="3" colspan="2">Product quality at the background of foreign competitors</td><td>2010</td><td>Quite high (7)*) or Medium (5)**)</td><td>Moderate (6)</td><td>Quite low (4)</td></tr>
<tr><td>2020</td><td>Quite high (7)*) or Medium (5)**)</td><td>Moderate (6)</td><td>Quite low (4)</td></tr>
<tr><td>2030</td><td>Quite high (7)*) or Quite low (4)**)</td><td>Medium (5)</td><td>Quite low (4)</td></tr>
<tr><td>7.</td><td colspan="2">Improved material properties</td><td>2010-2030</td><td>Fatigue strength (mainly to pitting), hardness, wear resistance, resistance to erosion, corrosion, contact strength (to fretting and scuffing)</td><td>Hardness, resistance to strain wear, adhesive wear and abrasive wear, resistance to pitting and spalling, contact strength (to fretting and scuffing), erosion strength</td><td>Hardness, resistance to high temperature, wear resistance, resistance to erosion and corrosion</td></tr>
</table>

No.	Analysed factor	Time interval	Analysed technology		
			A	B	C
8.	Diagnostic-research equipment	2010-2030	Light microscope, hardness and microhardness tester, optionally: scanning and transmission electron microscopes, X-ray diffractometer, work-stand for testing of thermo-mechanical fatigue strength, abrasion and corrosion resistance		
9.	Life cycle period	2010	Early mature (6)- plasma nitriding Growth (7)- low pressure nitriding Late mature (4)- gas nitriding	Late mature (4)	Late mature (4)
		2020	Early mature (6)- plasma nitriding Growth (7)- low pressure nitriding Base (3)- gas nitriding	Base (3)	Late mature (4)
		2030	Early mature (6)- plasma nitriding Growth (7)- low pressure nitriding Base (3)- gas nitriding	Base (3)	Late mature (4)
10.	Production type	2010	Small- and medium-scale serial Large-scale serial and mass- only *)	Serial and mass	Small- and medium-scale serial
		2020		Serial and mass	Small- and medium-scale serial
		2030		Serial and mass	Small- and medium-scale serial
11.	Production organisation form	2010	Non-direct-line production in cells and at line	Non-direct-line production in cells and at line	Non-direct-line production in process cells
		2020	Non-direct-line production in cells and at line, synchronic direct-line production	Non-direct-line production at line , synchronic direct-line production	Non-direct-line production in process cells
		2030	Non-direct-line production in cells and at line, synchronic and automated direct-line production	Non-direct-line production at line, synchronic and automated direct-line production	Non-direct-line production in process cells
12.	Machine park modernity	2010	Quite high (7)*) or Medium (5)**)	Medium (5)	Quite low (4)
		2020	Quite high (7)*) or Quite low (4)**)	Quite low (4)	Low (3)
		2030	High (8)*) or Quite low (4)**)	Very high (9)	Low (3)

No.	Analysed factor	Time interval	Analysed technology		
			A	B	C
13.	Automation and robotisation	2010	Moderate (6)	Moderate (6)	Low (3)
		2020	Moderate (6)	Medium (5)	Low (3)
		2030	Moderate (6)	Medium (5)	Low (3)
14.	Quality and reliability	2010	Moderate (6)	Moderate (6)	Quite low (4)
		2020	Quite high (7)*) or Quite low (4)**)	Medium (5)	Low (3)
		2030	Quite high (7)*) or Quite low (4)**)	Medium (5)	Low (3)
15.	Proecology	2010	Quite high (7)*) or Quite low (4)**)	Quite low (4)	Low (3)
		2020	Quite high (7)*) or Quite low (4)**)	Quite low (4)	Low (3)
		2030	Quite high (7)*) or Quite low (4)**)	Medium (5)	Low (3)
16.	Organisation type	2010	Higher education institutions, research institutes, small- and medium-sized enterprises	Small-, medium- and large sized enterprises, microenterprises, R&S centers	Small-, medium- and large sized enterprises
		2020	Higher education institutions, research institutes, small- and medium-sized enterprises	Large-sized as well as small- and medium-sized enterprises, microenterprises	Small-, medium- and large sized enterprises
		2030	Higher education institutions, research institutes, small- and medium-sized enterprises	Large-sized as well as small- and medium-sized enterprises, microenterprises	Small-, medium- and large sized enterprises, microenterprises
17.	Represented industrial branches	2010-2030	Automotive industry, machine industry, tool industry, aviation industry, ship industry	Automotive industry, machine industry (for mining sector, agriculture, motor industry), aviation industry, electrical industry	Machine industry (especially for road and mining sector), electrical machine industry, electrical industry
18.	Staff education level	2010	High (8)*) or Quite low (4)**)	Quite low (4)	Quite low (4)
		2020	High (8)*) or Quite low (4)**)	Quite low (4)	Quite low (4)

No.	Analysed factor	Time interval	Analysed technology		
			A	B	C
		2030	High (8)*) or Quite low (4)**)	Quite low (4)	Quite low (4)
19.	Engagement of scientific-research staff	2010	Quite high (7)*) or Quite low (4)**)	Quite low (4)	Medium (5)
		2020	Quite high (7)*) or Quite low (4)**)	Quite low (4)	Medium (5)
		2030	Quite high (7)*) or Low (3)**)	Quite low (4)	Medium (5)
20.	Capital requirements	2010	Quite high (7)*) or Quite low (4)**)	High (8)	Quite low (4)
		2020	Quite high (7)*) or Medium (5)**)	Moderate (6)	Quite low (4)
		2030	Quite high (7)*) or Medium (5)**)	Moderate (6)	Quite low (4)
21.	Production size determining profitability in firm	2010	Moderate (6)	Medium (5)	Quite low (4)
		2020	Moderate (6)	Quite high (7)	Low (3)
		2030	Moderate (6)	Moderate (6)	Low (3)
22.	Production size in the country	2010	Moderate (6)	Very high (9)	Quite low (4)
		2020	Moderate (6)	High (8)	Low (3)
		2030	Moderate (6)	Moderate (6)	Low (3)

*) for plasma nitriding and for low-pressure nitriding
**) for gas nitriding

Table 7. *The selected data provided in the technology information sheets concerning the relevant, analysed groups of thermochemical steel treatment, as follows: (A) nitriding and its variants, (B) carburising and carbonitriding, (C) diffusion boriding*

No.	Analysed factor	Analysed technology group					
		A		B		C	
1.	Impact of technology application on the predicted and expected material properties	Properties	Level	Properties	Level	Properties	Level
		Fatigue strength	Quite high (7)	Fatigue strength	Medium (5)	Hardness	Quite high (7)
		Erosion resistance	Quite high (7)	Hardness	Medium (5)	Resistance to high temperature	Quite high (7)
		Hardness	Quite high (7)	Ductility	Medium (5)	Wear resistance	Quite high (7)

No.	Analysed factor	Analysed technology group					
		A		B		C	
		Wear resistance	Quite high (7)	Wear resistance	Medium (5)	Erosion resistance	Moderate (6)
		Corrosion resistance	Medium (5)	Erosion resistance	Medium (5)	Corrosion resistance	Quite low (4)
2.	Efficiency of preventing the consequence s of wear	Wear mechanisms	Level	Wear mechanisms	Level	Wear mechanisms	Level
		Strain and fatigue corrosion	Quite high (7)	Strain wear	Moderate (6)	Abrasive wear	High (8)
		Attrition wear	Quite high (7)	Pitting	Moderate (6)	Attrition wear	Quite high (7)
		Fretting	Quite high (7)	Spalling	Moderate (6)	Corrosion determined by contact with liquid Zn or Al	Quite high (7)
		Pitting	Quite high (6)	Abrasive wear	Medium (5)	Uniform corrosion	Medium (6)
		Erosion	Moderat e (6)	Fretting	Medium (5)	Local and pitting corrosion	Medium (5)
3.	Industry section acc. to the PKD nationwide classifica-tion with the highest technology applicability	Industry sections	Level	Industry sections	Level	Industry sections	Level
		Scientific research and development works	High (8)	Production of metal ready products excluding machinery and equipment	High (8)	Production of metal ready products excluding machinery and equipment	Quite low (4)
		Manufacture of vehicles, semi-trailers and trailers	Quite high (7)	Manufacture of vehicles, semi-trailers and trailers	High (8)	Scientific research and development works	Quite low (4)
		Manufacture of other transport equipment	Moderat e (6)	Production of machinery and equipment not elsewhere classified	Quite high (7)	Production of machinery and equipment not elsewhere classified	Medium (5)
		Production of metal ready products excluding machinery and equipment	Moderat e (6)	Scientific research and development works	Quite high (7)	Manufacture of vehicles, semi-trailers and trailers	Quite low (4)
		Production of machinery and equipment not elsewhere classified	Quito low (4)	Manufacture of other transport equipment	Moderate (6)	Architectural and engineering activity; technical testing and analyses	Low (3)

When evaluating the importance of the selected groups of steel thermochemical technologies presented in the chapter, their broad scale of contemporary applications in the industry should be emphasised, and in many cases the fact that they cannot be replaced with reasonable alternatives having similar costs. Hence, they will certainly still be important in the nearest 20 years amongst other technologies of engineer materials surface engineering, which justifies their position in The Critical Technologies Book.

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10 Strategic development perspectives of laser processing on polycrystalline silicon surface

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Abstract

Purpose: *The goal of this chapter is to evaluate the strategic perspectives of polycrystalline silicon texturisation according to custom foresight methodology. The texturing type was the technology division criterion. Thus, in the chapter three technologies, as following: polycrystalline silicon texturisation by alkaline etching, laser treatment and laser treatment with chemical etching were compared.*

Design/methodology/approach: *In the framework of the foresight-materials science research, a foresight matrices set were prepared, the strategic development tracks were determined, as well as materials science experiments using a Nd:YAG laser, a scanning electron microscope, a confocal laser scanning microscope and a spectrophotometer were conducted. Finally, on the basis of the obtained results the technology roadmaps were prepared.*

Findings: *The carried out research pointed out the industrial importance of polycrystalline silicon texturisation and good perspectives for these technology groups.*

Research limitations/implications: *Research concerning polycrystalline silicon texturisation constitute a part of a larger research project aimed at identifying, researching, and characterising the priority innovative technologies in the field of materials surface engineering.*

Practical implications: *The presented results of experimental materials science research were proved the significant positive impact of texturisation on the structure and mechanical properties of polycrystalline silicon surface layers, which leads to the justification of their including into the set of priority innovative technologies recommended for application in industrial practice.*

Originality/value: *The novelty of this chapter is to evaluate the value of polycrystalline silicon texturisation in the background environment with their future development perspectives determination.*

Keywords: *Manufacturing and processing; Surface treatment; Polycrystalline silicon texturisation; Foresight; Technology Roadmapping*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, A. Drygała, Strategic development perspectives of laser processing on polycrystalline silicon surface, Archives of Materials Science and Engineering 50/1 (2011) 5-20.

1. Introduction

In line with the European Union's development strategy formulated in the recent years called Europe 2020, it is indispensable to undertake comprehensive measures at a European, national and regional level aiming to support a more effective, competitive and low-emission economy based on knowledge and innovation ensuring high employment as well as social and territorial cohesion. It is envisaged that the level of investments for R&D and innovation until 2020 is to reach 3% of the EU's GDP from public and private funds. For the economic and social effects achieved to be satisfactory, the stream of investments has to be channelled into those fields of science and industries bringing the highest added value, with special consideration given to the role of small- and medium-sized enterprises representing 99.8% of all Polish enterprises generating 68% of the GDP. The outcomes presented have been decisive for a lively interest in the recent decade in technology foresight the purpose of which is to identify the priority innovative technologies and directions of their strategic development, also with regard to material engineering [1-5]. An analysis of the results and scope of such foresight research and development trends in the industry observed in technologically advanced countries has set a basis for conducting technology foresight for materials surface engineering [6]. Nearly 300 independent foreign and domestic experts representing scientific, business and public administration circles have taken part in the FORSURF technology foresight up till now at the different stages of works. They have completed approx. 600 multi-question surveys and held thematic discussions during 7 Workshops. 14 thematic areas with 10 critical technologies having the best development prospects and/or being of key significance for the industry within the nearest 20 years were chosen as a result of the preliminary studies. A set of 140 critical technologies was thoroughly analysed according to three iterations of the Delphi method carried out in consistency with the idea of e-foresight [7] using information technology encompassing a virtual organisation, web platform and neural networks. Laser technologies in surface engineering including the laser texturisation of polycrystalline silicon with chemical etching were among the thematic areas analysed highly rated by the experts.

An interest in polycrystalline silicon texturisation technologies stems from economic reasons. The contemporary industry experiences growing demand for energy accompanied by the gradual depletion of the most conventional energy sources (hard coal and lignite, peat, crude oil and natural gas), constantly rising prices and supplies uncertainty for such natural fuels as well as controversies evolving concerning nuclear fuel security (uranium 235). Those factors combined with endeavours to reduce the greenhouse effect and the emissions of pollutions to the natural environment are contributing to a growing interest in the sourcing of renewable energy: solar energy, wind energy, hydro energy, geothermal energy, energy of sea currents, tidal energy and wave energy, thermal ocean energy as well as the manufacture of biofuels, biomass and biogas [8-11]. Boost in demand and the related growth in the industrial production of photovoltaic cells permitting to converts solar radiation energy directly into electric energy has been inscribing in this trend. The following is notably used for producing solar cells: gallium arsenide (GaAs), cadmium telluride (CdTe), copper-indium selenide ($CuInSe_2$), indium phosphide (InP), however a 95% market share is held by silicon (Si). The dominant role of silicon in this field is highly substantiated as it is a second, after oxygen, most widespread element on the Earth with its share in the earth crust of 27%. It occurs in nature most often in combination with oxygen in form of silica SiO_2 [12-14]. Solar cells are made from mono- and polycrystalline silicon. The cells made from monocrystalline silicon that is characterised by the ordered spatial arrangement of atoms with the same orientation of all elementary network cells in the entire volume of crystal are achieving high efficiencies, but are relatively expensive, however. A polycrystalline silicon crystallisation process with the ordered structure of grains having, however, a random crystallographic orientation, occurs at a much higher speed and consumes less energy, hence it is cheaper. A disadvantage of this solution is the presence of structural defects and, as a result, the efficiency of polycrystalline cells is lower by approx.

2-3% in respect of monocrystalline cells [8, 9]. Economic calculation justifies, therefore, scientific studies into the development of new technologies of producing solar cells from poly-crystalline silicon with higher efficiencies as compared to the situation seen up till now.

The purpose of this interdisciplinary study is a comparative analysis of three alternative technologies of polycrystalline silicon texturisation using a custom foresight-materials science methodology [15]. The subject of the comparative studies are the results of investigations into the optical properties of polycrystalline silicon and electrical properties of the photovoltaic cells made of them and the results of expert studies presenting the value of the individual polycrystalline silicon texturisation technologies against the environment together with the recommended management strategies and forecast strategic development tracks. The chapter also presents the outcomes of foresight research pertaining to the position of laser technologies against other surface engineering technologies, including laser texturisation of polycrystalline silicon with chemical etching. Technology roadmaps have been established according to the results of the foresight and material sciences research being a comparative analysis tool especially helpful for small- and medium-sized enterprises not having funds for conducting own research in this domain.

2. Polycrystalline silicon texturisation usefulness and methods

High-efficiency photovoltaic cells require that optical losses are minimised extensively by decreasing the coefficient of solar radiation reflection from the illuminated surface. Electromagnetic radiation photons reaching a semiconductor surface may be either reflected from the surface, absorbed or transit through the material (Fig. 1a). In terms of photovoltaics, reflection and transmission are undesired as the unabsorbed photons cannot take part in the photovoltaic effect [8-10, 16, 17]. The $R(\lambda)$ solar radiation reflection coefficient for silicon wafers subjected to etching to remove damages made due to cutting is within 35-50% for the wavelength of about 400-1100 nm. Two methods exist for reducing the coefficient: deposition of the antireflective coating (ARC) and silicon surface texturing [17]. Through solar cell surface texturing, the photon reflected from the surface can be absorbed again (Fig. 1b).

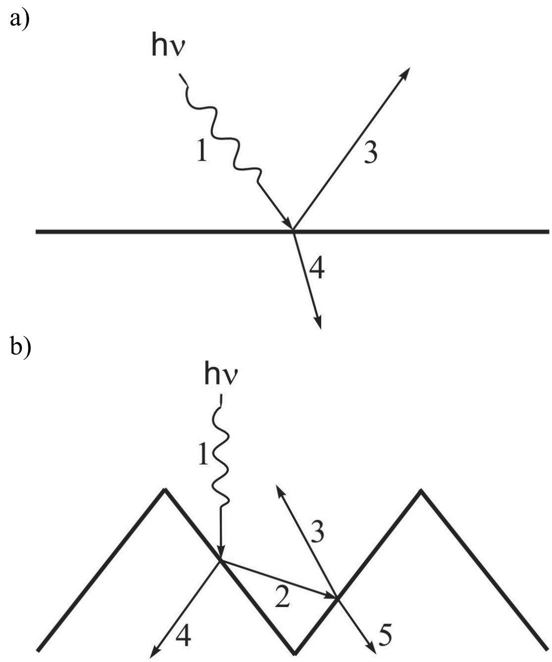


Figure 1. *The impact of surface texturing on radiation absorption: a) flat surface, b) textured surface; where: 1 – incident photon, 2, 3 – reflected photons, 4, 5 – absorbed photons*

A conventional method of surface texturisation commonly used in relation to monocrystalline silicon is anisotropic etching taking place during wet etching in alkali solutions, e.g. KOH or NaOH. A crystal is etched at various speeds according to different crystallographic directions which creates immense opportunities for its spatial shapening (e.g. pyramid structure for (100) orientation) [18, 19]. Etching anisotropy is measured with a relative relationship between (100) plane etching speed and (110) or (111) planes etching speed which for silicon create the following relationships most often [9]:

$$v_{100} > v_{110} > v_{111} \tag{1}$$

$$\frac{v_{100}}{v_{111}} \approx 100 \tag{2}$$

where:

v_{100} – (100) plane etching speed,

v_{110} – (110) plane etching speed,

v_{111} – (111) plane etching speed.

The high selectivity of such etching reagents according to different crystallographic orientations is restraining their use for polycrystalline silicon texturing. In addition, excessive grain faults between grains lead to gaps in metal contacts deposited with the screen printing method.

Other polycrystalline silicon texturisation methods described in the literature are as follows: etching in acidic solutions based on HNO_3:HF, HNO_3:HF:CH_3COOH [20-22], mechanical texturing using a diamond edge [23], reactive ion etching [24]. The surface of polycrystalline silicon can also be formed using a laser beam and this was a subject of own research described herein. The laser radiation properties permitting the precision processing of different materials with efficiency and accuracy significantly surpassing the conventional methods have a major effect on the utilisation of laser processing in various technological operations [25-35].

3. Interdisciplinary research approach

The research presented in this chapter are interdisciplinary. The foresight-materials science research method [15] employed origins directly from organisation and management (technology foresight) as well as materials science (surface engineering). The subject of the comparative analysis are both, the results of studies into the optical properties of polycrystalline silicon and electrical properties of photovoltaic cells made from them as well as a value of the individual technologies determined through expert investigations against the environment and their long-term development prospects together with the recommended management strategies and the forecast multi-variant strategic development tracks. The following selected polycrystalline silicon texturisation technologies have been analysed for the foresight and materials science efforts performed:

(A) polycrystalline silicon alkaline texturisation,

(B) polycrystalline silicon laser texturisation,

(C) polycrystalline silicon laser texturisation with chemical etching.

The materials science experiments were performed on the wafers made of polycrystalline silicon with a boron dopant with the area of 50 x 50 mm and the thickness of ~330 μm of the Bayer company. The wafer shape and dimensions are presented in Fig. 2. The properties of the tested material are given in Table 1.

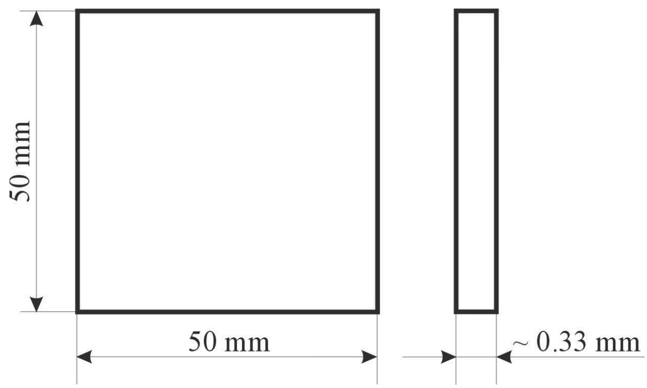


Figure 2. *Silicon wafer shape and dimensions*

Table 1. *Test material*

Basic properties of polycrystalline silicon	
Conductivity	*p*-type
Dopant type	boron
Resistivity	~1 Ωcm
Diffusion length	≥ 80 μm
Boron concentration	$5x10^{16}$ of atoms per cm^3
Oxygen concentration	$5x10^{17}$ of atoms per cm^3
Carbon concentration	$1x10^{18}$ of atoms per cm^3
Concentration of transient metals	10^{11}-10^{15} of atoms per cm^3

3.1. Materials science methodology

Laser silicon surface texturing was carried out with an Allprint DN 50A laser system by Alltec with a laser equipped with a constant active medium – an yttrium-aluminium garnet crystal doped with neodymium ions (Nd:YAG). The laser used is a small-capacity device employed for precision processing in surface engineering. An optoacoustic modulator (Q-switch) is used to produce high-capacity laser pulses in a jogging work mode in the laser system. The surface processing of polycrystalline silicon was undertaken for the following conditions: laser beam output power of 100 %, laser beam release frequency from $f = 15$ kHz, laser beam movement speed $v = 20$ mm/s. A texture was made corresponding to the lattice of grooves with the interspace of 0.09 mm.

Production scheme of polycrystalline silicon solar cells in Fig. 3 is presented. This production process were performed according to the following steps:

- saw damage removal,

- surface texturisation,
- laser induced surface damage removal,
- contamination removal,
- phosphorous diffusion,
- junction insulation and phosphorous-silicate glass removal,
- antireflection coating deposition,
- screen-printing and co-firing of metal contacts.

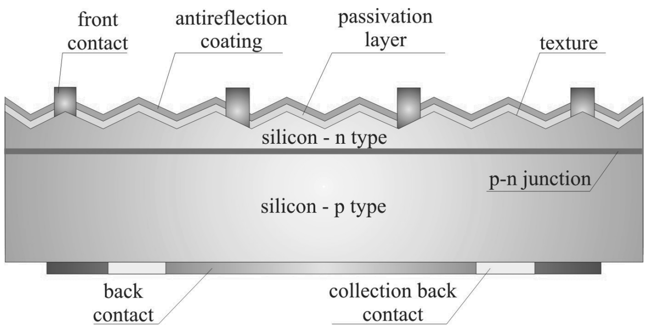


Figure 3. *Production scheme of polycrystalline silicon solar cells*

A silicon surface topography test after laser treatment was performed with a Scanning Electron Microscope (SEM) ZEISS SUPRA 25 and with a Confocal Laser Scanning Microscope (CLSM) 5 Pascal by ZEISS.

The electromagnetic radiation reflection coefficient was measured with a Perkin-Elmer Lambda spectrophotometer for the wavelength of 300 nm to 1300 nm fitted with integrating sphere. The $R(\lambda)$ reflection coefficient values obtained in the measurement were converted into the R_{eff} effective reflection coefficient value according to the following relationship [36,37]:

$$R_{eff} = \frac{\int_{\lambda_{\min}}^{\lambda_{\max}} R(\lambda) N_{ph}(\lambda) d\lambda}{\int_{\lambda_{\min}}^{\lambda_{\max}} N_{ph}(\lambda) d\lambda} \tag{3}$$

where:

$N_{ph}(\lambda)$ – the number of photons falling on an area unit for the specific wavelength in 1 second in AM1,5 conditions.

This coefficient informs whether the curve identifying the reflection coefficient relationship to the tested textured surface according to wavelength matches the solar radiation emission spectrum.

The tests of electrical properties of photovoltaic cells produced using laser textured wafers were made with a computerised SOLAR-LAB station for measuring voltage-current characteristics (I-V) of solar cells for a standard radiation spectrum of AM 1.5 with the radiation concentration of 1000 W/m^2 and the photovoltaic cell temperature of 25°C.

3.2. Foresight methodology

The reference data acquired as a result of implementing foresight research under the project “Foresight of surface properties formation leading technologies of engineering materials and biomaterials. FORSURF” [6] has been used in order to determine the strategic position of laser technologies against materials surface engineering and the laser texturisation of polycrystalline silicon against the surface engineering laser technologies. The research was held according to three iterations of the Delphi method carried out in consistency with the idea of e-foresight [7] using information technology encompassing a virtual organisation, web platform and neural networks. Neural networks were used in a novel and experimental manner to analyse the cross impacts emerging between the analysed trends and the events likely to occur in the future within the considered timeframe. The specific polycrystalline silicon texturisation technologies analysed in this chapter were evaluated based on experts’ opinions using a custom foresight-materials science research methodology [15]. A universal scale of relative states being a single-pole scale without zero was used in the research undertaken, where 1 is a minimum rate and 10 an extraordinarily high rate. The relevant technologies were evaluated for their potential representing a realistic objective value of the particular technology and for their attractiveness reflecting the subjective perception of a specific technology by its potential users. The results were entered into one of the quarters of the **dendrological matrix of technology value** serving to visualise the objectivised values of the specific separated groups of technologies. A wide-stretching oak is the most promising quarter guaranteeing the future success. A soaring cypress characterises highly attractive technologies with a limited potential and a rooted dwarf mountain pine symbolises technologies with a high potential and limited attractiveness likely to ensure a strong technology position if an adequate strategy is employed. The least promising technologies are placed in a quarter of the matrix called a quaking aspen with their future success being unlikely or infeasible. The results of the experts' assessment concerning the influence of the environment on the technologies analysed according to opportunities and difficulties were entered into one of the quarters of the **metrological matrix of environment influence.** Sunny spring illustrates the most favourable external situation ensuring the future success. Rainy autumn gives a chance for steady progress. Hot summer signifies a stormy environment where the success of a technology is risky but feasible. Frosty winter informs that technology development is difficult or infeasible.

The results of the expert investigations visualised with the dendrological and meteorological matrix were next entered with software based on the previously formulated mathematic relationships [15] into the **matrix of strategies for technologies** comprised of sixteen fields. The matrix presents graphically a place of each group of technologies according to its value and environment influence intensity and identifies a recommended action strategy. The strategic development prospects of a given technology expressed in numbers using a universal scale of relative states (min: 1, max: 10) correspond to the relevant areas of the matrix (Fig. 4). The anticipated development of the technologies analysed according to three variants: a positive, most probable and negative variant, was visualised by entering **strategic development tracks** into the matrix of strategies for technologies. The forecast established presents a vision of future events consisting of several variants for a 20-year time horizon for the time intervals of 2015, 2020, 2025 and 2030.

On the basis on the results of foresight-materials science research **technology roadmaps** have been predated. The set-up of the custom technology roadmap corresponds to the first quarter of the Cartesian system of coordinates. Three time intervals for the years of: 2010-11 (current situation), 2020 (goals fulfilment methods), 2030 (long-term objectives) are provided on the axis of abscissa. Seven main layers were applied onto the axis of coordinates of the technology roadmap: time, concept, product, technology, spatial, staff and quantitative ones, made up of more detailed sub-layers. The main technology roadmap layers were hierarchised starting with the top, most general layers determining all-social and economic reasons and causes of the actions taken, through the middle layers characterising a product and its manufacturing technology, to the bottom layers detailing organisational and technical matters concerning the place, contractor and costs. The relationships between the individual layers and sub-layers of the technology roadmap are presented with the different types of arrows representing, respectively, cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow.

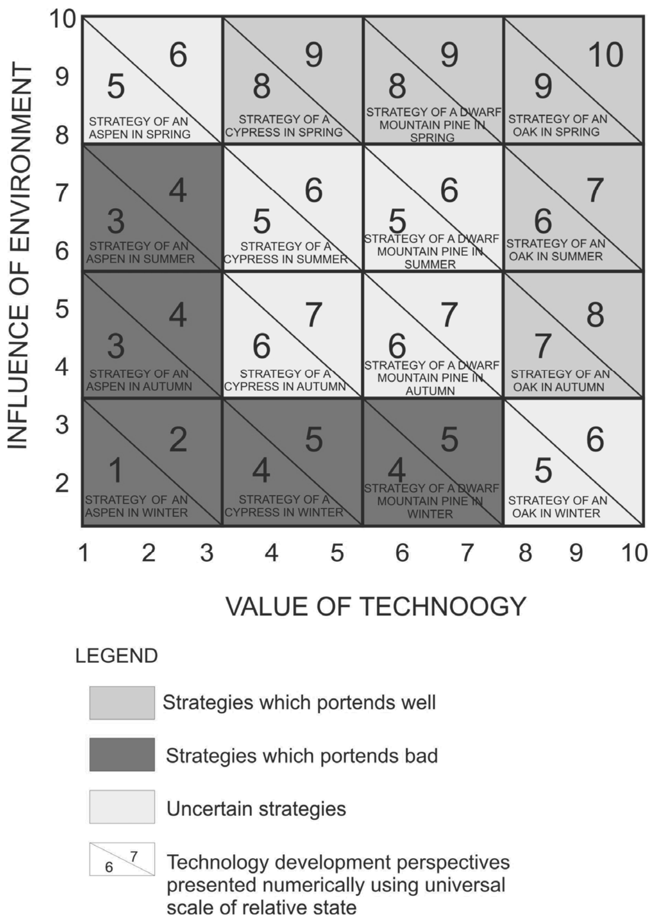


Figure 4. *The framework of the matrix of strategies for technologies with numerically expressed technology development perspectives*

4. Materials science research results

The use of the base 40% KOH : IPA : DIH_2O solution at the temperature of 80°C causes significant differences in the etching speed of polycrystalline silicon grains with a different crystallographic orientation (Fig. 5). This restricts the etching reagent's use for the texturing of polycrystalline silicon where the distribution of crystallographic grains orientation is random. If the alkaline etching time extends, the faults of the textured surface at the grain boundaries are formed [38].

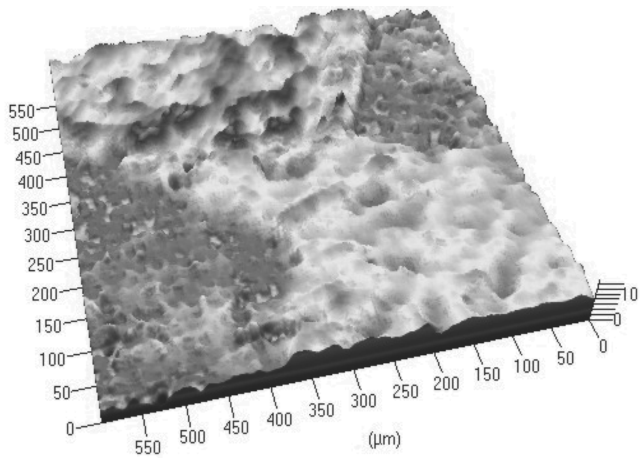
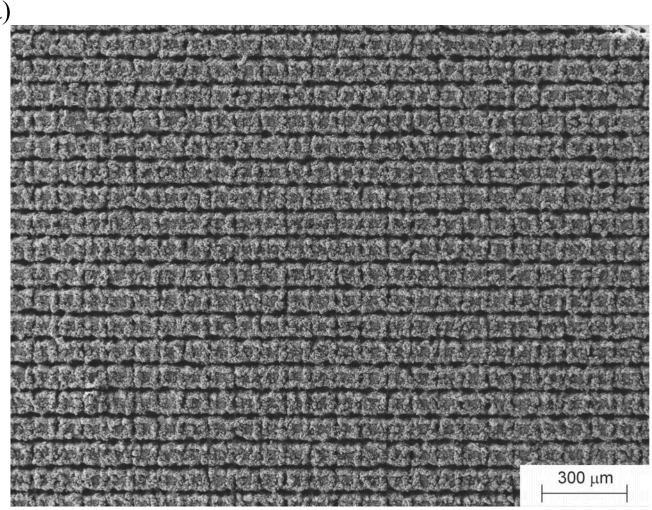


Figure 5. *CLSM three-dimensional topography of the textured wafer surface in 40% KOH:IPA:DIH_2O solution*

It was found by observing the surface topography of wafers with the texture corresponding to the lattice of grooves in a scanning electron microscope that the shape of grooves is irregular with flashes at the peripheries (Fig. 6). The hollows formed are secondary and filled with molten and incompletely evaporated material. The areas between the flashes of the neighbouring grooves are covered with clotted material ejected from the grooves and with products deposited from the gaseous phase released when the material is evaporated outside the groove. Deformed, crystallised silicon beads, so-called inflows, exist at the surface within the groove and flashes having varied dimensions. The clotted beads of the pre-melted material that is substantially ground (diameter of below 0.5 μm) are present at the material surface within the areas between the grooves. Microcracks and microgrooves are present at the surface subjected to texturing, both in the hollows and in flashes.

A laser texture was created by repeating the sequences of parallel grooves in two directions perpendicular to each other. The tracks created in the first place are largely flooded with the molten and incompletely evaporated material, thus they are not visible. The final texture appears only after etching (Fig. 7). Initially, the flashes width and height gradually decreases, then gaps appear in them and then they are completely removed. The side walls and the bottom of the hollow are also etched. Perpendicular grooves appear in the initial phase of etching. Hollows with a higher depth occur where the hollows intersect. Flashes are completely removed during etching and hollows repeating on the whole textured area appear with a regular polyhedral shaped depending on the substrate crystallographic orientation.

a)



b)

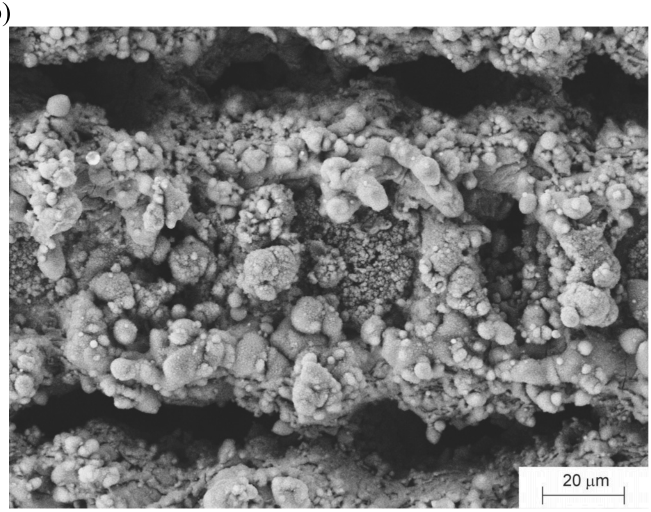
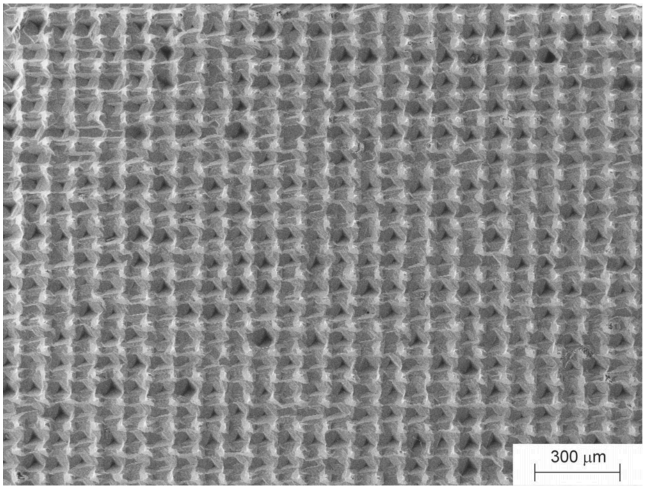


Figure 6. *SEM topography of the laser textured surface of polycrystalline silicon: a) x 150, b) x 2 000*

a)



b)

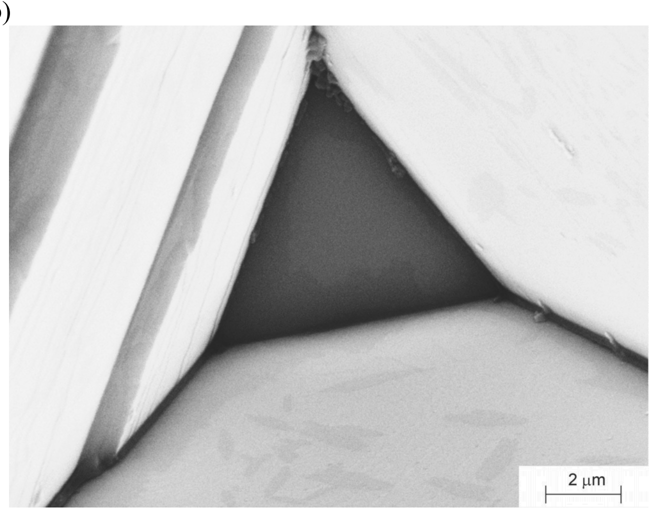


Figure 7. *SEM topography of the laser textured surface after removing 80 μm of the removed layer: a) x 150, b) x 20 000*

The light reflection coefficient was examined for untextured wafers after removing the surface layer damages formed while cutting a silicon block. The results obtained were compared to the light reflection coefficient for a wafer subjected to alkaline texturing in a 40% KOH:IPA:DIH_2O solution. Fig. 8 shows the light reflection coefficient according to the wavelength of the incident radiation for such wafers. Alkaline texturing reduces the light reflection coefficient compared to the wafers subject to no surface treatment. The optical properties of the laser-textured wafers are highly dependable upon the laser treatment conditions. If the surface of wafers is textured corresponding to the lattice of grooves, this causes a decrease in the light reflection coefficient as compared to the coefficient for untextured wafers. As the etching of laser-textured wafers is progressing gradually, the R_{eff} coefficient is clearly growing only after removing 80 μm.

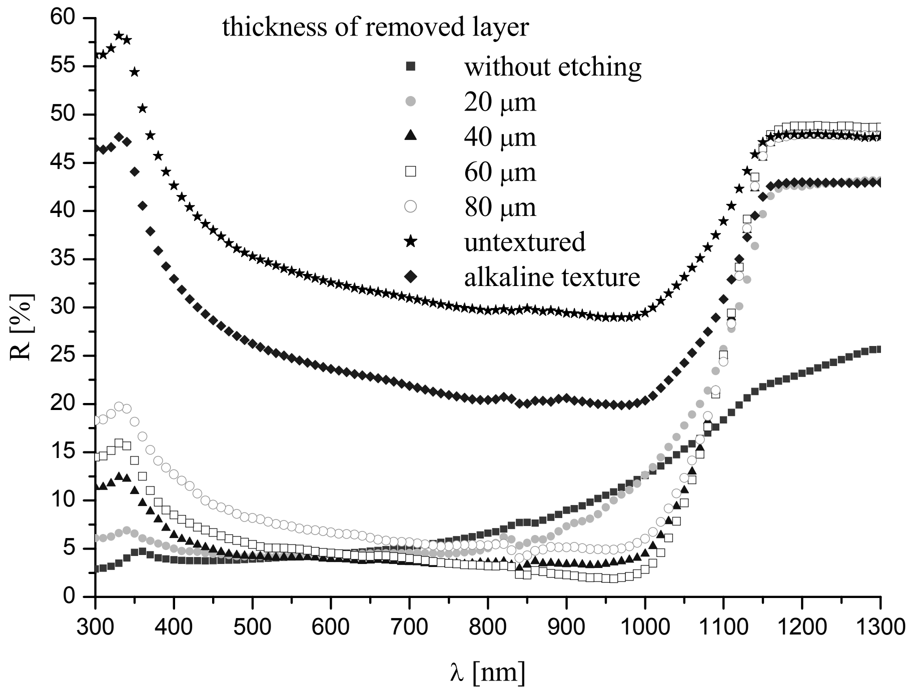


Figure 8. *The light reflection coefficient for the following polycrystalline silicon wafers: untextured, alkaline-textured, unetched laser-textured and laser-textured ones with varied thickness of layers removed during chemical etching*

It was found on the basis of the results of measuring the current-voltage characteristics that the texturing of polycrystalline silicon in an aqueous potassium hydroxide solution improves the electrical properties of the produced photovoltaic cells and enhances efficiency as compared to the cells made of wafers featuring untextured surface (Fig. 9). An increase in such cell's efficiency is negligible, as a texture is produced on the surface as a result of the alkaline etching of polycrystalline silicon in a 40% KOH : IPA : DIH_2O solution being dependent on the crystallographic orientation of the specific grains. Laser polycrystalline silicon surface texturing is deteriorating the electrical properties of photovoltaic cells made of the so prepared wafers. A layer of the damaged material is formed on the entire laser-textured area immediately after creating a lattice of grooves. The layer is produced due to the condensation of the liquid-gaseous phase occurring during laser processing. When the damaged layer of material is removed through etching, the efficiency of photovoltaic cells increases and is largest when a 80 μm thick layer has been etched.

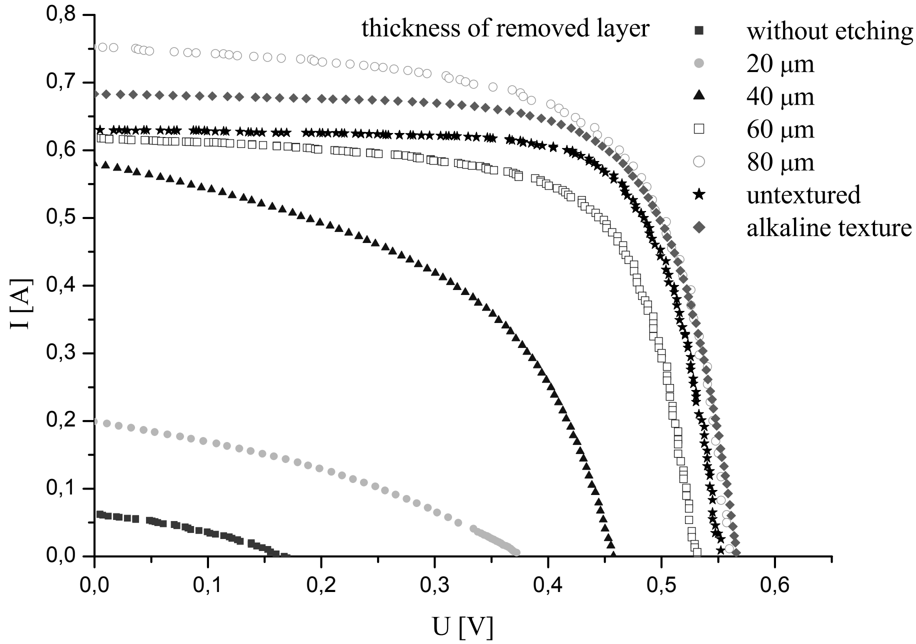


Figure 9. *The voltage-current characteristics of photovoltaic cells made of the following polycrystalline silicon wafers: untextured, alkaline textured, unetched laser-textured and laser-textured ones with varied thickness of layers removed during chemical etching*

Table 2. *The effective reflection coefficient and the efficiency of photovoltaic cells determined for the following polycrystalline silicon wafers: untextured, alkaline-textured, unetched laser-textured and laser-textured ones with varied thickness of layers removed during chemical etching*

Technology symbol	Wafer surface		The R_{eff} effective reflection coefficient, %	Photovoltaic cell efficiency, %
None	Untextured		34.08	10.21
(A)	Alkaline-textured		24.65	10.79
(B)	Unetched laser-textured		10.21	0.14
(C)	Laser-textured and chemical etched; the removed layers with the thickness of, μm	20	12.96	1.05
		40	11.71	5.09
		60	11.79	8.96
		80	13.56	11.01

The detailed values of the effective light reflection coefficient and the efficiency of photovoltaic cells determined for the following wafers: untextured, alkaline-textured, unetched laser-textured and laser-textured ones with varied thickness of layers removed during chemical etching in Table 2 is presented.

5. Polycrystalline silicon texturisation in the future

5.1. Laser treatment versus surface engineering progress

Foresight investigations with the sample size of 198 have revealed a very robust strategic position of laser technologies among other materials surface engineering technologies. The experts found that that laser technologies have the best industrial application prospects in the group of all the analysed materials surface engineering technologies in the nearest 20 years. 78% of the surveyed held such a view. Nearly a three fourth of the respondents (73%) maintain that numerous scientific and research studies will be devoted to such technologies in the analysed time horizon. 70% of the surveyed claim that the thematic area of “Laser technologies in surface engineering” is crucial and its importance should be absolutely rising so that an optimistic scenario can come true of the country's development – "Race won" – assuming that the potential available is adequately utilised to fulfil the strategic objectives of development and so that people, statistically, are better off, social attitudes are optimistic and prospects for the coming years bright. 81% of the surveyed persons argue that the significance of laser technologies in relation to other materials surface engineering technologies will be growing, whereas 18% maintain it will remain on the same level with only 3 individuals asserting that the role will diminish over the next 20 years. The very strong results of technology foresight elaborated with reference data point to the anticipated key role of laser technologies in the development of materials surface engineering in general (mezo scale) and in the development of Poland's overall economy (macro scale).

5.2. Strategic position of the texturisation technologies

The results of the foresight research described in this chapter include the assessment of the potential and attractiveness of the analysed technologies against the micro- and macroenvironment performed based on the key experts' opinions and a recommended strategy of managing a relevant technology together with the predicted strategic development tracks resulting from such assessment.

The analysed polycrystalline silicon texturisation technologies were evaluated by experts using a universal scale of relative states (1: min, 10: max) for their: business, economic, humane, natural and system attractiveness as well as for their creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and a result obtained for the individual groups of technologies was entered into the dendrological matrix of technologies value (Fig. 10). The analysis has revealed that the laser texturisation of polycrystalline silicon with chemical etching (C) is characterised by the highest attractiveness and potential. The technology was classified to the matrix quarter called a wide-stretching oak for highly attractive technologies with a large potential. The silicon wafers produced with the technology feature the lowest (most advantageous) effective reflection coefficient R_{eff}, especially after etching chemically a 40 and 60 μm thick layer (respectively, 11.71 and 11.79%). In addition, the efficiency of solar cells prepared from such wafers had the highest (most advantageous) value of 11.01% after etching an 80 μm thick layer. The (A) technology: the alkali texturisation of polycrystalline silicon was entered into the quarter called a rooted dwarf mountain pine with technologies having a high potential and a low attractiveness. This technology, used successfully for monocrystalline silicon, is ineffective for polycrystalline silicon due to the random crystalline orientation of the individual grains. In relation to the (C) technology, the silicon wafers produced with the (A) technology exhibit much more inferior optical properties (the effective reflection coefficient of R_{eff}: 24.65%), and the solar cells made from them feature worse electrical properties (efficiency of 10.79%). The laser texturisation of polycrystalline silicon (B) is highly attractive considering very promising optical properties expressed with the lowest R_{eff} value (10.21%) for the silicon wafers made with this silicon wafer technique. A relatively low potential of the technology derives from the fact that the laser texturisation of silicon surface is drastically deteriorating the properties of solar cells made of the wafers prepared this way (efficiency of 0.14%). A reason for this phenomenon is a layer of the damaged material formed in the liquid and gaseous phase condensation present during laser treatment. This technology was thus assigned to the matrix quarter called a soaring cypress grouping highly attractive technologies with a limited potential.

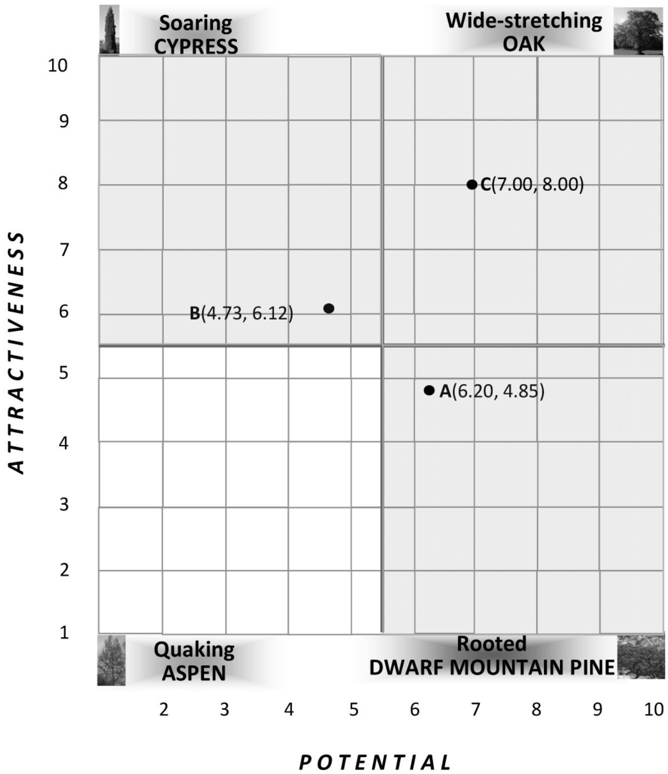


Figure 10. *The dendrological matrix of technology value for prepared for the following polycrystalline silicon texturisation: the (A) alkaline, the (B) laser, the (C) laser with chemical etching ones*

The evaluation results of positive and negative environment influence on the relevant technologies were visualised with a meteorological matrix of environment influence, as illustrated in Fig. 11. The experts surveyed have found that the environment of fresh technologies (B) and (C) is a stormy one considering a very attractive, perspective area of future industrial applications (ample opportunities) and the related fierce global competition and a far-reaching alternative search for effective solar cells manufacturing technologies (numerous difficulties). In case of a mature technology (A) being is industrial use for years, for monocrystalline silicon, an environment is predictable and stable with a neutral character. Polycrystalline silicon produced with alkaline permits to, as compared to its monocrystalline form, produce cheaper, but less effective solar cells. Considering that no specific, clearly better alternatives are at hand, this may suffice for the technology to develop further at a low rate.

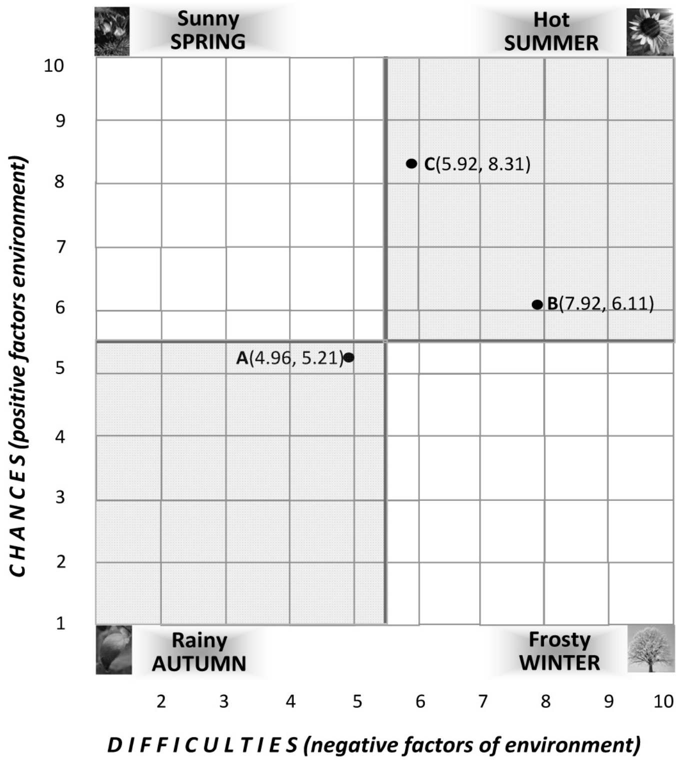


Figure 11. *Meteorological matrix of environment influence prepared for the following polycrystalline silicon texturisation: the (A) alkaline, the (B) laser, the (C) laser with chemical etching ones*

At the next stage of the research work, the results of the studies presented graphically with the dendrological matrix of technologies value and meteorological matrix of environment influence were entered into the matrix of strategies for technologies (Fig. 12). The matrix is presenting, graphically, the place of the investigated polycrystalline silicon texturisation technologies according to their value and environment influence intensity, indicating the relevant managing strategy. Using the pre-defined mathematical relationships, the specific numerical values provided in the following four-field matrices: the dendrological and meteorological matrix, were moved to the sixteen-field technology strategy matrix. The circles mark the strategic development prospects of a given group of technologies expressed in numbers. With reference to the (C) technology valued most highly, that was awarded 7 points in a ten-degree scale, it is recommended to apply an oak in summer strategy. The technology's attractiveness and potential in a risky environment should be used in line with the strategy,

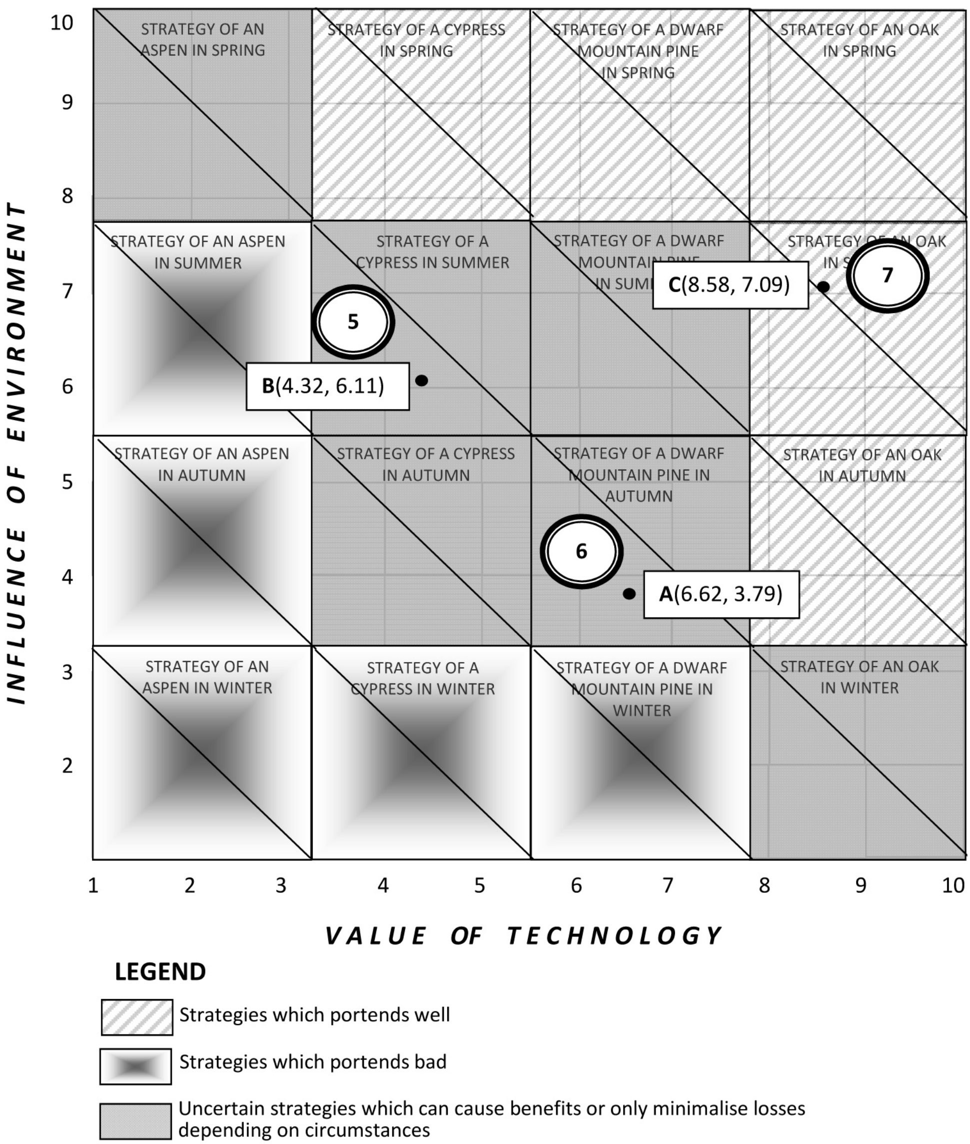


Figure 12. *The matrix of strategies for technologies prepared for prepared for the following polycrystalline silicon texturisation: the (A) alkaline, the (B) laser, the (C) laser with chemical etching ones*

opportunities should be sought for and difficulties should be avoided and the technology should be strongly promoted with publicity measures being preceded with marketing research to tailor a product to the client's demands as far as possible. A strategy of a dwarf mountain pine in autumn recommended for the (A) technology given 6 points assumes that profits are derived from running production in a stable, predictable environment using a solid technology that should be upgraded and intensively promoted to enhance attractiveness. The (B) technology of the laser texturisation of polycrystalline silicon, for which a cypress in summer strategy is recommended, has moderate development prospects (5 points). The technology consists of strengthening an attractive technology's potential in the risky environment conditions and of risk assessment. Either a customer should be fought for aggressively or the technology should be phased out from the market depending on the result of such evaluation.

Strategic development tracks for the individual technology groups were prepared based on the expert opinions in the next part of the research works according to the three variants: optimistic, most probable and pessimistic variant for the relevant time intervals of: 2015, 2020, 2025 and 2030.

The most encouraging (C) technology is the polycrystalline silicon laser texturisation with chemical etching. The excellent optical properties of silicon wafers produced as well as good properties of the photovoltaic cells made of them are ensured by this technology. The most probable track of (C) technology development assumes that its potential is to be strengthened in 2015-2020 and environment conditions bettered in the subsequent years (2025-2030) thus moving the (C) technology to the most auspicious matrix quarter: oak in spring. The greatest hopes are connected with shortening the activity of laser impulse to nano- (10^{-9}), pico- (10^{-12}) or even femto- (10^{-15}) seconds. The experiments undertaken have revealed that the shorter impulse activity time the smaller substrate material damage. It seems for the time being that although a laser acts for a very short time, the damage to the top layer of polycrystalline silicon will compromise the electrical properties of the solar cells prepared from it to such an extent that even short chemical etching will be necessary to improve such properties. An optimistic (C) technology development track envisages that the opportunities derived from the environment will rapidly exceed the related difficulties and already in 2020 this technology will be found among those having the best prospects best and fast progress will be maintained. Since an incipient the (C) technology is in a stormy environment, an adverse surprising scenario is also possible. According to such scenario, the value of the technology analysed including its potential and attractiveness will be declining gradually (2015-20) as the increasing predominance of one of the alternative technologies will be seen (etching in acidic solutions, reactive ion etching, mechanical texturisation using a diamond edge). External development prospects will be thus completely limited in 2025 (oak in winter strategy) and the technology will be ultimately forced out from the market in 2030 (aspen in winter strategy). The outcomes of the research pursued with the Delphic method confirm a good, anticipated, strategic position of the laser texturisation of polycrystalline silicon with chemical etching [6]. 73% of the experts surveyed think that the technology is critical and its importance should be absolutely rising so that an optimistic scenario of the country's development, i.e. "Race Won" can come true in the nearest 20 years.

In congruence with the most probable scenario, the potential and attractiveness of the (A) technology of polycrystalline silicon alkaline texturisation should be slowly strengthening and should be maintained for the duration of the forecast (2015-2030) in area of the dwarf mountain pine in autumn. An optimistic scenario provides for that the alternative methods of producing solar cells from polycrystalline silicon will suffer a defeat if the parameters of the cells' alkaline texturisation are fine-tuned optimally. As a result, in 2030 the technology will be transferred to the "oak in autumn" field. A pessimistic scenario of technology development (A) envisages that alternative polycrystalline silicon texturisation technologies ensuring both, better optical properties of silicon wafers and electrical properties of solar cells made from them, are developing rapidly and robustly. This will contribute to shifting the (A) technology in 2020 to the area of unfavourable influence of a dwarf mountain pine in winter and progressing degradation in 2025-2030 (aspen in winter).

The most probable scenario of the (B) technology's development: laser texturisation of polycrystalline silicon assumes that its potential will be growing slowly with the neutral environment conditions maintained (2015-20). Next, its value against other alternative technologies will diminish in 2025-30, hence it will be shifted to the "dwarf mountain pine in autumn" field. An optimistic scenario of technology development (B) assumes that its potential will be reinforced substantially while the existing attractiveness is maintained. This may, unexpectedly, improve the optical properties of silicon wafers and the electrical properties of cells prepared from them. The experiments carried out may indicate with little probability that a very short laser impulse (of pico- or femtoseconds) may cause such a little damage to the substrate material that its chemical etching will not be necessary to improve the electrical properties of cells. A pessimistic (B) technology scenario assumes that it has been surely proved that, to ensure the expected electrical properties of solar cells made from the laser-textured silicon wafers, it is necessary to etch them chemically to remove damages formed in the condensation of the liquid and gaseous phase during laser treatment. The years of 2015-2020 are witnessing a slow decline in the technology value coupled with the increasingly more difficult environment conditions with the technology being completely phased out from the market in 2025-2030 (aspen in winter). A graphical example of the (B) technology strategy matrix with the strategic development tracks entered according to three variants is presented in Fig. 13. The numerical values being a result of all the investigations performed for the three analysed technologies are shown in Table 3.

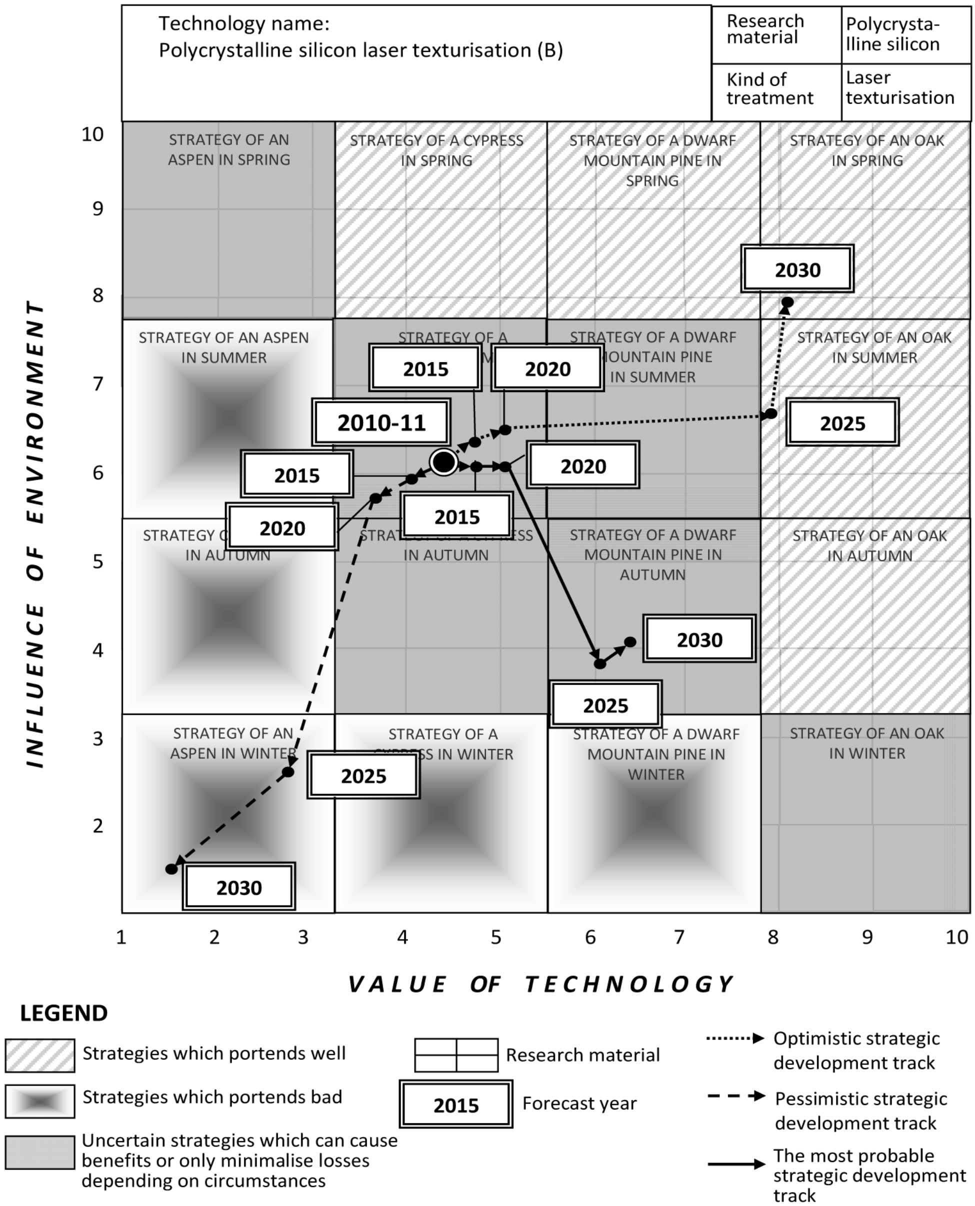


Figure 13. *The strategic development tracks created for the (B) demonstration technology: polycrystalline silicon laser texturisation*

Table 3. *The strategic development tracks of polycrystalline silicon texturisation. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic, (MP) – the most probable*

Technology symbol	Technology name	Steady state 2010-11	Type of strategic development tracks	Years			
				2015	2020	2025	2030
(A)	Polycrystalline silicon alkaline texturisation	Strategy of a dwarf mountain pine in autumn A (6.6, 3.8)	(O)	(7.0, 4.0)	(7.4, 4.1)	(7.7, 4.3)	(8.2, 4.5)
			(P)	(6.2, 3.6)	(5.8, 3.4)	(2.6, 2.4)	(1.4, 1.8)
			(MP)	(6.8, 3.9)	(7.0, 4.0)	(7.2, 4.1)	(7.4, 4.3)
(B)	Polycrystalline silicon laser texturisation	Strategy of a cypress in summer B (4.3, 6.1)	(O)	(4.7, 6.4)	(5.1, 6.5)	(7.9, 6.7)	(8.1, 7.9)
			(P)	(4.1, 5.9)	(3.7, 5.7)	(2.8, 3.6)	(1.4, 1.4)
			(MP)	(4.7, 6.1)	(5.1, 6.1)	(6.0, 3.8)	(6.4, 4.1)
(C)	Polycrystalline silicon laser texturisation with chemical etching	Strategy of an oak in summer C (8.6, 7.1)	(O)	(8.8, 7.7)	(9.0, 8.1)	(9.2, 8.5)	(9.4, 8.9)
			(P)	(8.5, 6.7)	(8.3, 6.3)	(8.1, 2.4)	(2.7, 2.1)
			(MP)	(8.6, 7.5)	(8.7, 7.7)	(8.7, 8.0)	(8.8, 8.5)

The results of the foresight–materials science research conducted represent reference data serving to create a technology roadmap for each of the three technologies analysed. An example of a technology map was prepared for the (C) technology: polycrystalline silicon laser texturisation with chemical etching, presented in Fig. 14.

6. Summary

Economic reasons underlie an interest in polycrystalline silicon texturisation technologies. Silicon occurs in the earth crust in abundance primarily as silica and this is a reason why it is purposeful to search its wide-scale industrial applications. In the wake of the growing energy demand, depleting conventional energy sources, controversies surrounding the security of nuclear fuel utilisation and the society's growing environmental awareness, a feasible use of silicon in the processes of converting solar radiation energy into electric energy paves a way for its extensive utilisation in the future. The production of solar cells from silicon is preceded with a crystal-lisation process as a result of which two forms are produced: mono- and polycrystalline ones.

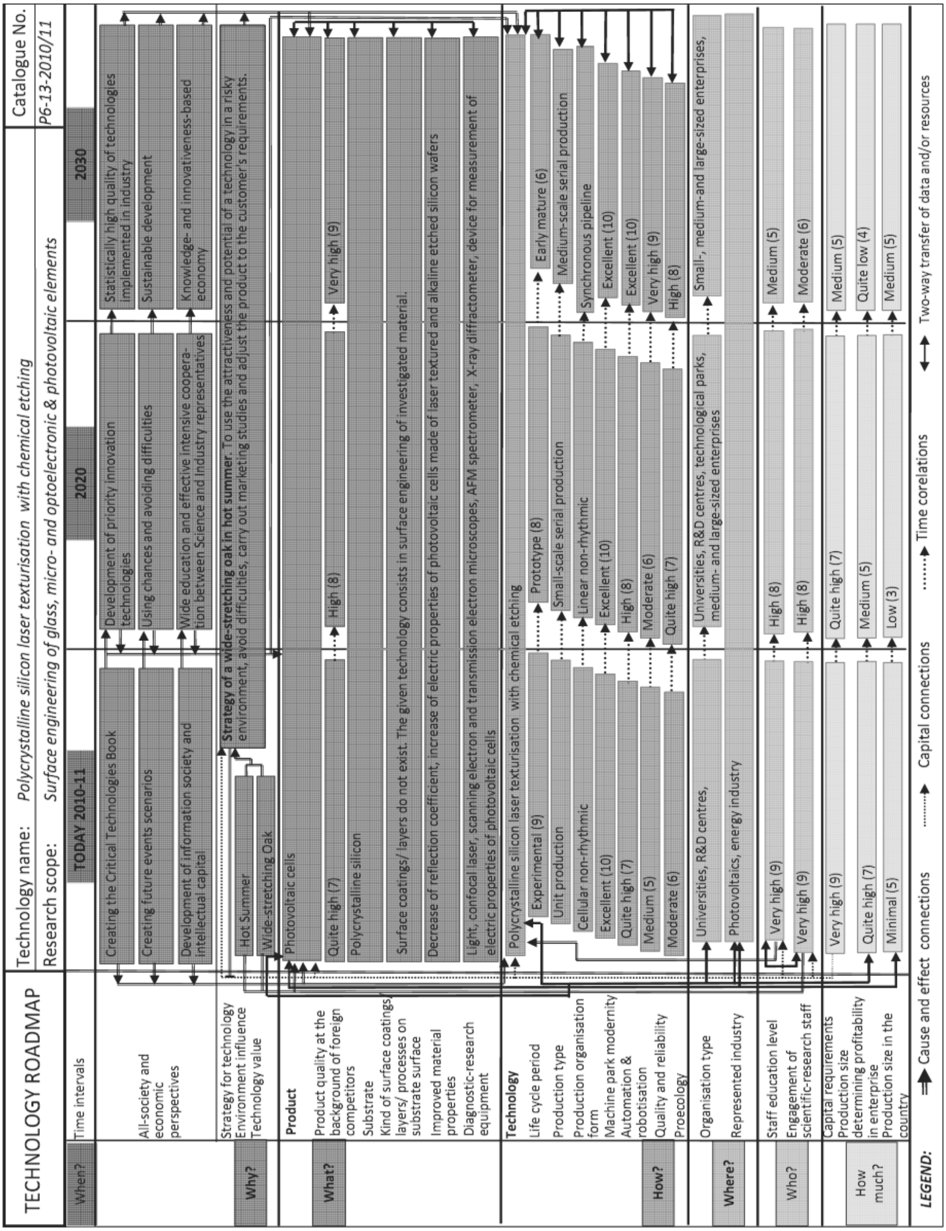


Figure 14. *An example technology roadmap made for the (C) technology: polycrystalline silicon laser texturisation with chemical etching*

It is more expensive to produce monocrystalline silicon having grains with a uniform crystallographic orientation; however, the solar cells produced are highly efficient. The crystallisation process of polycrystalline silicon with a random crystallographic orientation of grains is faster and cheaper, however, the solar cells produced from them feature lower efficiency than the cells produced of its monocrystalline form due to structural defects present. It is justified, therefore, to seek new cells manufacturing technologies from polycrystalline silicon ensuring higher efficiency at relatively low costs. The outcome of such quests is the (C) technology of polycrystalline silicon laser texturisation with chemical etching (C) described in this study. This highly attractive technology with a large potential has been valued most highly in the group of three polycrystalline silicon texturisation technologies subjected to a comparative analysis in this chapter. Better optical properties for silicon and better electrical properties for the cells prepared from them have been attained as compared to the (A) technology: polycrystalline silicon alkaline texturisation. The (B) technology: although the polycrystalline silicon laser texturisation without chemical etching allows to obtain the lowest (most beneficial) effective reflection coefficient, nonetheless, the efficiency of the so produced solar cells is sharply falling. Taking into account the strategic development of the (C) technology, a stormy environment where it is situated is the biggest issue. The environment offers multiple opportunities coming from a very attractive, prospective area of future industrial applications as well as multiple inconveniences connected with intense global competition and broad alternative quests for effective solar production technologies such as: etching in acidic solutions, reactive ion etching, mechanical texturisation and with a use of a diamond edge. The results of foresight-materials science research presented in this chapter are part of a broader project [39] aimed at selecting the priority innovative technologies of materials surface engineering and setting the directions of strategic development, as discussed in a series of publications, in particular [40-44].

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11 Manufacturing technologies of sintered graded tool materials evaluated according to foresight methodology

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Abstract

Purpose: *The goal of this chapter is to evaluate the development efficiency of conventional technologies of powder metallurgy used for graded tool materials manufacturing. The technologies were divided into three groups according to the matrix type and the percent fraction volume of components in the powders layers.*

Design/methodology/approach: *In the framework of foresight-materials science research a foresight matrices set was created, materials science experiments using light, transmission and scanning electron microscopes, X-ray diffractometer, microhardness tester, work-stands for testing of fatigue resistance, mechanical fatigue strength, fracture toughness were conducted and technology roadmaps were prepared.*

Findings: *Quite high potential and attractiveness of the analysed technologies against the environment, as well as good development perspectives in industry were shown.*

Research limitations/implications: *Research concerning graded tool materials constitute a part of a larger research project aimed at identifying, researching, and characterising the priority innovative technologies in the field of materials surface engineering.*

Practical implications: *The presented materials science results prove a manufacturing possibility of elements with ductile cores and hard coatings using conventional technologies of powder metallurgy. These technologies are recommended for practical implementation in industry, especially for cutting tools.*

Originality/value: *The originality of this chapter the value evaluation of manufacturing technologies of graded tool materials against background environment including the influence of the chemical composition and sintering conditions on the surface layers hardness.*

Keywords: *Manufacturing and processing; Powder metallurgy; Graded tool materials; Foresight; Technology Roadmapping*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, A. Kloc-Ptaszna, B. Dołżańska, Manufacturing technologies of sintered graded tool materials evaluated according to foresight methodology, Archives of Materials Science and Engineering 50/2 (2011) 69-96.

1. Introduction

In line with the latest European development trends in the current, hard economic situation and the accompanying issues concerning the climate change, increased consumption and depletion of conventional energy sources, food safety, healthcare and the advancing ageing of the society, innovation understood as valuable, innovative ideas should be the way to achieve the set strategic objectives and represent the source of economic growth. Having this concept in mind, the European Commission has prepared a draft on the establishment of the Innovation Union [1], by focussing on the innovations that should substantially contribute to facing the key challenges named in the valid Europe 2020 strategy. The strategy provides that the level of R&D and innovation investments until 2020 is to reach aggregately 3% of the EU's GDP from public and private funds. For the economic and social effects achieved to be satisfactory, the stream of investments has to be channelled into those fields of science and industries bringing the highest added value, with special consideration given to the role of small- and medium-sized enterprises representing 99.8% of all the Polish enterprises generating 68% of the GDP. The aim of the foresight studies conducted widely in Europe and Poland in the recent decade, also for materials science [2-5], is a quest for innovative areas deserving financial support. Technology foresight serving to identify the priority, innovative technologies and their strategic development directions, has been undertaken for materials surface engineering, as well [6, 7]. Surface engineering of tool materials represents one of the 14 thematic areas analysed for these foresight studies. Of 10 critical technologies having the best development prospects and/or being of key significance for the industry selected for thorough research carried out with the Delphi method, the powder metallurgy methods were found allowing to achieve changes to chemical composition and/or phase composition in the surface layer.

Graded tool materials, characterised by their chemical composition, phase composition and the structure or arrangement of atoms varying according to the location, can be produced in laser treatment (remelting and alloying) [8-11], Physical Vapour Deposition (PVD) [12-18], hybrid (PVD and thermochemical treatment) [19-22] and sintering processes with the conventional powder metallurgy methods [23-27]. This chapter discusses the last of the mentioned groups of manufacturing the graded tool materials. The production of graded tool materials in the conventional powder metallurgy method consists of filling a die with the subsequent layers of powder mixtures differing in their chemical and/or phase composition according to the required properties of the material produced with the mixtures being then pressed and sintered [28-31]. The output composition of the moulded piece can, therefore, be controlled in order to produce sinter with its chemical and/or phase composition varying according to the material volume and position. Such materials are called Functionally Graded Materials (FGM) [32-35]. If the powder metallurgy method is applied, high resistance to abrasive wear, distinctive for cemented carbides and cermets, can be accomplished for the surface layer while maintaining high core ductility typical for high-speed steels [24-27, 36] and traditional carbide steels [37-40] at a relatively low cost. Such structure of the material allows to develop its properties freely depending on the working conditions of the tool. A layer of hard material is used for example in those locations at the tool being exposed to wear while high ductility is ensured in those locations of the tool material that are susceptible to impact.

The purpose of this study is a comparative analysis of three specific technologies of producing the following graded tool materials with the conventional powder metallurgy method: GM-90HSS/10WC, GM-75HSS/25WC, GM-3Co/97WC. Categorisation is done according to the criterion of the matrix material and the fraction of powder in the mixture. The subject of the comparative analysis are both, the outcomes of investigations into the structure and properties of the analysed materials as well as the value of the individual technologies against the environment and their long-term development prospects together with the recommended management strategies and forecast multi-variant development tracks with this value being determined through expert studies according to the custom methodology [41]. The relevance and adequacy of the assessments performed is ensured by the synergic interaction and cross supplementation of the foresight-materials science research. The chapter also presents the outcomes of e-foresight research (Fig. 1), based on row data [6] pertaining to the position of tool materials surface engineering, including the powder metallurgy methods, permitting to change chemical and/or phase composition in the surface layer, vis-à-vis other surface engineering technologies. Technology roadmaps being a comparative analysis tool especially helpful for the small- and medium-sized enterprises lacking funds for conducting own research in this domain have been established at the last stage of the efforts. The results of the foresight and materials science research presented in this chapter are part of a broader project [7] aimed at selecting the priority innovative technologies of materials surface engineering and setting their directions of strategic development, as discussed in a series of publications, *inter alia* [42-47].

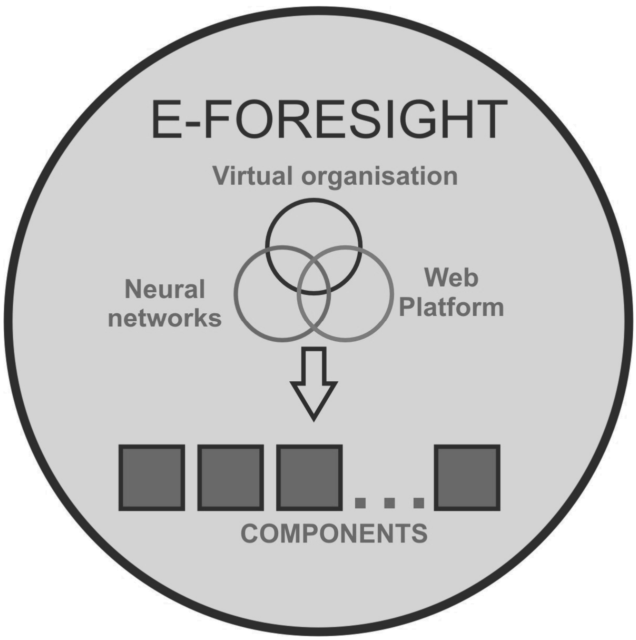

Figure 1. *E-foresight research*

2. Subject matter of interdisciplinary research

The research pursuits concentrated on graded tool materials, differing in the matrix material and a volume fraction of components in the individual layers of powders, are of an interdisciplinary character. The research methodology applied concerns primarily technology foresight (Fig. 1) being part of the field of science referred to as organisation and management and of surface engineering comprised in the widely-understood materials science. A much broader insight into the concept was, however, required at some stages of the research, hence the following areas of detailed knowledge were also derived from: information science with information technology and artificial intelligence [48, 49] (neural networks, Monte Carlo method); statistics; econometrics; operational studies; construction and operation of machines; automation and robotisation of industrial processes; strategic, tactical and operational management; quality and environment management; accounting and finance.

The conventional powder metallurgy method has been used for producing the materials. The method consists of the uniaxial pressing of powder in a closed matrix and then sintering it. The graded materials were produced by filling the matrix with the subsequent layers of powders mixtures and next by pressing and sintering the layers. The following three homogenous specific technologies were distinguished between for the purpose of the foresight and materials science works carried out under this study by adopting, as a criterion of grouping, the matrix material and the volume fraction of components in the individual layers of powders:

(A) Manufacturing of the GM-90HSS/10WC graded tool materials based on the high-speed steel matrix with 10% volume fraction of the tungsten carbide reinforcing phase in the surface layer,
(B) Manufacturing of the GM-75HSS/25WC graded tool materials based on the high-speed steel matrix with 25% volume fraction of the tungsten carbide reinforcing phase in the surface layer,
(C) Manufacturing of the GM-3Co/97WC sintered graded carbide steels based on the cobalt matrix with 97% volume fraction of the tungsten carbide reinforcing phase in the surface layer.

2.1. Test materials

The research has been carried out with specimens manufactured with a conventional powder metallurgy method. The powders of HS6-5-2 high-speed steels (Fig. 2), tungsten carbide (Fig. 3) and cobalt (Fig. 4) have been used for the research. The chemical composition and the basic properties of powders are juxtaposed in Table 1.

Table 1. *Chemical composition and properties of HS6-5-3, WC and Co powders*

Element	Mass concentration, %		
	HS6-5-2	WC	Co
C	0.75-0.90	6.11	0.02
Mn	0.20-0.45	-	< 0.001
Si	≤ 0.45	≤ 0.002	< 0.002
P	≤ 0.04	-	-
S	≤ 0.04	0.003	< 0.002
Cr	3.75-4.5	-	-
Ni	0.2	-	< 0.002
Mo	4.5-5.5	≤ 0.001	-
W	5.50-6.75	rest	-
V	1.6-2.2	0.19	-
Co	0.1	-	rest
Cu	0.1	-	< 0.002
Fe	rest	0.003	-
Ca	-	0.003	< 0.001
Al	-	≤ 0.002	-
Mg	-	≤ 0.001	-
K	-	≤ 0.001	-
Na	-	≤ 0.001	-
C free	-	0.02	0.02
Grain size, µm	> 150	> 0.86	> 6
Additional information	High-speed steel powder, atomised with water by Hoeganaes	Tungsten carbide powder by Unicore	Cobalt powder by Unicore

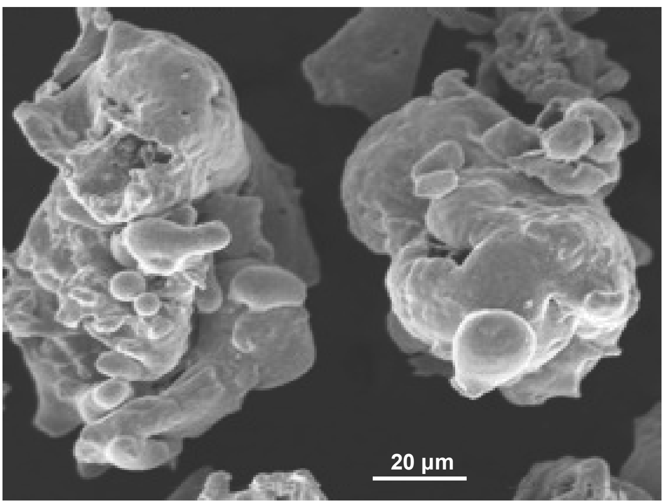

Figure 2. *A scanning electron micrograph of HS6-5-2 water atomised powders*

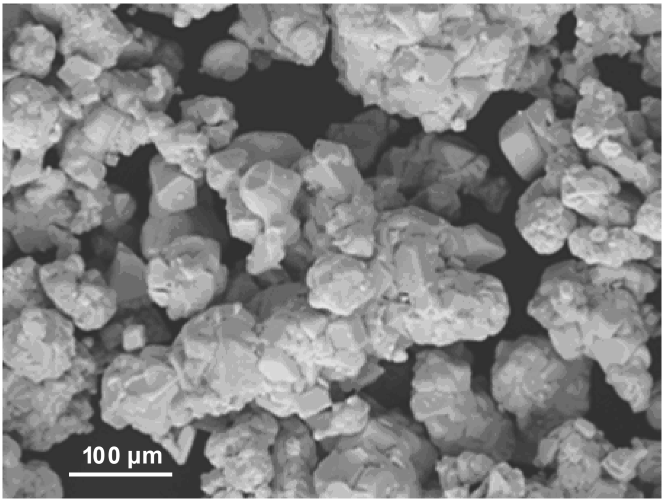

Figure 3. *A scanning electron micrograph of WC powders*

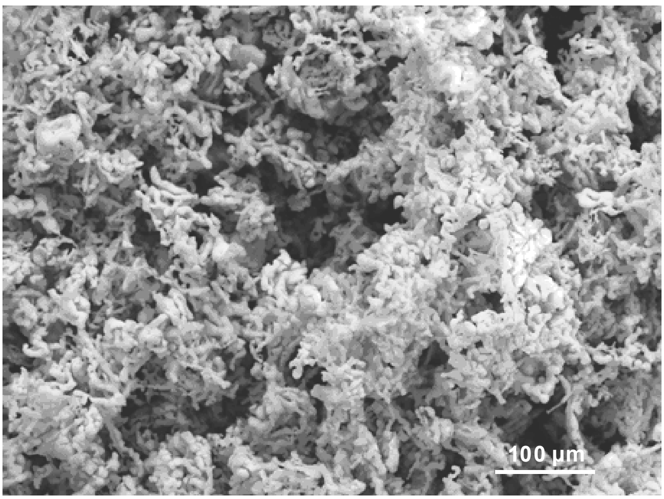

Figure 4. *A scanning electron micrograph of Co powders*

According to the (A) and (B) technologies four-layer specimens were made of HS6-5-2 high-speed steel powder and WC powder. These specimens were made where the subsequent transitory layers with a smaller and smaller volume fraction of tungsten carbide were established from the surface layer side until a surface layer was produced containing high-speed steel only. An assumption was made that such combination of layers can correspond to the material used for producing tools for cutting edges. The powders were mixed for an hour in a T2F WAB-TURBULA mixer. The powder mixtures were next filled to the matrix thus producing layers with the gradually changing volume fraction of carbides in high-speed steel. The next transitory layers were established according to the (A) technology for the volume fraction in the surface layer of 10% of WC containing, respectively, 7 and 4% of such carbides. In the case of the (B) technology, where the surface layer containing 25% of the WC fraction volume, the transitory layers contained respectively, 15 and 5% of WC. The specimens were pressed under the pressure of 500 MPa. The sintering conditions were selected experimentally by changing a temperature, duration and atmosphere of mouldings sintering. The specimens were sintered in a vacuum furnace and with the atmosphere of flowing nitrogen with the addition of hydrogen (N_2+5% H_2), at a temperature of 1210, 1230, 1250 and 1270°C, for 30 and 60 minutes.

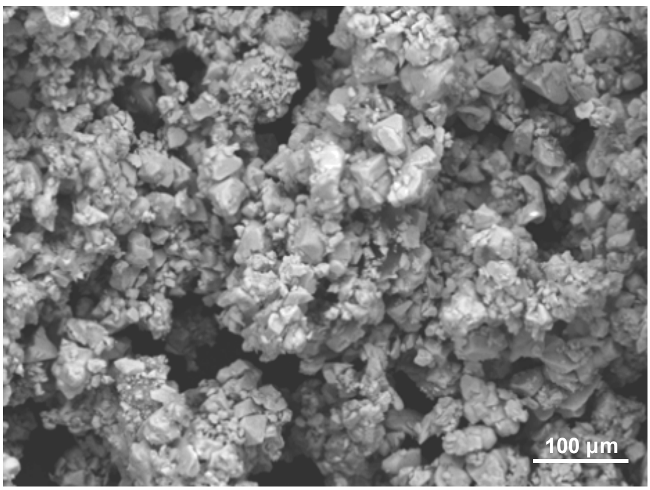

Figure 5. *Mixture of WC (93%) and Co (7%) powder after 8 hours of crushing in a ball mill*

According to the (C) technology four-layer specimens were made of tungsten carbide powders (WC) and cobalt powder. These specimens were made with the individual transitory layers being established in the surface layer side with an increasingly higher volume fraction of cobalt carbide until a layer has been produced containing 9% of Co and 91% of WC. An assumption was made that such combination of layers can correspond to the material designed for producing tools for cutting edges. Co and WC powders were crushed in a ball mill with carbide balls for 8 hours (Fig. 5). The powder mixtures obtained were next filled to the matrix thus producing layers with the gradually changing volumetric concentration of cobalt and tungsten carbide fraction. The subsequent four transitory layers were established in the surface layer of the material with the volume concentration of 3% of Co and 97% of WC with a 2% increase of cobalt concentration until a surface layer was produced containing 9% of Co and 91% of WC. The mouldings were produced as a result of pressing under the pressure of 340 MPa. The sintering conditions were selected experimentally by changing a temperature, duration and atmosphere of mouldings sintering [50,51]. The specimens were sintered in a vacuum furnace, freely at a temperature of 1460ºC, for 30 minutes and with isostatic pressing at 1425ºC, for 90 min.

Table 2 shows the detailed marking of the specimens and their layers with the volume fraction of mixture components provided for the individual layers.

Table 2. *The detailed marking of the GM-HSS/WC and GM-Co/WC specimens and their layers with fraction volume of components in the layers*

Specimen marking*)	Layer marking*)	Layer number	Layer type	Fraction volume of components in layers, %			
					Powder		Powder
GM-90HSS/10WC	GM-90HSS/10WC	1	Surface layer	90	HS6-5-2	10	WC
	GM-93HSS/7WC	2	Intermediate layers	93		7	
	GM-96HSS/4WC	3		96		4	
	GM-100HSS	4	Substrate	100		0	
GM-75HSS/25WC	GM-75HSS/25WC	1	Surface layer	75		25	
	GM-85HSS/15WC	2	Intermediate layers	85		15	
	GM-95HSS/5WC	3		95		5	
	GM-100HSS	4	Substrate	100		0	
GM-3Co/97WC	GM-3Co/97WC	1	Surface layer	3	Co	97	
	GM-5Co/95WC	2	Intermediate layers	5		95	
	GM-7Co/93WC	3		7		93	
	GM-9Co/91WC	4	Substrate	9		91	

*) GM – graded material; HSS – high-speed steels; WC – tungsten carbide; Co – cobalt

2.2. Materials science methodology

In the framework of this chapter have been carried out research of structure as well as mechanical and physical properties of GM-HSS/WC and GM-Co/WC manufactured by the conventional powder metallurgy method.

Metallographic research has been performed on the fractures of the sintered and heat treated specimens. The specimens were cut in a plane perpendicular to the surface layer. Metallographic research has been conducted with light microscopes (MEF4A by Leica and Axiovert by OPTON) with the magnification of 100 to 1000x and with scanning electron microscopes (XL30 by Philips Company and Supra 35 by Zeiss) fitted with secondary electrons detectors (SE) and back-scattered electrons detectors (BSE or QBSD) with the accelerating voltage of 5 to 20 kV, with the magnification of 50 to 10000 times. The primary austenite grain size was measured with the Snyder-Graff method. The fraction volume of carbides was determined with the methods of quantitative metallography using Image-Pro Plus computer aided image analysis software.

The quantitative and qualitative X-ray microanalysis and the surface distribution analysis of alloy elements was performed on the ground and polished fractures from the surface layers of gradient sintered carbide steels in a scanning electron microscope (XL30 by Philips Company), at the accelerating voltage of 20 kV, equipped additionally with an X-ray radiation analyser with energy dispersion (EDAX D4) by Philips Company. The chemical composition (mass concentration and atomic concentration of metallic elements) was determined with an X-ray microanalysis in the selected micro-areas of the matrix and carbides on the cross-section of the investigated graded tool materials.

The phase composition of powders, mouldings and graded materials in the sintered and heat-treated condition was evaluated with an X-ray X'PertPRO diffractometer by PANalytical using filtered Kα radiation of a cobalt lamp with the rated voltage of 40 kV and the filament current of 30 mA. Deflected radiation concentration measurements were carried out within the 2Θ angular range of 30 to 120° with a 0.05° step and with the counting time of 10 seconds.

The fraction volume of residual austenite was calculated using the software developed at the Division of Materials Processing Technologies and Computer Techniques in Materials Science. The software enables fraction volume calculations for residual austenite in steels with an X-ray Averbach-Cohen method based on the results of measurements of total concentrations of diffraction maximums for X-ray radiation from the lattice planes of γ and α phase.

Diffraction investigations and the investigations of thin foil structures from the selected locations of the graded material specimens in the sintered and heat-treated condition were undertaken with a Transmission Electron Microscope (TEM) JEM 3010UHR transmission electron microscope by JEOL, with the accelerating voltage of 300 kV. Diffraction patterns from the TEM were solved using Eldyf software.

The proper (actual) density of high-speed steel and WC powders was measured automatically with a gas AccuPyc 1330 pycnometer by Micromeritics. The measurements were made in a helium atmosphere. The density, porosity and hardness was measured for the sintered specimens with the Vickers method and the Rockwell method at the A scale. A densitometric method was used for measuring the density of sinters. The method consists in measuring the apparent loss of the specimens mass when submerged in water. A hardness measurement was performed with the Vickers method with the indenter load of 4.903 N. The working time of the indenter total loading force was 15 seconds. The measurement was made across the entire width of the sintered specimens cross-section by starting the measurement within 0.2 mm from the outer surface layer and ending in the surface layer area (about four measuring points were used for each layer). Hardness testing with the Rockwell method at the A scale was made at the faces of the surface layers of the sintered gradient carbide steels. The measurement was made in 10 randomly points selected from the surface layers areas. Stereological tests using an Axiowert 405M optical microscope fitted with computer aided image analysis software were made to identify porosity. The tests were made at the fractures using Image-ProPlus computer-assisted image analysis software. The surface fraction of pores was determined − expressed as a ratio of the pores area to the total area of the analysed area. Five random points from the areas of the individual layers were chosen each time for measurement. Hardness tests with the Rockwell method at the C scale were made at the faces of the surface layers of graded materials on a heat-treated high-speed steel matrix. A measurement was made for each heat treatment variant in the randomly picked points from the area of surface layers and substrate (10 measurements for each layer). The results of density, porosity and hardness tests were developed statistically by calculating, for each series of measurements, the average value, standard deviation and average value confidence interval for the confidence level of $\alpha = 0.05$. A regression function was also determined approximating the relationship between the investigated output variable (e.g. material hardness or density) and the input variables (e.g. volume fraction of components, production or thermal treatment process conditions).

The tests of abrasive wear resistance for GM-3Co/97WC were carried out using the device shown in Fig. 6. Tests with a counter-specimen, i.e. an Al_2O_3 ceramic ball were made on the prepared specimens. The tests were made using a varied number of 1000 cycles and at a varied load of 2.5 and 10 N. Four results were obtained for the surface layers of each tested specimen due to such applied tests conditions and this allowed to define an abrasive wear measurement. The degree of wear was determined through the geometric measurements of wear and by calculating the wear volume. Wear observations were also made with a Confocal Laser Scanning Microscope (CLSM) 5 Exciter confocal microscope and in an Scanning Electron Microscope (SEM).

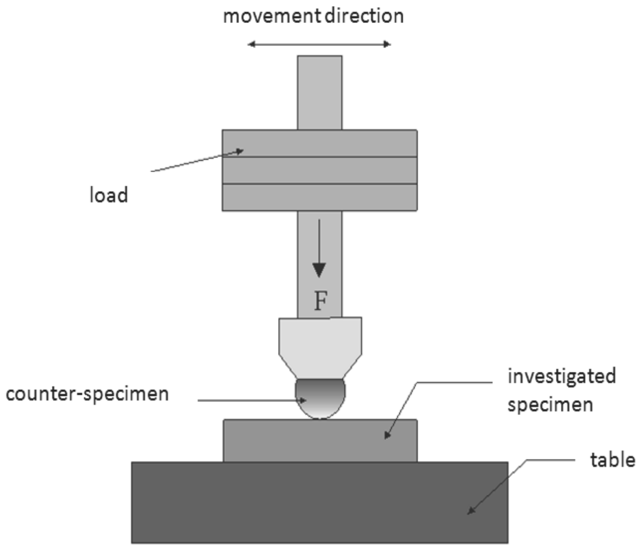

Figure 6. *Diagram of the abrasive wear resistance testing device*

Resistance to brittle cracks (K_{IC}) was tested according to [52] using the Palmqvist method (Fig. 7). The tests were made at the appropriate samples prepared in advance.

The following formulae were used to determine the K_{IC} co-efficient:

$$H = \frac{1.854 \times P}{\left[(d_1 + d_2) \times \frac{1}{2} \right]^2}, \quad \text{N/mm}^2, \tag{1}$$

where:

P – load applied, N,

d_1, d_2 – length of the indention diagonal, mm,

$$T = l_1 + l_2 + l_3 + l_4 , \quad (2)$$

where:

T – sum of cracks length, mm,

$$K_{IC} = A\sqrt{H} \times \sqrt{\frac{P}{T}}, \text{ MNm}^{-3/2}, \quad (3)$$

where:

A – constant 0.0028.

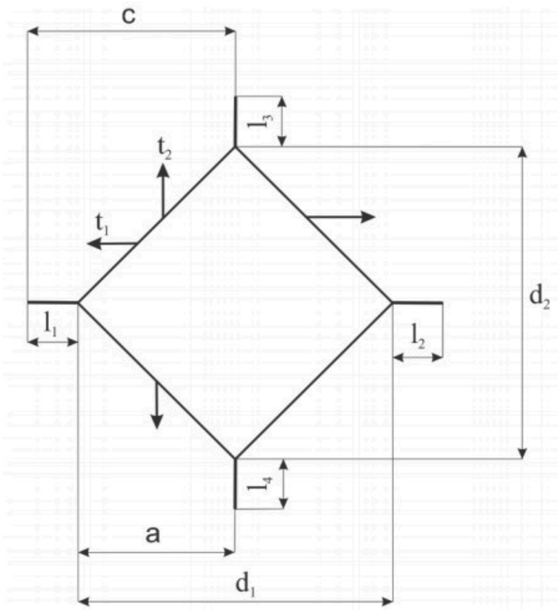

Figure 7. *Diagram of cracks system obtained with the Palmqvist method*

During investigations accept traditional material science methods also the Finite Element Method (FEM) was used. Next, the research results were experimentally verified using the X-ray spectrometer. FEM was used for performing a computer simulation of gradient stresses of carbide tool materials [25, 53], internal stresses and material work deformations. The actual model of the graded tool material was designed with Inventor 11 software and the strength analysis was performed with ANSYS 12.0 software. The following boundary conditions were assumed to simulate the internal stresses of the graded tool material:

- a variation in the sintering temperature is reflected by the specimens cooling process from 1460°C to the ambient temperature of 22°C,

- material properties for the material produced were assumed based on the product sheets of the MatWeb catalogue provided in Table 3.

Table 3. *Mechanical and physical properties assumed for the computer simulation of internal stresses occurring in the produced material made up of four layers with a varied fraction of tungsten carbide and a varied cobalt concentration [54]*

Properties	Four layers of GM-Co/WC			
	3% Co+97% WC	5% Co+95% WC	7% Co+93% WC	9% Co+91% WC
Young modulus, GPa	665	640	615	590
Poisson co-efficient	0.2809	0.2815	0.4774	0.5338
Density, g/cm^3	15.4	15.1	14.8	14.5
Thermal expansion, 10^{-6} 1/K	4.1	4.3	4.5	4.7
Heat conductivity, W/(m·K)	98	90	82	76
Specific heat, J/(kg·K)	138.7	144.5	150.3	156.1
Resistivity, Ω·m	5.4252	5.442	5.4588	5.4756
Tensile strength, MPa	1670.75	1641.25	1611.75	1580.25

A model for determining the internal stresses of tool work was prepared using the finite element method by assuming the actual specimens dimensions. The actual model was subjected to discretisation. The calculation model comprises 4968 nodes and 760 elements. The actual internal stresses in the tested materials were calculated in order to verify experimentally the results achieved by means of the modelling of the finite element method based on the measurements made using X-ray spectrometry. The calculations were made with the $\sin^2\psi$ method with the custom X'Pert Stress Plus software. The software incorporates data, as a database, necessary to calculate the values of material constants. A comparative analysis of computer simulations to the experimental results was then made.

2.3. Foresight methodology

The own reference data gathered when implementing the project "Foresight of surface properties formation leading technologies of engineering materials and biomaterials. FORSURF" [6] was used in order to determine the strategic position of the thematic area "Surface engineering of tool materials" against materials surface engineering and the manufacturing technologies of graded tool materials with the powder metallurgy methods in relation to other tool materials manufacturing methods. Nearly 300 independent foreign and domestic experts representing scientific, business and public administration circles have taken part in the foresight research up till now at the different stages of the works. The experts have completed approx. 600 multi-question surveys and held thematic discussions during 7 Workshops. The initial phase of the foresight research, including the evaluation of the steady state, technology review and strategic analysis with integrated methods, was conducted for about 500 groups of detailed materials surface engineering technologies. The following scientific and research methods were applied at this stage: extrapolation of trends, environment scanning, STEEP analysis, SWOT analysis, expert panels, brainstorming, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and statistical analysis. 10 critical technologies with the best development prospects and/or of crucial importance for the industry in the next 20 years were chosen from each of the 14 thematic areas analysed as a result of the research performed. A collection of 140 critical technologies were thoroughly analysed according to three iterations of the Delphi method carried out in consistency with the idea of e-foresight [55, 56] using information technology encompassing a virtual organisation, web platform and neural networks. Neural networks were used in a novel and experimental manner to analyse the cross impacts between events to determine how growth, stabilisation or a decrease in the importance of the relevant factors of the analysis will contribute to the occurrence of each of the three anticipated scenarios at a macro scale. The relevant manufacturing technologies of sintered graded tool materials analysed in this chapter were evaluated based on the opinions of the key experts using the custom foresight-materials science research methodology [41]. The weighted scores method was used for a comparative evaluation aimed at classifying the importance of specific objects in the context of relationships between them. The approach adopted requires that the principle of relativising the evaluation criteria has to be applied, i.e. differences are assumed in the relevance of the criteria used and the principle of admissibility assuming that there is a certain set of admissibility conditions representing a selection filter classifying a certain object positively or negatively [57]. The weighted scores method enables to make a multi-criteria aggregate evaluation using a scale with intervals. A single-pole positive scale without zero, referred to as a universal scale of relative states, was used in the foresight research undertaken, where 1 is a minimum rate and 10 an extraordinarily high rate. The results of the expert evaluation of the technology for its potential, representing a realistic objective value of the particular technology, and for its attractiveness, reflecting the subjective perception of a specific technology by its potential users, were entered into one of the quarters of the **dendrological matrix of the technology value**. Wide-stretching oak is the most promising quarter guaranteeing the future success. A quarter of soaring cypress groups highly attractive technologies with a limited potential and rooted dwarf mountain pine symbolises technologies with a high potential and limited attractiveness. If an appropriate strategy is used in both cases for a specific technology, its further progress and a robust strategic position in the future is feasible. The technologies with their success being either unlikely or impossible are placed in the field of quaking aspen. **The metrological matrix of environment influence** presents graphically the results of evaluating the influence of external positive factors, i.e. opportunities and difficulties, on the technologies analysed. Each of the technologies assessed by the experts was entered into one of the matrix quarters. Sunny spring illustrates the most favourable external situation ensuring the future success. Rainy autumn, providing a chance for steady progress, corresponds to a neutral environment and hot summer to a stormy environment where the success of a technology is risky but feasible. Frosty winter informs that adverse factors exist mainly with technology development being difficult or unachievable. The results of the expert investigations visualised with the dendrological and meteorological matrix were at the next stage of scientific work entered into the **matrix of strategies for technologies** made up of sixteen fields by means of software based on pre-defined mathematic relationships [41]. The matrix presents graphically a position of each technology according to its value and environment influence intensity and identifies a recommended action strategy. The strategic development prospects of a given technology expressed in numbers correspond to the relevant areas of the matrix. **Strategic development tracks** were also entered into the matrix of strategies for technologies reflecting the anticipating growth of the technologies analysed for the three variants: a positive, neutral or negative ones in the nearest 20 years for the time intervals of 2015, 2020, 2025 and 2030.

3. Materials science research results

3.1. Sintering conditions and the reinforcing phase fraction versus GM-HSS/WC properties

Figures 8 to 10 present the Regression Function Plots (RFP) describing a relationship between the tungsten carbide reinforcing phase density and fraction volume, sintering temperature and sintering time for materials in a vacuum furnace and Figs. 11 to 13 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen (N_2+5% H_2). It was found based on the results of density measurements for the sintered gradient carbide steels that the sintering temperature and atmosphere have a substantial effect on the density values. The sintering time, therefore, does not have a major effect on the density of the tested materials. The density of gradient carbide steels is between 6.4 to 8.3 g/cm^3 and between 7.5 to 8.6 g/cm^3, respectively for GM-90HSS/10WC and GM-75HSS/25WC.

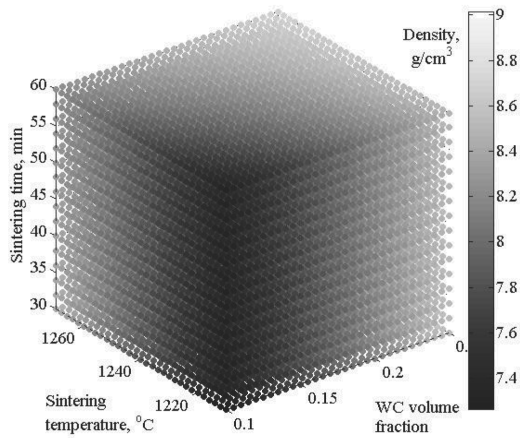

Figure 8. *RFP describing relationship between density and the WC volume fraction, temperature, and sintering time, for GM-HSS/WC sintered in a vacuum furnace*

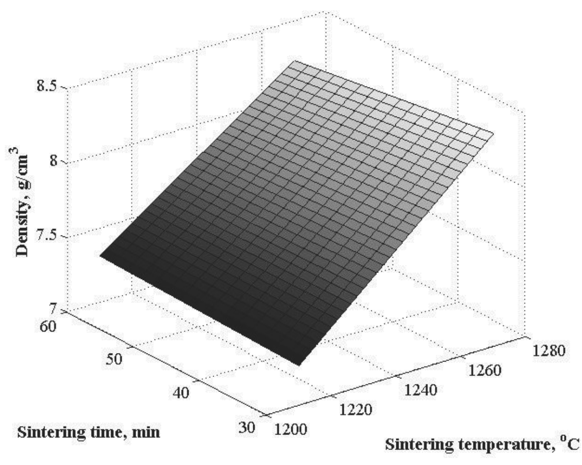

Figure 9. *RFP describing relationship between density and the temperature, and sintering time, for GM-90HSS/10WC, sintered in the the vacuum furnace*

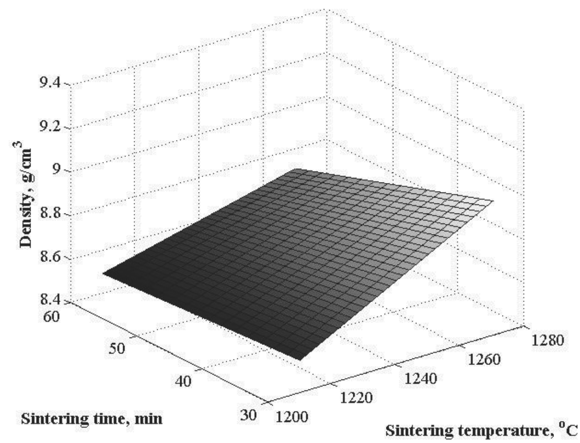

Figure 10. *RFP describing relationship between density and the temperature, and sintering time, for GM-75HSS/25WC, sintered in the the vacuum furnace*

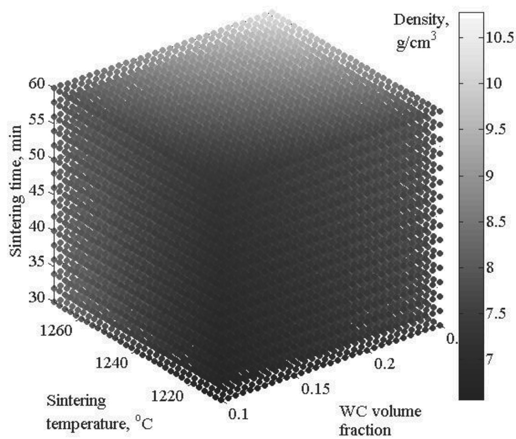

Figure 11. *RFP describing relationship between density and the WC volume fraction, temperature, and sintering time, for GM-HSS/WC, sintered in furnace with the atmosphere of the N_2+5% H_2*

Figures 14 to 16 show the regression function plots describing the relationship between HRA hardness and the reinforcing phase volume fraction, sintering temperature and sintering time for materials in a vacuum furnace and Figs. 17 to 19 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen (N_2+5% H_2). The results of hardness measurements for the surface layers of the tested materials sintered in a vacuum furnace and with the atmosphere of flowing nitrogen with an addition of hydrogen indicate that the sintering conditions and the fraction of the applied reinforcing phase significantly influence the hardness of graded materials. The hardness of surface layers for the tested materials is within 55.7-80.8 HRA for GM-90HSS/10WC and 64.7-84.2 HRA for GM-75HSS/25WC. Moreover, no significant impact of the type of the applied sintering atmosphere on the tested materials hardness has been identified. As the temperature and volume fraction of WC is growing, so is growing the hardness of such materials surface layers. Variations in the sintering time within the sintering temperature of 1210 to 1270°C do not cause substantial changes to the hardness of the graded materials. The maximum surface layer hardness of approx. 84.2 HRA was achieved for GM-75HSS/25WC sintered in a vacuum furnace at a temperature of 1230°C for 30 min.

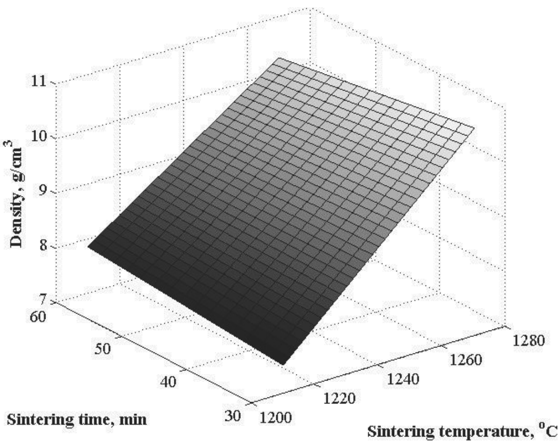

Figure 12. *RFP describing relationship between density and the temperature, and sintering time, for GM-90HSS/10WC, sintered in furnace with the atmosphere of the* N_2*+5%* H_2

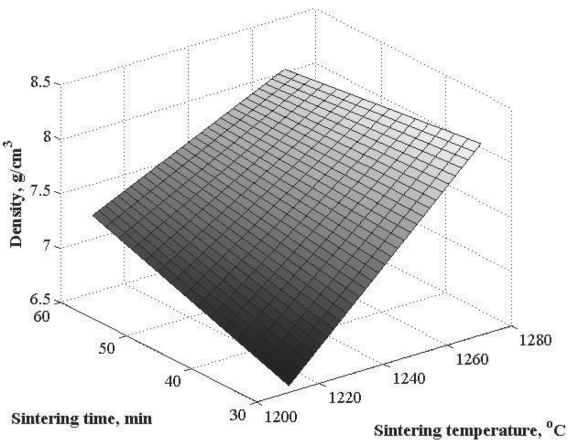

Figure 13. *RFP describing relationship between density and the temperature, and sintering time, for GM-75HSS/25WC, sintered in furnace with the atmosphere of the* N_2*+5%* H_2

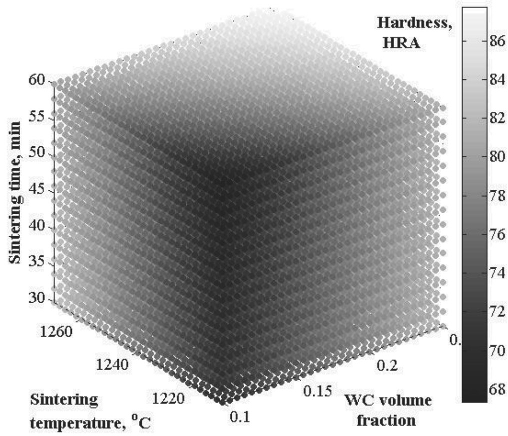

Figure 14. *RFP describing relationship between hardness and WC volume fraction, temperature, and sintering time, for GM-HSS/WC sintered in a vacuum furnace*

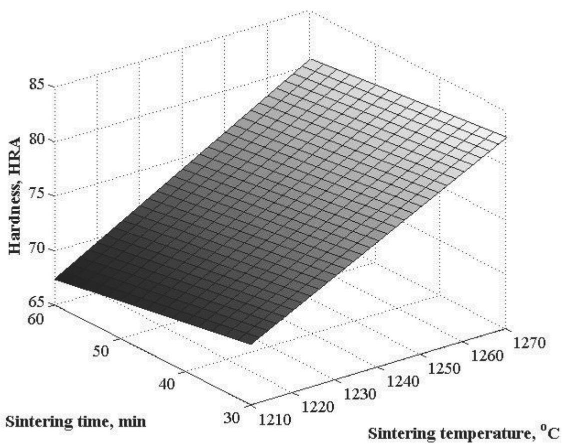

Figure 15. *RFP describing relationship between hardness and the temperature, and sintering time, for GM-90HSS/10WC sintered in a vacuum furnace*

Figures 20 to 21 illustrate the regression function plots describing the relationship between HV hardness and the measuring point location, sintering temperature and sintering time for materials in a vacuum furnace and Figs. 22 and 23 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen (N_2+5% H_2). The results of HV hardness measurements show a gradient variation in the properties of the tested materials in their volume. The hardness value of all the tested materials, regardless the sintering conditions, changes along with the changing distance of the measuring point from the outer surface of the surface layer.

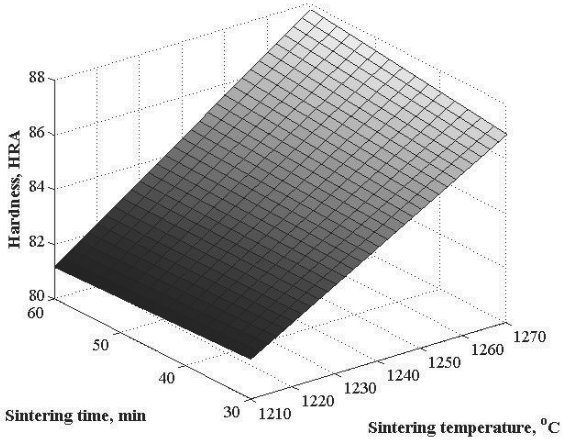

Figure 16. *RFP describing relationship between hardness and the temperature, and sintering time, for GM-75HSS/25WC sintered in a vacuum furnace*

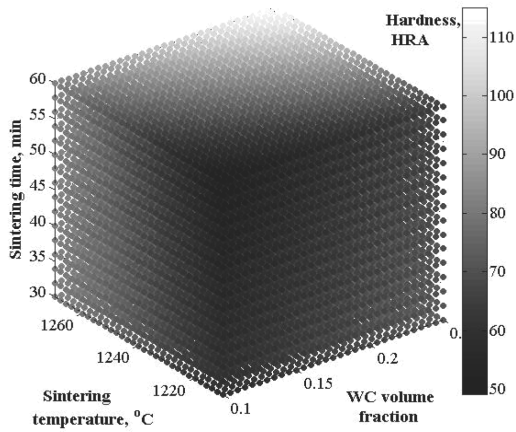

Figure 17. *RFP describing relationship between hardness and WC volume fraction, temperature, and sintering time, for GM-HSS/WC, sintered in the furnace with the atmosphere of the flowing N_2+ 5% H_2*

The hardness of GM-90HSS/10WC sintered in vacuum depending on the sintering temperature is within 500-800 HV in the surface layer and decreases along with the growing distance between the measuring point and the outer surface of the surface layer up to 270-510 HV in the substrate layer. The hardness of GM-90HSS/10WC sintered in the atmosphere of the flowing gas mixture (N_2+5% H_2) is within 580-680 HV in the surface layer and also declines along with the growing distance of the measuring point from the outer surface of the surface layer up to 350-540 HV. In GM-75HSS/25WC where the fraction of the WC reinforcing fraction in the individual layers is higher (25% of WC in the surface layer), a hardness variation increases by approx. 100 HV in the surface layer. For GM-75HSS/25WC sintered in vacuum, it is within the range of 600-900 HV and decreases to 250-470 HV, and for GM-75HSS/25WC sintered with the atmosphere of the flowing mixture of gases, it is 300-860 HV in the surface layer and decreases along with the growing distance of the measuring point from the outer surface of the surface layer to 240-510 HV in the substrate layer.

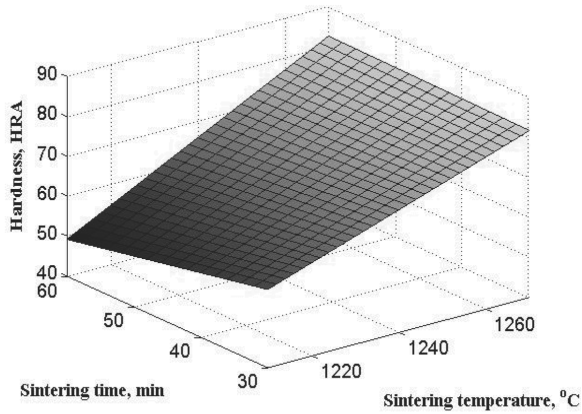

Figure 18. *RFP describing relationship between hardness and the temperature, and sintering time, for GM-90HSS/10WC, sintered in the furnace with the atmosphere of the flowing* N_2*+5%* H_2

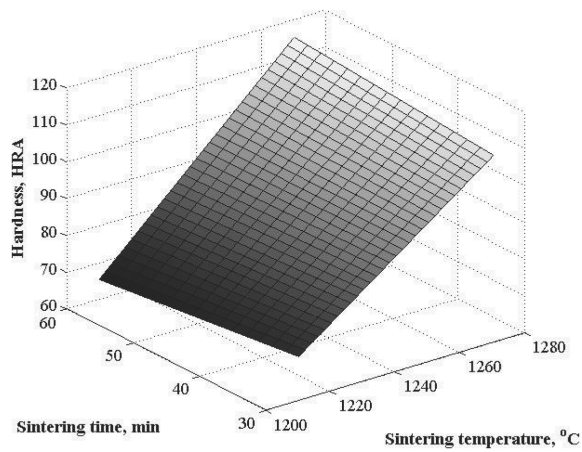

Figure 19. *RFP describing relationship between hardness and the temperature, and sintering time, for GM-75HSS/25WC, sintered in the furnace with the atmosphere of the flowing* N_2*+5%* H_2

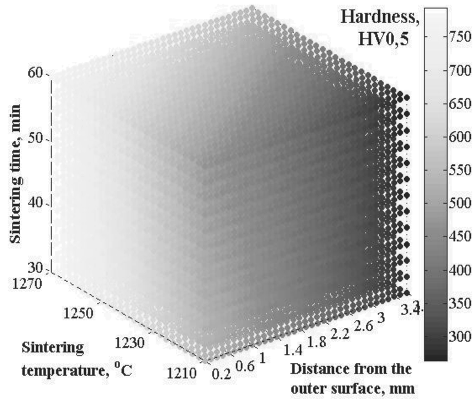

Figure 20. *RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-90HSS/10WC sintered in a vacuum furnace*

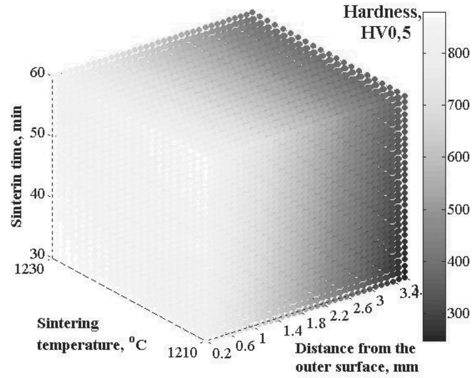

Figure 21. *RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-75HSS/25WC sintered in a vacuum furnace*

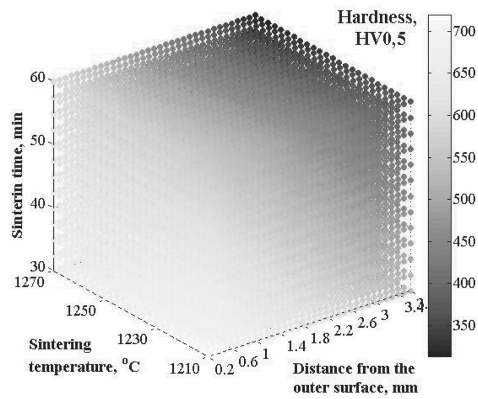

Figure 22. *RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-90HSS/10WC, sintered in the furnace with the atmosphere of the flowing N_2+5% H_2*

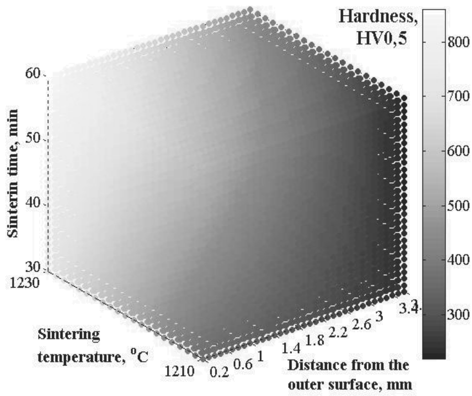

Figure 23. *RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-75HSS/25WC, sintered in the furnace with the atmosphere of the flowing* N_2*+5%* H_2

The regression function plot describing the relationship between porosity and the fraction volume of the reinforcing phase in the individual layers and sintering temperature is shown in Fig. 24. It was found that the area with higher porosity in the materials sintered at 1210°C is limited only to the surface layers and disappears almost completely along with the growing sintering temperature.

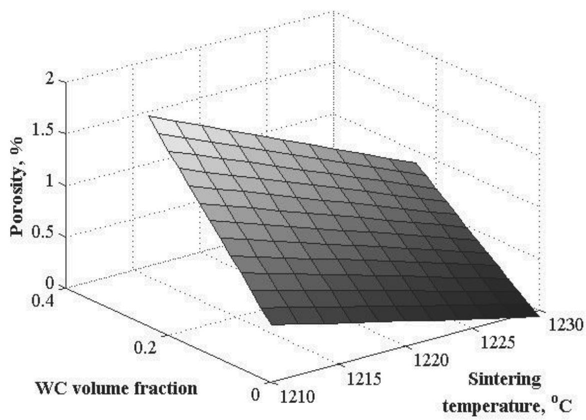

Figure 24. *RFP describing relationship between porosity and the volume fraction of the reinforcing phase and sintering temperature, for GM-HSS/WC sintered in a vacuum furnace for 30 min*

3.2. Sintering conditions and the reinforcing phase fraction versus GM-HSS/WC structure

It was found on the basis of metallographic research that such type of materials can be produced with a conventional method of powders metallurgy and both, the technological conditions of sintering and the fraction volume of the reinforcing phase impact the materials structure (Figs. 25, 26). The structure of the graded materials substrate layer is represented by alloy ferrite and MC and M_6C primary carbides (Fig. 27), whereas WC carbide and W_2C phase not occurring in the moulding exist in the intermediate layers and in the surface layer of graded materials (Fig. 28). Figs. 29 to 32 present the impact of sintering conditions on the structure of the substrate structure containing high-speed steel only, of intermediate layers and the surface layer of GM-90HSS/10WC and GM-75HSS/25WC.

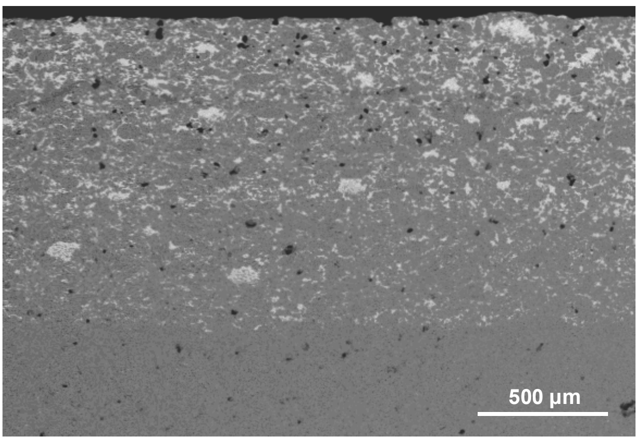

Figure 25. *Microstructures of GM-90HSS/WC sintered in the vacuum furnace at the temperature of 1230°C, for 30 min*

It was found by observing the structure of graded materials that the impact of temperature and sintering time is clear. The structure of the sintered GM-90HSS/10WC substrate layers has been changed considerably. Some of primary carbides are occupying the areas with concave surface and have an elongated shape characteristic for eutectic carbides. If sintering temperature is increased, the primary carbides will grow as a result in the material structure. A structure distinctive for remelted high-speed steel is present. Large carbides (approx. 5 µm)

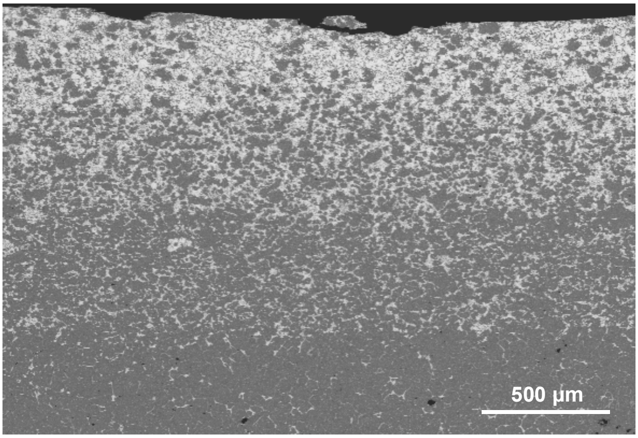

Figure 26. *Microstructures of GM-90HSS/10WC sintered in the vacuum furnace at the temperature of 1230°C, for 30 min*

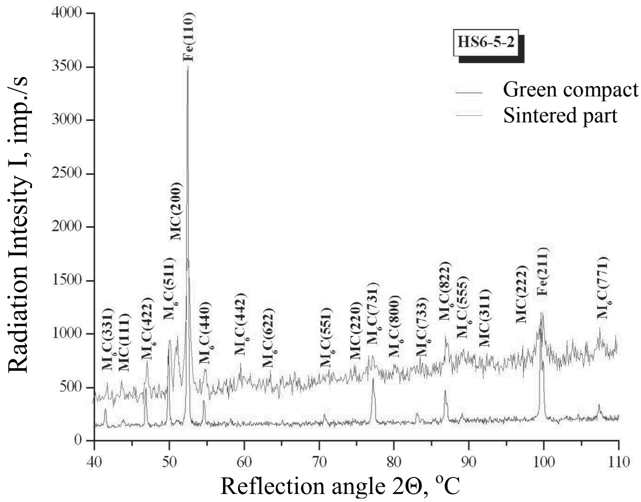

Figure 27. *Results of the X-ray phase analysis for the sintered test piece with the HS6-5-2 layer; diffraction patterns were shifted along the vertical axis to show the results more clearly*

are arranged mainly at the boundaries of grains and small carbides (< 1 µm) are arranged inside the grains. If the sintering temperature is raised, then the eutectic carbides in the substrate layer structure occupy larger and larger areas and, at the sintering temperature of 1270°C, fill spaces between the grains of high-speed steel. The growing volume fraction of the WC strengthening phase in carbide steels causes primary carbides to grow. The growth of primary carbides in the substrate layers structure of GM-75HSS/25WC can be explained with the fact that the carbon originating from the dissolving WC carbide located in the intermediate layer and in the surface layer is reducing the sintering temperature. For this reason, while the fraction of WC carbide is growing, so is growing the concentration of the dissolved carbon thus reducing the sintering temperature value of the substrate layer (of high-speed steel) from 1250°C to 1210°C. A higher sintering temperature intensifies, most of all, proneness to the uncontrolled growth of carbides at the final stage of sintering.

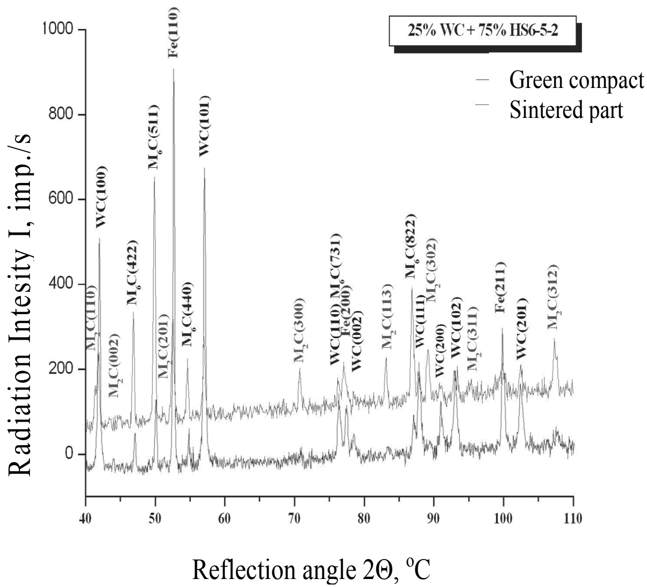

Figure 28. *Results of the X-ray phase analysis for the sintered test piece with the 75HSS/25WC surface layer; diffraction patterns were shifted along the vertical axis to show the results more clearly*

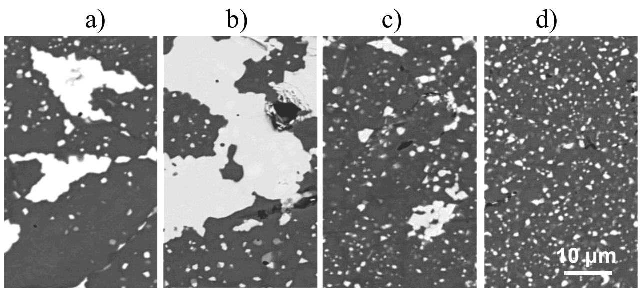

Figure 29. *Layers structure of GM-90HSS/10WC sintered in a vacuum furnace at the temperature of 1210°C, for 30 min; a) surface layer, b), c) intermediate layers, d) substrate layer*

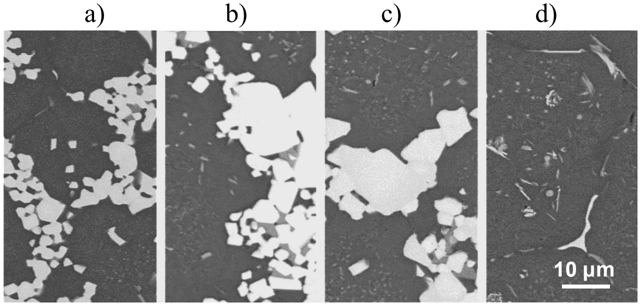

Figure 30. *Layers structure of GM-90HSS/10WC sintered in a vacuum furnace at the temperature of 1270°C, for 60 min; a) surface layer, b),c) intermediate layers, d) substrate layer*

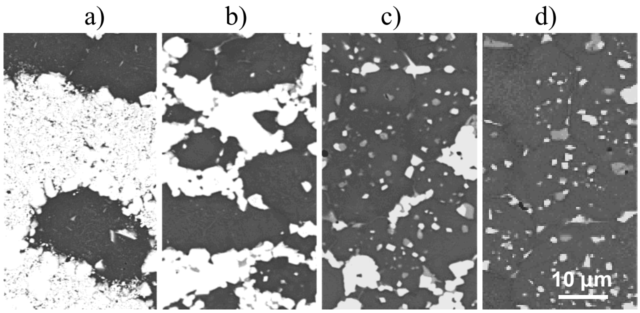

Figure 31. *Layers structure of GM-75HSS/25WC sintered in a vacuum furnace at the temperature of 1210°C, for 30 min; a) surface layer, b),c) intermediate layers, d) substrate layer*

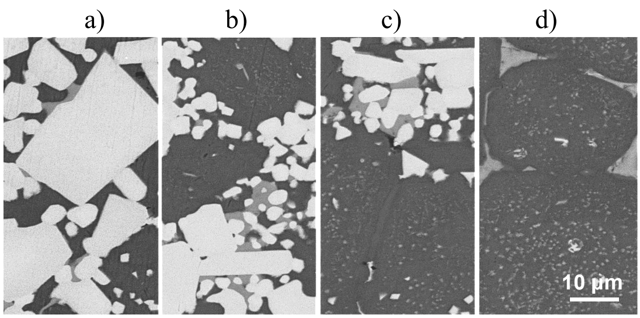

Figure 32. *Layers structure of GM-75HSS/25WC sintered in a vacuum furnace at the temperature of 1230°C, for 60 min; a) surface layer, b),c) intermediate layers, d) substrate layer*

3.3. Heat treatment conditions versus GM-75-HSS/25WC structure and properties

It was found based on the hardness tests that the impact of heat treatment conditions on the hardness of the tested graded materials in the quenched and tempered condition is noticeable in the substrate layers composed of the same high-speed steel (Figs. 33, 34). The HRC hardness tests of graded materials substrate layers in the heat-treated condition show a clear effect of the tempering temperature on the hardness value. The maximum secondary hardness effect of approx. 66.7 HRC was achieved in the materials austenitised at 1210°C for 80 s, quenched and tempered at 560°C. If the tempering temperature is raised to 590°C, hardness is lowering as compared to the condition corresponding to the secondary hardness effect, by approx. 1 HRC – for the materials austenitised at 1180 and 1210°C and by approx. 2 HRC – for the materials austenitised at 1150°C (Fig. 35). The greatest decline in hardness after tempering in the corresponding conditions, by approx. 3 HRC, has been seen for the substrate layers of the graded materials pre-quenched from the temperature of 1120°C.

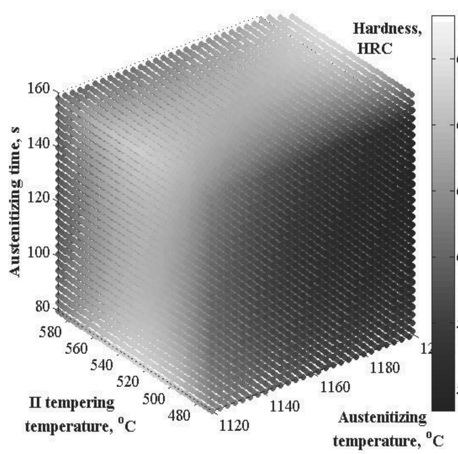

Figure 33. *RFP describing relationship between hardness and the temperature, and austenitising time, and second tempering temperature for the GM-100HSS substrate layer of GM-75HSS/25WC*

The hardness of the graded materials surface layer is within the range of 69.2-71.6 HRC (Figs. 36-38). Heat treatment performed in the conditions applied in work is substantially improving hardness in the surface layer of materials by 5.8-8.2 HRC up to the value of 69.2-

71.6 HRC. The material austenitised at the temperature of 1120°C for 120 s, quenched and then tempered twice at the temperature of 530°C shows the highest hardness of the surface layer of 71.6 HRC. No statistically significant change in the impact of heat treatment conditions within the investigated range has been found on the hardness of surface layers after quenching and tempering the graded materials containing 25% of WC.

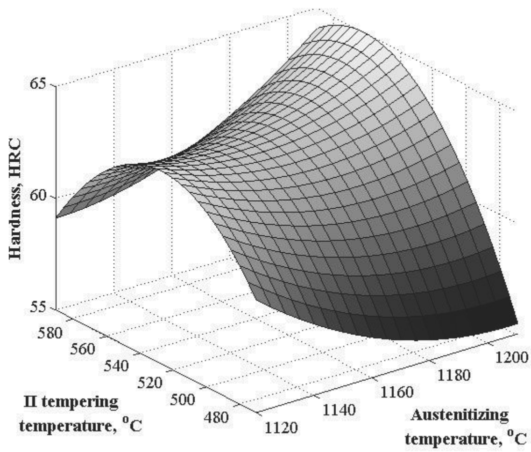

Figure 34. *RFP describing relationship between hardness and the second tempering temperature, and the austenitising temperature, for the GM-100HSS substrate layer of GM-75HSS/25WC austenitised for 120 s*

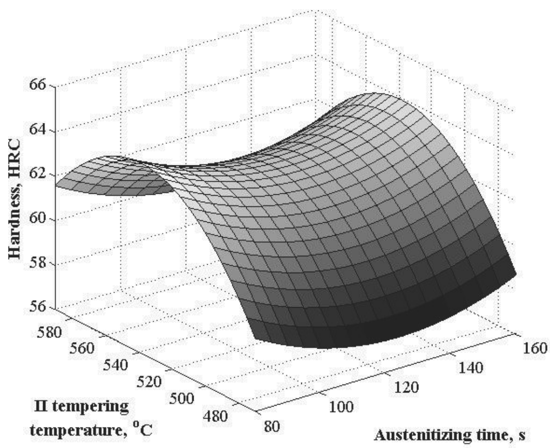

Figure 35. *RFP describing relationship between hardness and the second tempering temperature, and the austenitising time, for the GM-100HSS substrate layer of GM-75HSS/25WC austenitised at the temperature of 1150°C*

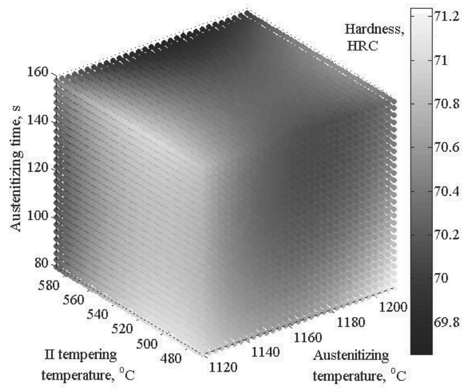

***Figure 36.** RFP describing relationship between hardness and the temperature, and austenitising time, and second tempering temperature for the GM-75HSS/25WC surface layer*

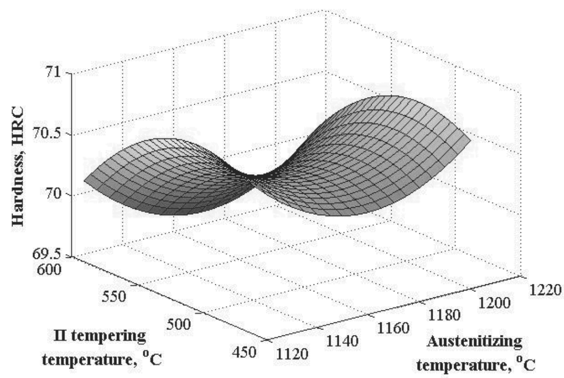

***Figure 37.** RFP describing relationship between hardness and the second tempering temperature, and the austenitising time, for the GM-75HSS/25WC surface layer austenitised for 120 s*

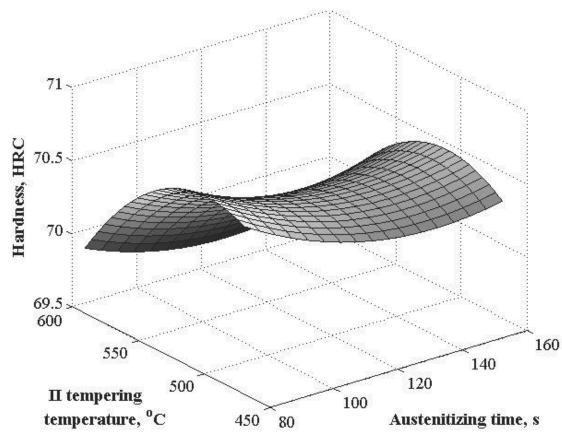

***Figure 38.** RFP describing relationship between hardness and the second tempering temperature, and austenitising time, for the GM-75HSS/25WC surface layer austenitised at the temperature of 1150°C*

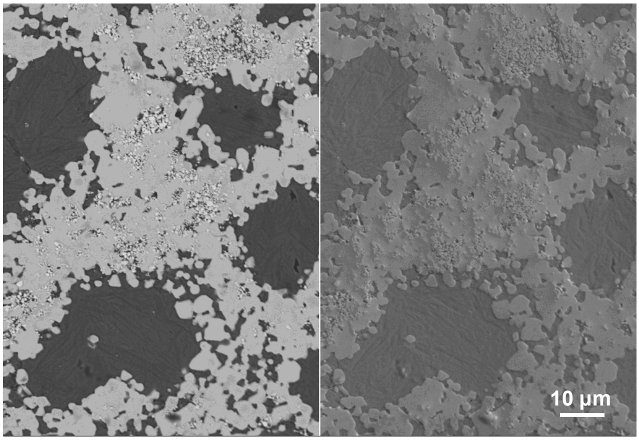

Figure 39. *Structure of the quenched and twice tempered GM-75HSS/25WC surface layer*

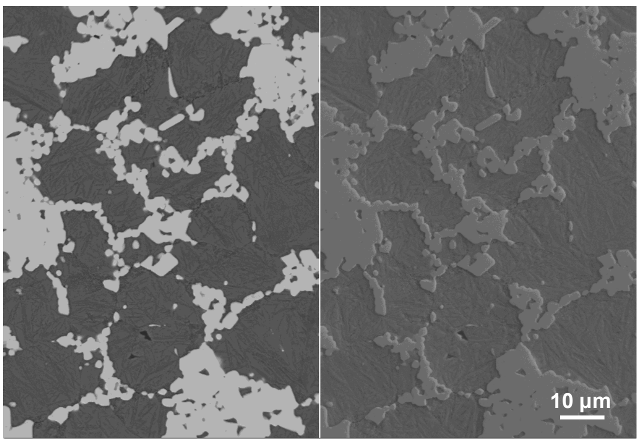

Figure 40. *Structure of the quenched and twice tempered GM-85HSS/15WC intermediate layer*

The structure of individual layers of the quenched and twice tempered GM-75HSS/25WC is presented in Figs. 39-42. The structure of the tested graded materials in the quenched condition represents martensite with residual austenite, M_6C and MC type carbides, both primary and secondary, unsolved in a solid solution during austenitising and WC carbides in the surface layer of materials (Fig. 43). The primary and secondary carbides unsolved in a solid solution during austenitising have a large effect on the grain size of the primary austenite. Fig. 44 presents the results of the analysis of the volume fraction of carbides calculated with quantitative metallography methods. Variations in the primary austenite grain size indicator acc. to Snyder-Graff in the substrate layers containing 100% of HS6-5-2 high-speed steel of graded materials, depending on temperature and austenitising time are presented in Fig. 45. The substrate layer of the gradient carbide steel quenched from 1120°C after austenitising for 80 s shows the primary austenite grain of approx. 12 acc. Snyder-Graff. The primary austenite grain increases along with the higher austenitising temperature, reaching the indicator value of approx. 6 acc. to Snyder-Graff – after quenching from 1210°C. The extension of austenitising time also supports the growth of primary austenite grain, whereas the extension of austenitising time is less intensive than increasing the austenitising temperature.

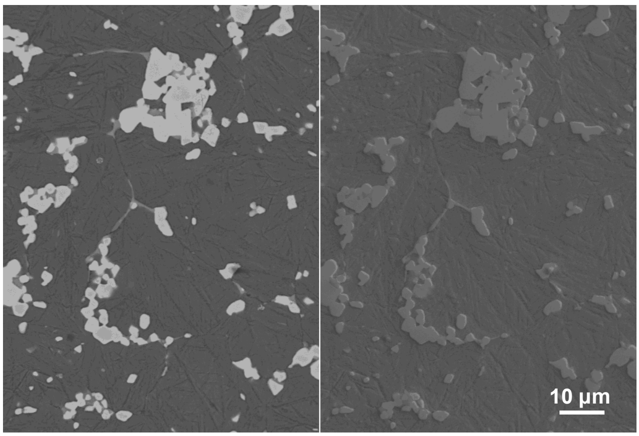

Figure 41. *Structure of the quenched and twice tempered GM-95HSS/5WC intermediate layer*

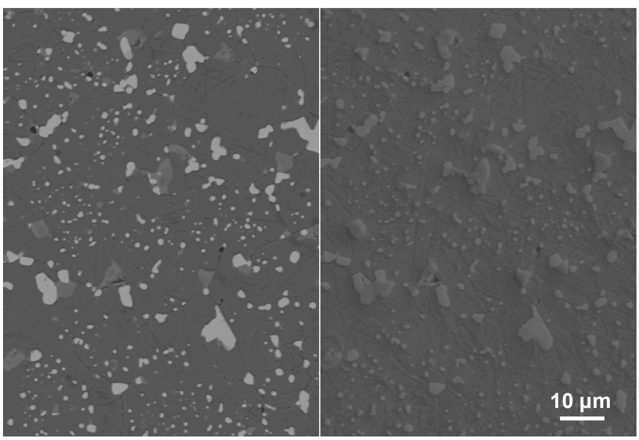

Figure 42. *Structure of the quenched and twice tempered GM-100HSS substrate layer of GM-75HSS/25WC*

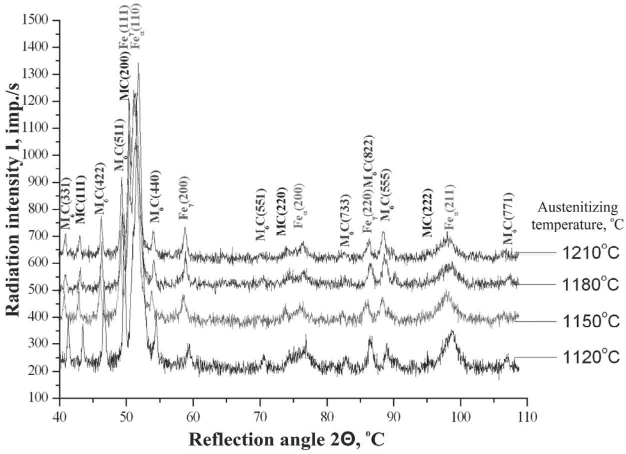

Figure 43. *Results of the X-ray phase analysis from the GM-100HSS substrate layer of the GM-75HSS/25WC test piece, austenitised at the temperature of 1210°C, for 120 s; diffraction patterns were shifted in respect to the vertical axis to show the results more clearly*

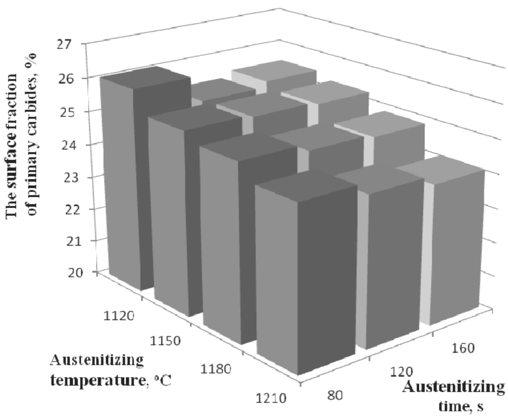

Figure 44. *Relationship between the surface portion of carbides in the GM-100HSS substrate layer of hardened GM-75HSS/25WC and austenitising conditions*

The matrix of the tested gradient carbide steels in the quenched condition from the temperature of 1210°C, ensuring maximum secondary hardness after tempering, is represented by martensite with residual austenite. The fraction volume of residual austenite in the structure of the tested substrate layer of the quenched gradient carbide steels is dependent upon the austenitising conditions. It was found with the X-ray quantitative phase analysis that the fraction volume of residual austenite of the quenched specimens is within the range of approx. 5.7 to 26.5% (Fig. 46). The fraction volume of residual austenite in the substrate layer of the gradient carbide

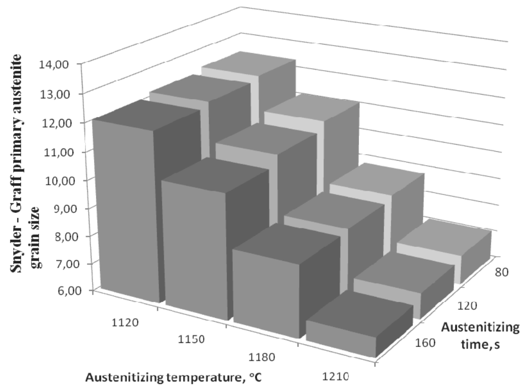

Figure 45. *Relationship between the primary austenite grain size in the GM-100HSS substrate layer of hardened GM-75HSS/25WC and austenitising conditions*

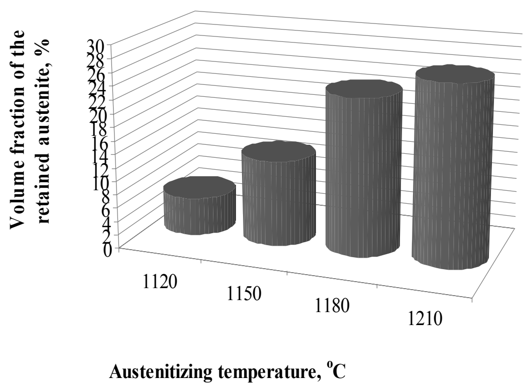

Figure 46. *Relationship between the volume portion of the retained austenite and austenitising temperature in the GM-100HSS substrate layer of hardened GM-75HSS/25WC austenitised for 120 s*

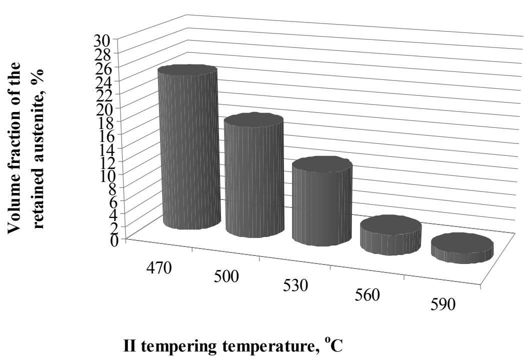

Figure 47. *Relationship between the volume portion of the retained austenite with the second tempering temperature in the GM-100HSS substrate layer of GM-75HSS/25WC hardened at the temperature of 1210°C after austenitising for 120 s*

steels quenched from the temperature of 1210°C, ensuring the maximum secondary hardness after subsequent double tempering within the temperature range of 470 to 590°C decreases and, according to the temperature of the second tempering, is within, accordingly, 1.6-23.8% (Fig. 47).

3.4. GM-3Co/97WC structure and properties

It was found based on the density measurements of sinters for the newly developed graded tool materials in the cobalt matrix that the material sintered using isostatic hot pressing and pressure sintering exhibits the highest density. The density of materials produced after sintering with isostatic hot pressing at the temperature of 1460-1425°C is, respectively, 14.60 g/cm^3, and the density of the materials subjected to free sintering at the temperature of 1460°C, is 12.96 g/cm^3, accordingly. It was found when analysing the impact of the sintering process parameters on density that the density increases while reducing porosity along with extending the process time and temperature. The X-ray quantitative analysis method performed using an EDS scattered radiation spectrometer (Figs. 48, 49) confirms that W, C, and Co element is present, respectively, in the hard phase of tungsten carbide and the binding phase of cobalt in the specific layers of the graded tool material. The newly developed graded tool material is characterised by a compact structure due to the uniformly distributed fraction of the binding phase between the hard carbide phase. It was confirmed through the tests of thin foils made in a transmission electron microscope (Fig. 50) that the sintered graded tool materials contain tungsten carbide and cobalt grains.

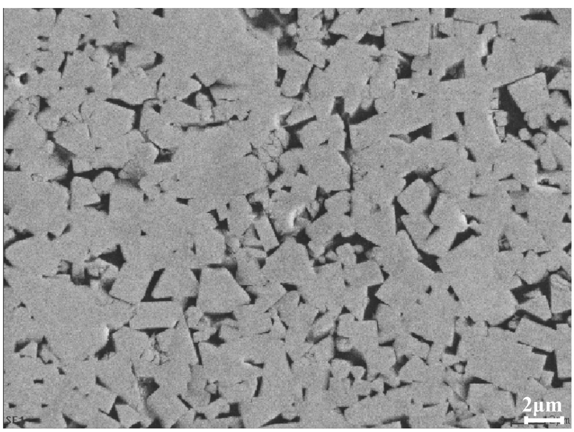

Figure 48. *Structure of the GM-3Co/97WC surface layer sintered in a vacuum furnace at the temperature of 1460°C*

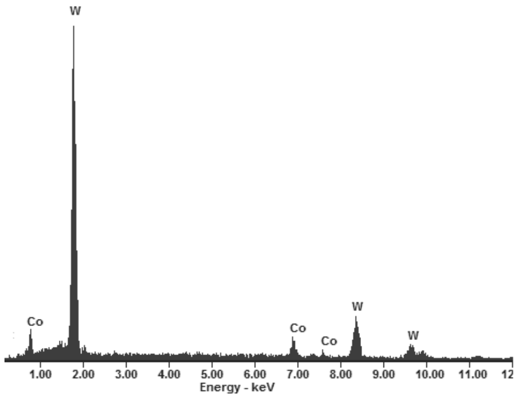

Figure 49. *Diagram of intensity according to energy of scattered X-ray radiation for the whole area for the surface layer*

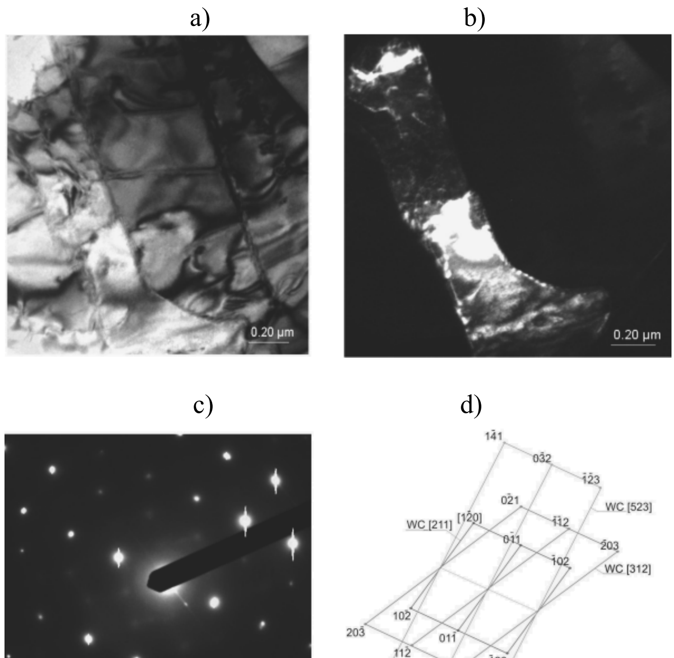

Figure 50. *The structure of thin foils made of WC-Co sintered carbide after sintering at the temperature of 1460°C; a) image in the light field; b) image in the dark field of Co and WC; c) diffraction pattern from the area as in figure a; d) diffraction pattern solution from figure c [58]*

The results of HV hardness measurement (Fig. 51) of the produced tool materials with the growing fraction of WC carbide in relation to the cobalt matrix towards the tool surface indicate gradual increase in hardness. The hardness of the material sintered at the temperature of 1460°C in vacuum is within 1410-1295 HV and decreases along with the growing distance of the measuring point from the outer surface of the surface layer to the substrate. The hardness of sintered material in 1460°C and pressed isostatically at the temperature of 1425°C is within the range of 1430-1326 HV in the surface layer and decreases towards the substrate.

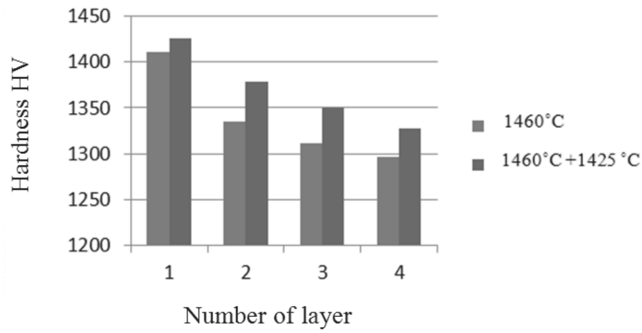

Figure 51. *Diagram of HV hardness, fraction volume and sintering temperature for four layers of GM-3Co/97WC*

The results of brittle cracking resistance tests K_{IC} for the sintered graded tool materials with a varied volume fraction of WC and Co phases in each layer of the material are presented in Fig. 52. The K_{IC} co-efficient results show there is a substantial relationship between sintering parameters and the cracking resistance of the individual tool materials [54, 59-61]. GM-3Co/ 97WC sintered at the temperature of 1460°C is characterised by high brittle cracking resistance. The average value of K_{IC} co-efficient of the material surface layer is 21 $MNm^{-3/2}$, and 16 $MNm^{-3/2}$ for the substrate. The fact that there is no clear difference for K_{IC} co-efficient in the surface layer and in the substrate of the materials sintered with isostatic pressing can be explained with the sintering time that is too long causing the gradient structure to fade partially or fully [27-28, 62]. The microscope observations of specimens fractures carried out (Fig. 53) are characterised by the systems of hollows and the convexities imparting the flaky character of the fracture, typical for brittle materials.

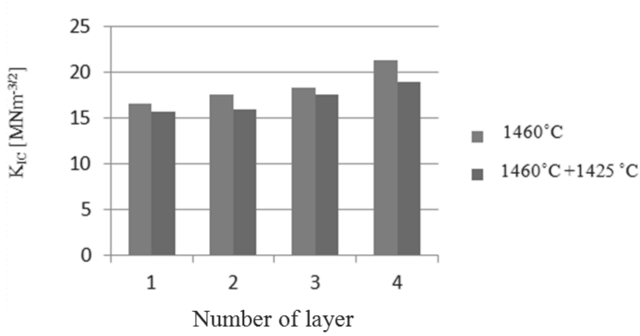

Figure 52. *Brittle cracking co-efficient diagram according to temperature and fraction volume of Co for four layers of GM-3Co/97WC sintered in a vacuum furnace*

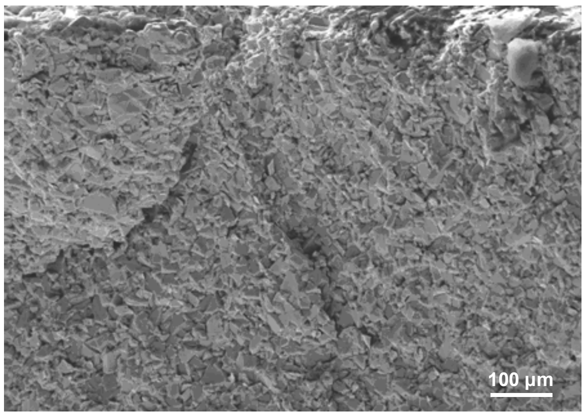

Figure 53. *Fracture structure of the GM-3Co/97WC surface layer sintered in a vacuum furnace at the temperature of 1460°C*

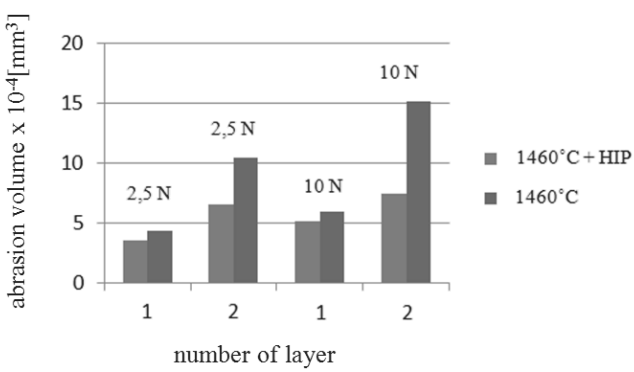

Figure 54. *Tribological wear of investigated GM-3Co/97WC*

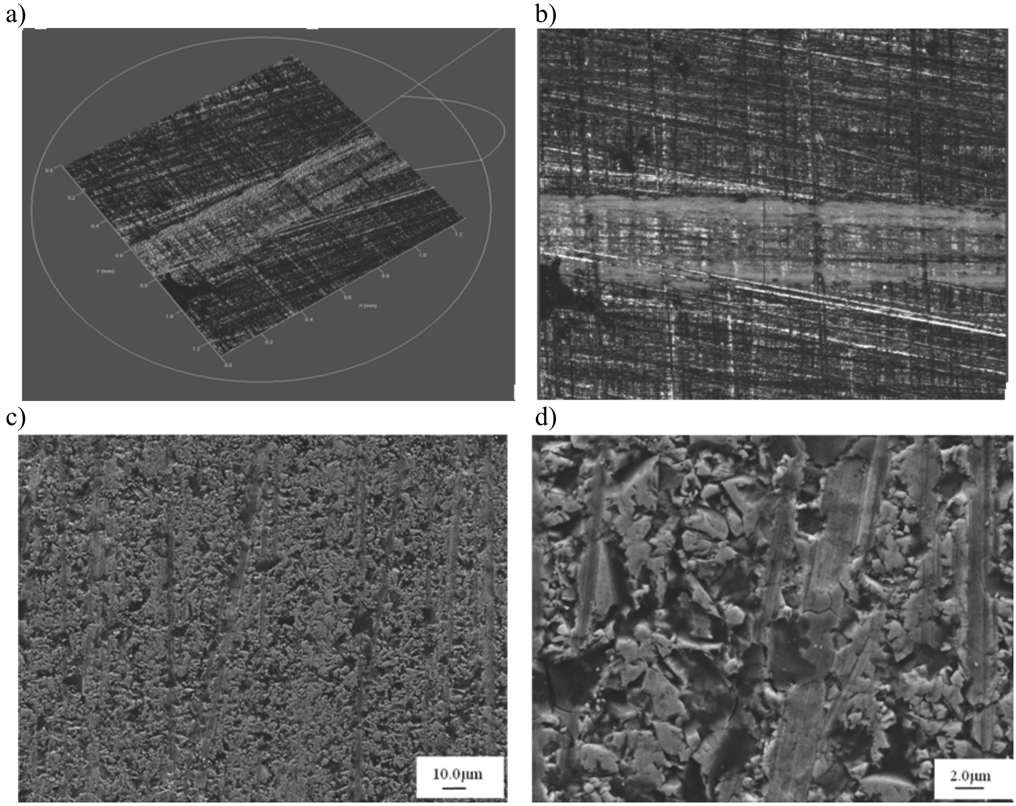

Figure 55. *The wear track of GM-3Co/97WC sintered in vacuum in the temperature of 1460°C and pressed isostatically at the temperature of 1425°C after 1000 cycles with the load of 2.5 N in the substrate; a) CLSM micrograph; b) wear track measurement; c), d) SEM micrographs*

The wear test results (Fig. 54) reveal that the materials sintered with isostatic pressing are characterised by much lower abrasive wear than those fabricated with free sintering. The material loss is caused by the detachment of particles due to micro-cutting or drawing in the counter-specimen – material friction area due to loose or restrained abradant particles or the protruding irregularities of the hard carbide phase (Fig. 55) [63]. The unequal width of wear signifies the presence of seizing whereupon the wear products are being attached to the counter-specimens and then detached elsewhere causing local roughness where the wear is smaller. The presence of aluminium and oxygen, most probably originating from aluminium oxide Al_2O_3 has been confirmed with the X-ray quantitative microanalysis method carried out with an EDS scattered radiation spectrometer in the material wear track. This stems from the wear products being attached to the counter-specimens and then detached elsewhere causing local roughness where the wear is smaller. The results of the abrasive wear measurement for the sintered graded tool materials of tungsten carbide in the cobalt matrix show the gradient shift of the tested materials properties depending on the presence of the binding phase. Numerous factors are, therefore, impacting the wear of graded materials: the presence of the binding phase, the counter-specimens load value and also the wear track (the number of cycles).

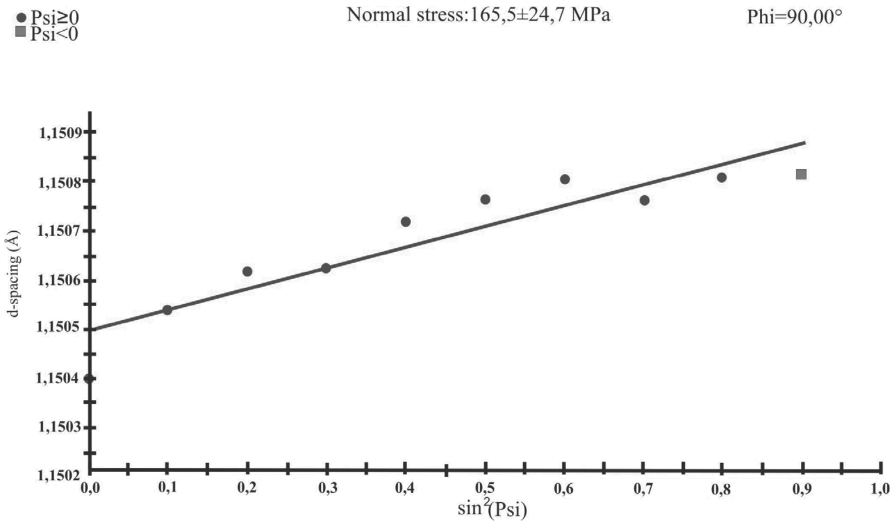

Figure 56. *Changes in the interplanar distance value of d reflex (201) according to sin^{2}ψ sintering temperature of 1460°C; results for the GM-3Co/97WC surface layer*

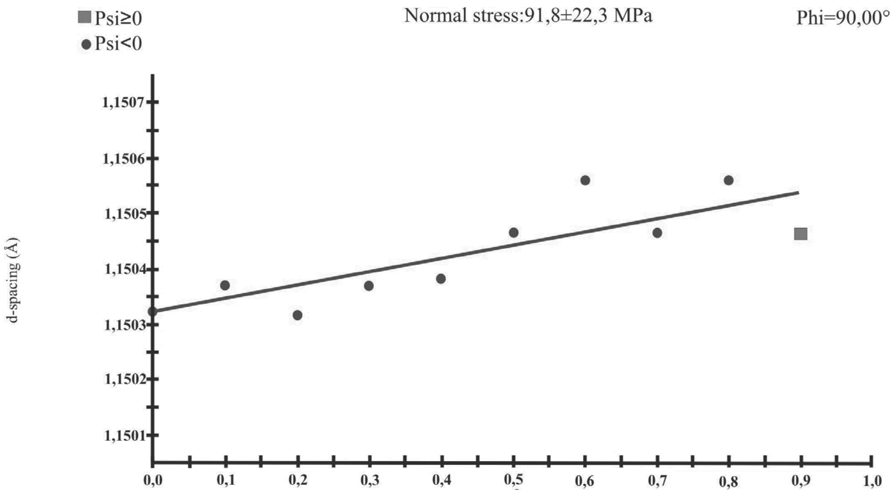

Figure 57. *Changes in the interplanar distance value of d reflex (201) according to sin^{2}ψ sintering temperature of 1460°C; results for the GM-9Co/91WC substrate layer*

The results of calculations of internal stresses in the tested materials obtained with an analysis of reflex shift (201) with the $sin^2\psi$ method performed to verify the modelling results are presented in Figs. 56, 57 and in Table 4. The results obtained with a computer simulation of internal stresses with the finite element method match the results of the stress measurements obtained with the $sin^2\psi$ method (Table 4).

Table 4. *The results of stresses obtained experimentally compared with the results of computer simulation*

	Sintering temperature, °C	Stresses determined experimentally, MPa	Simulated stresses, MPa
Surface layer	1460	162 ± 24	170
Substrate	1460	91 ± 22	80

4. Strategic position of graded tool materials manufacturing

4.1. Development perspectives of surface engineering of tool materials

The results of the technology foresight [6] performed with the sample size of 198 have revealed that tool materials surface engineering holds a stable position with respect to other thematic areas of surface engineering. According to 41% of respondents, tool materials surface engineering technologies are in the group of technologies with the extensive prospects of industrial applications. 32% of the respondents maintain that numerous scientific and research works will be frequently devoted in the nearest 20 years to the technologies. 35% of the surveyed claim that the thematic area of “Surface engineering of tool materials” is crucial and its importance should be absolutely rising so that an optimistic scenario can come true of the country's development – “Race won” – assuming that the potential available is adequately utilised to fulfil the strategic objectives of development and so that people, statistically, are better off, social attitudes are optimistic and the prospects for the coming years bright. 38% of the surveyed persons think that that the significance of tool materials surface engineering technologies in relation to other materials surface engineering technologies will be growing, whereas 61% assert it will remain on the same level with only 2 persons claiming that the importance will diminish over the next 20 years. In judging the anticipated role of tool materials surface engineering, its contemporary widespread industrial applications should be underlined and also an ability to formulate freely the different, sometimes contradictory properties of the core and the surface layer of tools at a cost-effective level. Considering these reasons and the technology foresight results obtained, the future strategic position of tool materials surface engineering technologies should be considered stable and unthreatened, and their role in the development of overall materials surface engineering (mezo scale) and the development of the whole Polish economy (macro scale) will be certainly substantial.

4.2. Manufacturing of sintered graded tool materials in the future

The anticipated position of the powders metallurgy method allowing to change chemical and/or phase composition in the surface layer was determined based on the results of the own foresight research performed with the Delphic method [6]. The outcomes of the research indicate the strong development prospects of the materials that are still used mainly at a laboratory or semi-technical scale. It is also justified to apply them on a larger scale in the future in the industrial practice. 58% of the experts surveyed think that the technology group is critical and its importance should be absolutely rising so that an optimistic scenario of the country’s development, i.e. “Race Won” comes true in the nearest 20 years.

The foresight-materials science research described herein include the assessment, made by the key experts, of the potential and attractiveness of the three specific technologies of manufacturing GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method against the environment. A ten-degree universal scale of relative states (1: min, 10: max) was used to assess the individual groups of technologies, and an action strategy for the specific technologies was developed based on it and the forecast strategic development tracks were devised.

The key experts valued the technologies with the ten-degree scale for their: business, economic, humane, natural and system attractiveness as well as for their creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and a result obtained for the individual technologies was entered into the dendrological matrix of technologies value (Fig. 58). All the investigations conducted show that all the technologies analysed are in their embryonic phase (10) of development and are characterised by high attractiveness and limited potential. The technologies were entered into the quarter of the dendrological matrix referred to as soaring cypress. The following technologies were evaluated very much the same: (B) and (C), respectively: (4.82, 8.11) and (4.51, 8.55). The (B) technology of manufacturing GM-75HSS/25WC ensures that the expected material properties are achieved including its density, porosity and surface layer hardness for sintering and for thermal treatment while maintaining the ductility of the high-speed steel substrate. The (C) technology of manufacturing GM-3Co/97WC permits to produce a material with a varying gradient of chemical composition in its individual layers. This relates to hardness in the individual material layers being gradually differed while securing expected resistance to abrasive wear, brittle cracking resistance and the values of tensile stresses in the surface layer ensuring resistance to the formation and propagation of cracks. The (A) technology of manufacturing GM-90HSS/10WC allows to achieve smaller hardness of the surface layer measured with the Vickers and Rockwell method as compared to the (C) technology.

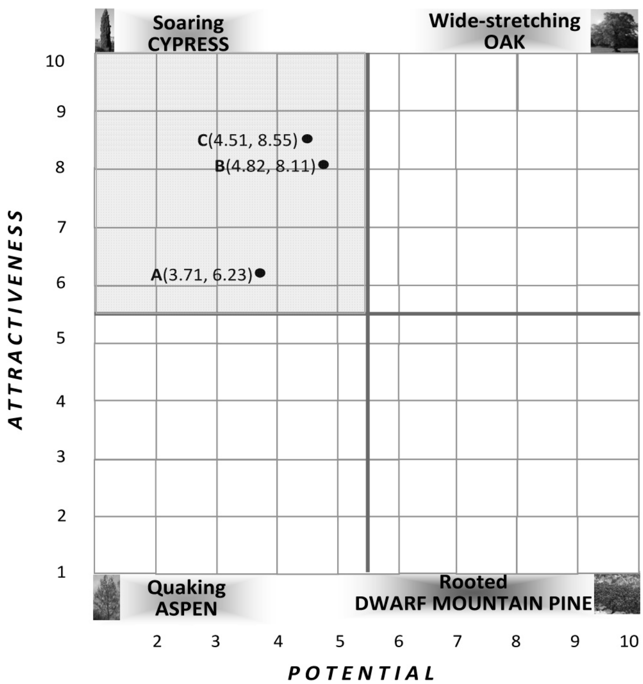

Figure 58. *The dendrological matrix of technology value for the manufacturing technologies of the following sintered graded tool materials; (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC*

Moreover, more time and a higher temperature for the (C) technology is required in the sintering process due to the lower contents of carbon in the powders mixture, hence it is less environmental friendly. For this reason the (A) technology has received relatively lowest scores from the experts (3.71, 6.23).

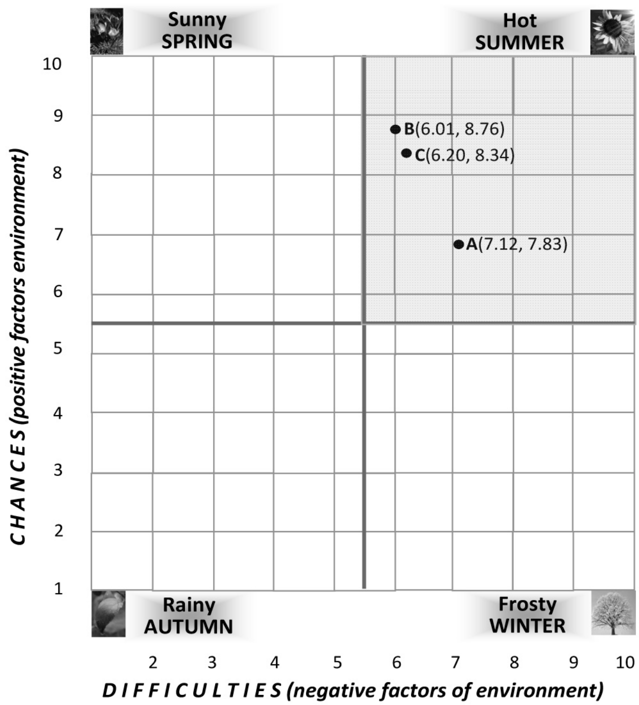

Figure 59. *The meteorological matrix of environment influence for the manufacturing technologies of the following sintered graded tool materials; (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC*

The evaluation results of the positive and negative environment influence on the relevant technologies were visualised with a meteorological matrix of environment influence, as illustrated in Fig. 59. The experts surveyed have found that the environment of all the technologies of manufacturing sintered gradient tool materials analysed experimentally is stormy. They were placed in the quarter of the meteorological matrix called hot summer for this reason. The environment of the technologies analysed brings ample opportunities related to the attractive prospect areas of future applications in the tool industry accompanied, however, by numerous difficulties such as fierce global competition and an alternative search for the effective technologies of manufacturing graded tool materials. Graded tool materials can also be manufactured through laser treatment (remelting and alloying) and in Physical Vapour Deposition (PVD), notably in combination with the glow thermochemical treatment hybridised in this process [41]. The (B) group of technologies (6.01, 8.76) and the (C) group of technologies saw similar results (6.20, 8.34), and the (A) group of technologies had a weaker position with the result of (7.12, 7.83) which stands for fewer opportunities and more difficulties in the future.

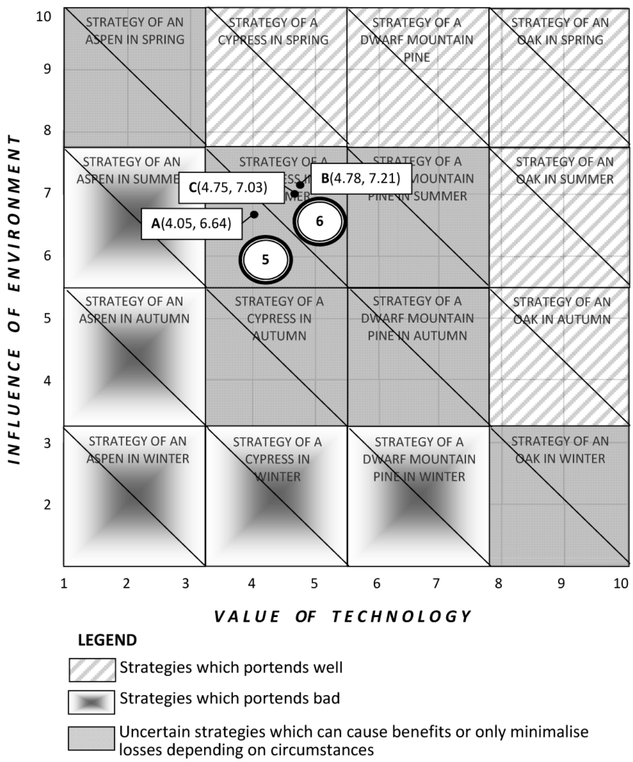

Figure 60. *The matrix of strategies for technologies prepared for the manufacturing technologies of the following sintered graded tool materials: (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC*

Using the pre-defined mathematical relationships, the specific numerical values provided in the following four-field matrices: the dendrological and meteorological matrix, were moved to the sixteen-field matrix of strategies for technologies (Fig. 60). The matrix depicts graphically the place of the examined technologies of manufacturing sintered graded tool materials according to their value and intensiveness of environment influence, indicating the relevant management strategy. The circles mark the strategic development prospects of a given group of technologies expressed in numbers. It is recommended to apply the cypress in summer strategy with reference to all the technologies analysed. According to the experts' assessment, the development prospects of the (B) and (C) technologies are moderate (6 points), and medium for the (A) technology (5 points). The recommended cypress in summer strategy assumes that the potential of attractive technologies must be enhanced and further scientific and research works need to be pursued to establish the optimum parameters and conditions of the manufacturing process in the risky environment conditions, and also a risk assessment is required. Either a customer should be fought for aggressively or the technology should be phased out slowly from the market depending on the result of the assessment. The cypress in summer strategy recommended for the analysed experimental technologies of manufacturing the sintered graded tool materials belongs to the unreliable ones. The greatest advantage of the analysed, newly developed technologies is their attractiveness connected with the promising mechanical and functional properties of the so produced tools. The risk factors, on the other hand, are as follows: the scale of future scientific studies in this scope, availability of financial resources for such research and the pace of development and the effectiveness of alternative technologies of manufacturing graded tool materials in laser treatment, Physical Vapour Deposition and hybrid (PVD and thermochemical treatment) processes.

The forecast strategic development tracks for the individual technology groups were prepared based on the expert opinions in the next part of the research works according to the three variants: optimistic, most probable and pessimistic ones for the relevant time intervals of: 2015, 2020, 2025 and 2030. The (B) and (C) technologies, i.e. manufacturing, respectively, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method are characterised by a moderate growth outlook evaluated by the experts at 6 points in a ten-degree scale. This evaluation is influenced by both, internal factors including further necessary scientific and research efforts to strengthen the potential of such technologies as well as external factors such as the sharply developing alternative technologies of manufacturing graded tool materials.

The most probable track of (B) technology development assumes that its potential is to be strengthened in 2015-2020 through continued scientific and research works concerned with the formation of the hardness of the surface layer of graded carbide steels reinforced with WC depending on their chemical composition and sintering conditions. The research held up till now has revealed that GM-75HSS/25WC after sintering at 1210°C for 30 minutes exhibits the most promising mechanical properties. Heat treatment improves the hardness of this material's surface layer considerably. The materials austenitised at 1120°C, quenched and then tempered twice at 530°C exhibit the highest hardness of the surface layer of 71.6 HRC. The adverse phenomena accompanying the (B) technology is that the specimen shrinks largely due to a high fraction of WC powder in the individual layers of the specimen subject to sintering related to the spatial deformation of the specimen. This disadvantage should be eliminated by reducing the fraction of tungsten carbide in the powders mixture and this requires further studies. The (B) technology will transit to the oak in summer field by strengthening the technology's potential in the years to follow (2025-2030) with the strong competition of alternative technologies. An optimistic track of the (B) technology development envisages extensive wide-scale research to strengthen the potential of the technology. As a consequence, the technology will be found in the oak in summer field already in 2020. The results of the research will allow to reinforce the position of the (B) technology versus the alternative technologies of manufacturing graded materials. This will eliminate in 2025 any major difficulties stemming from the environment and will allow for numerous industrial applications for the newly developed graded carbide steels reinforced with tungsten carbide and with the corresponding transition to the best field of the matrix called oak in spring. A pessimistic variant of (B) technology development, which is also probable due to its embryonic phase of development, envisages an insufficient interest from scientific and research and industrial circles. Its attractiveness will be declining gradually in 2015-2020 as a result with its limited potential being maintained and transition to the cypress in winter (2020) will be seen. The phenomena will be accompanied by the expansion of alternative technologies of manufacturing graded materials in laser treatment, Physical Vapour Deposition and (PVD) and hybrid (PVD and thermochemical treatment) processes causing further degradation to the (B) technology in the years to come (2025) and transition to the aspen in winter field with being eliminated entirely from the market.

The forecast development tracks of the (C) technology: most probable, optimistic and pessimistic are very similar to those predicted for the (B) technology. The future development of manufacturing GM-3Co/97WC with the conventional powder metallurgy method will be dependent upon the scale of scientific and research work conducted in this regard and the pace at which the alternative technologies of manufacturing graded tool materials are evolving. The directions of future research should focus on determining the optimum proportions of the cobalt and tungsten carbide powder mixture and on optimising the manufacturing process conditions including the crushing of powder mixture, forming the moulded pieces and sintering.

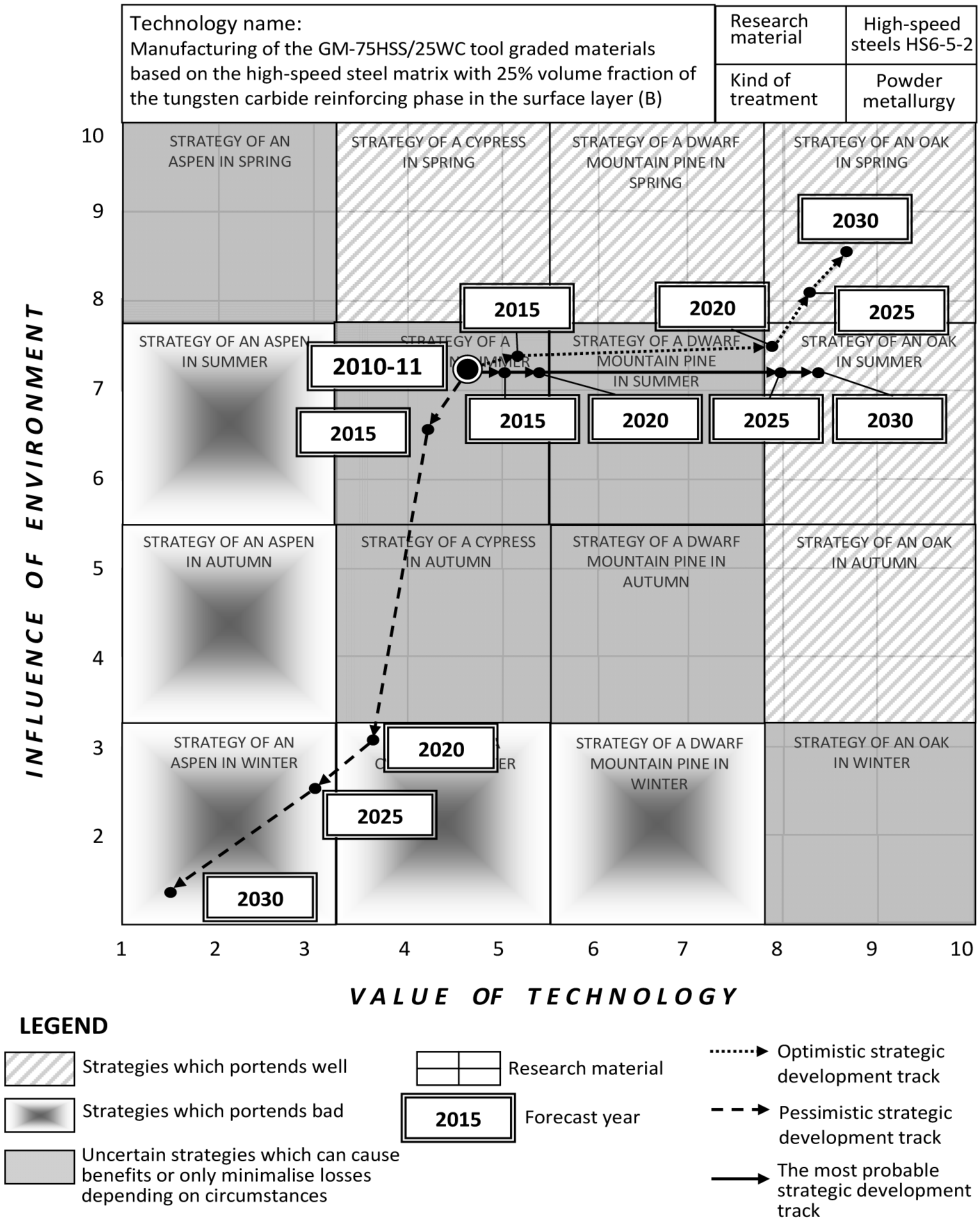

Figure 61. *Exemplary strategic development tracks prepared for the (B) technology: Manufacturing of the GM-75HSS/25WC sintered graded tool material*

This is because these factors are decisive for hardness, wear resistance, resistance to brittle cracking and the values of tensile stresses in the material surface layer, being crucial for its strength to the formation and propagation of cracks. It should also be noted when conducting future experiments that phase composition is equalising locally on the joint zones in the material that is heated too long (90 min) at a high sintering temperature of 1460°C and then compacted isostatically at 1425°C and the graded structure in the whole sinter volume is decaying.

Table 5. *The strategic development tracks of sintered graded tool materials. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic, (MP) – the most probable*

Technology symbol	Technology name	Steady state 2010-11	Type of strategic development tracks	Years			
				2015	2020	2025	2030
(A)	Manufacturing of the GM-90HSS/10WC sintered graded tool material	Strategy of a cypress in summer A (4.1, 6.6)	(O)	(4.8, 6.6)	(7.9, 6.6)	(8.2, 6.9)	(8.6, 7.2)
			(P)	(3.1, 2.8)	(2.3, 1.8)	(1.0, 1.0)	(1.0, 1.0)
			(MP)	(3.7, 3.2)	(3.1, 2.8)	(1.7, 1.4)	(1.0, 1.0)
(B)	Manufacturing of the GM-75HSS/25WC sintered graded tool material	Strategy of a cypress in summer B (4.8, 7.2)	(O)	(5.2, 7.3)	(7.9, 7.4)	(8.3, 8.1)	(8.7, 8.6)
			(P)	(4.2, 6.6)	(3.7, 3.1)	(3.1, 3.6)	(1.5, 1.3)
			(MP)	(5.1, 7.2)	(5.4, 7.2)	(7.9, 7.2)	(8.3, 7.2)
(C)	Manufacturing of the GM-3Co/97WC sintered graded tool material	Strategy of a cypress in summer C (4.8, 7.0)	(O)	(5.1, 7.1)	(7.8, 7.3)	(8.2, 8.0)	(8.6, 8.6)
			(P)	(4.1, 6.5)	(3.6, 3.0)	(3.0, 3.4)	(1.4, 1.2)
			(MP)	(5.0, 7.1)	(5.3, 7.1)	(7.7, 7.1)	(8.2, 7.1)

The most probable development variant of the (A) technology, i.e. manufacturing GM-90HSS/10WC with the conventional powder metallurgy method that consumes lots of energy and time as compared to the (C) technology and produces the lower hardness of the surface layer of the manufactured material and worse, other mechanical and functional properties of tools in relation to the (C) technology [26,36,62], assumes that the technology, having its potential limited and its attractiveness diminishing and being forced out by other, more effective technologies, will move in short term (2015) to the cypress in winter field, and will then have been found in 2020 in the weakest field of the matrix of strategies for technologies, i.e. aspen in winter and will be eliminated from the market. The (A) technology development variant, which is unlikely, provides that scientific and research works will be undertaken due to certain properties of the tool produced (e.g. much smaller specimen shrink than for the (C) technology) aimed at strengthening its potential. As a result, the technology will move in 2020 to the oak in summer field and will stay there for the next years of the forecast. A pessimistic

(A) technology development track provides that its attractiveness will fall dramatically with a small potential and will degrade even faster than in the most likely case and transition will be seen to the aspen in winter field already in 2015 and rapid withdrawal from the market.

A graphical example of the (B) technology strategy matrix: of manufacturing GM-75HSS/25WC with the conventional powder metallurgy method with the strategic development tracks applied is presented for the three variants in Fig. 61. The numerical values, being the result of all the investigations pursued for the three analysed groups of technologies, are given in Table 5.

5. Technology Roadmapping

The results of the foresight–materials science research conducted represent reference data serving to create **technology roadmaps** being a comparative analysis tool enabling to choose a technology or a group of technologies which is best for the criterion selected. The roadmaps prepared with a custom concept have their set-up corresponding to the first quarter of the Cartesian system of coordinates. The following time intervals, respectively: current situation (2010-11), goals fulfilment methods (2020) and long-term objectives (2030) are provided on the axis of abscissa. Seven main layers ordered by their hierarchy are provided onto the axis of coordinates of the technology roadmap: time layer, concept layer, product layer, technology layer, spatial layer, staff layer and quantitative layer, made up of more detailed sub-layers. The upper-most layers of the technology roadmap are most general and determine the all-social and economic reasons and causes of the actions taken. The middle layers are characterising a product and its manufacturing technology. The bottom layers are determining organisational and technical matters concerning the place, contractor and costs. The cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow take place between the individual layers and sub-layers as signified graphically with the different types of arrows. Figure 62 presents a technology roadmap made for the (B) technology: manufacturing of GM-75HSS/25WC based on the high-speed steel matrix with 25% volume fraction of the WC reinforcing phase in the surface layer. Table 6 presents an aggregate list containing selected data being an extract from all the technology roadmaps developed under this chapter concerning sintered graded tool materials. **Technology information cards** are detailing out and supplementing technology roadmaps. They contain technical information very helpful in implementing a specific technology in the industrial practice, especially in SMEs lacking the capital allowing to conduct own research in this field.

Table 6. *Selected main source data used for preparation of technology roadmaps for investigated sintered graded tool materials*

Analysed factors	Time interval	Manufacturing of the		
		(A) GM-90HSS/10WC	(B) GM-75HSS/25WC	(C) GM-3Co/97WC
		sintered graded tool materials		
Live cycle period	2010-11 years	Embryonic (10)	Embryonic (10)	Embryonic (10)
	2020 year	Embryonic (10)	Prototype (8)	Prototype (8)
	2030 year	Embryonic (10)	Early mature (6)	Early mature (6)
Machine park modernity	2010-11 years	High (8)	High (8)	Quite high (7)
	2020 year	High (8)	Very high (9)	High (8)
	2030 year	High (8)	Excellent (10)	Very high (9)
Quality and reliability	2010-11 years	Medium (5)	Medium (5)	Medium (5)
	2020 year	Quite low (4)	Quite high (7)	Moderate (6)
	2030 year	Low (3)	Very high (9)	Very high (9)
Proecology	2010-11 years	Medium (5)	Medium (5)	Low (3)
	2020 year	Medium (5)	Moderate (6)	Medium (5)
	2030 year	Medium (5)	Very high (9)	High (8)
Staff education level	2010-11 years	High (8)	High (8)	Very high (9)
	2020 year	Quite high (7)	Quite high (7)	High (8)
	2030 year	Quite high (7)	Medium (5)	Medium (5)
Capital requirements	2010-11 years	Very high (9)	Very high (9)	Very high (9)
	2020 year	High (8)	Quite high (7)	Quite high (7)
	2030 year	High (8)	Medium (5)	Medium (5)
Production size determining profitability in enterprise	2010-11 years	Quite high (7)	Quite high (7)	Very high (9)
	2020 year	Quite high (7)	Medium (5)	Quite high (7)
	2030 year	Quite high (7)	Quite low (4)	Medium (5)
Production size in the country	2010-11 years	Minimal (1)	Minimal (1)	Minimal (1)
	2020 year	Very low (2)	Low (3)	Low (3)
	2030 year	Very low (2)	Medium (5)	Medium (5)
Note: Research results are presented in universal scale of relative state, where:1 is minimal and 10 is excellent level.				

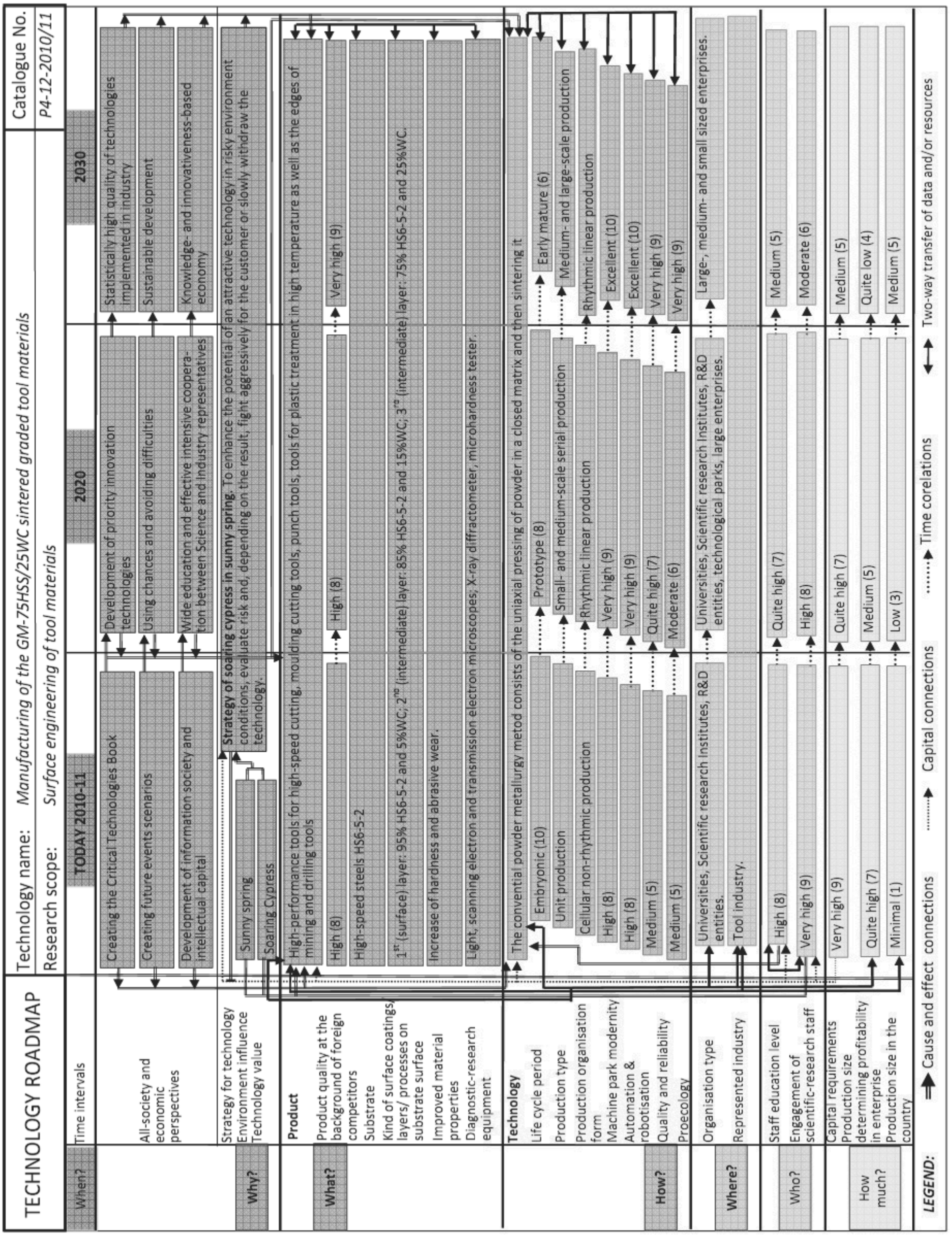

Figure 62. *An example technology roadmap prepared for the (B) technology: Manufacturing of the GM-75HSS/25WC sintered graded tool material*

6. Summary

This chapter presents the results of interdisciplinary experimental and comparative research concerning graded tool materials manufactured with the conventional powder metallurgy method.

Three specific technologies for manufacturing the following graded materials, i.e. GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC are distinguished between for the purpose of the research followed by adopting the matrix material and fraction of powders in the mixture as a selection criterion. The materials science part of the work included in particular investigations into the influence of sintering conditions and the fraction of the reinforcing phase on the properties and structure of GM-HSS/WC, into the influence of heat treatment conditions on the properties and structure of GM-75HSS/25WC and into the structure and following properties of GM-3Co/97WC: density, hardness, toughness, abrasive wear, values of tensile stresses in the surface layer. A strategic position of tool materials surface engineering was identified for the foresight research in relation to surface engineering in general and a position of the powder metallurgy method, allowing to achieve changes to surface layer chemical composition and/or phase composition, in relation to other critical tool materials surface engineering technologies. The chapter also presents the results of the expert assessment of the potential and attractiveness in relation to the macro- and micro-environment of the three specific technologies of manufacturing GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method and action strategies for the technologies were formulated as well as forecast strategic development tracks determined. The results of the investigations are presented graphically using a set of matrices. Technology roadmaps were prepared at the final stage of the works illustrating, in a concise manner, basic information on the technologies analysed. The analysis made has revealed that the (B) and (C) technologies of manufacturing, respectively: GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method are characterised by moderate development prospects evaluated by the experts with 6 points in the ten-degree scale. According to the expert assessment, the development prospects of the (A) technology are medium (5 points). This severe score is affected by a high uncertainty with regard to internal factors including the necessity to continue scientific and research works to strengthen the potential of experimental technologies with high attractiveness as well as to external factors such as intensively evolving alternative technologies of manufacturing graded tool materials in the laser treatment, Physical Vapour Deposition and hybrid (PVD and thermochemical treatment) processes.

A surface layer can be provided with high resistance to abrasive wear while maintaining high core ductility at relatively low costs if the powder metallurgy method is applied. Such structure of the material allows to develop its properties freely depending on the working conditions of the tool. Hard surface layers can be used for example in the locations exposed to wear, and a ductile core can be used in other locations exposed to impact. It is predicted that further scientific and research work should support the future wide industrial applications of the technologies analysed since the technologies have been used at a laboratory and semi-technical scale to date [64]. The advantages of manufacturing the sintered graded tool materials in industrial conditions include waste-free production and a short production cycle and a constraint is a possibility of manufacturing small-sized products with their shape determined with the construction parameters of dies. The specific properties of sintered graded tool materials make them suitable, notably, for use as high-performance tools for high-speed cutting, moulding cutting tools, punch tools, tools for plastic treatment in high temperature as well as the edges of mining and drilling tools.

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12 Development perspectives of selected technologies of polymer surface layers modification

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Abstract

Purpose: *The purpose of the chapter is to present the results of a comparative analysis of the five selected technologies of modification of polymer surface layers based on the outcomes of own materials science and foresight research. A criterion of dividing is represented by physical processes the result of which is polymer material surface layer modification.*

Design/methodology/approach: *This chapter presents the results of own materials science research of surface modification for selected polymer materials subjected to the activity of corona discharges, laser light and high-temperature electron-beam irradiation. The technologies analysed include also modification with low-temperature plasma generated in the air by a generator situated outside the material modification zone and with low-temperature plasma in the conditions of low pressure (0.05-5 hPa). The value of the individual technologies as well as the type and intensity of environment influence was assessed in the course of foresight works. The results of the research are provided graphically with a bar chart, pool of context matrices, strategic development tracks and technology roadmaps.*

Findings: *An important role of polymer surface layers modification technologies for the development of overall materials surface engineering was pointed out.*

Research limitations/implications: *The research pursued represents part of a larger project aimed at identifying and characterising the priority, innovative materials surface engineering technologies.*

Originality/value: *The value of this chapter is represented by an original contribution consisting of objectivised assessment of the selected polymer materials surface layers modification technologies and presenting the directions of their strategic development against the overall materials surface engineering based on the results of foresight- materials science research.*

Keywords: *Manufacturing and processing; Surface treatment; Polymers; Foresight; Technology Roadmapping*

1. Introduction

The European Union's priority strategy set out in the recent years called Europe 2020 assumes that the development of the continent should be intelligent, supportive to social inclusion and sustainable [1]. For this to be feasible, it is necessary to take extensive actions at the European, national and regional level, to support a more effective, competitive and low-emission economy based on knowledge and innovation, ensuring high employment and social and territorial cohesion, which is expressed by five strategic objectives (Fig. 1). Such objectives should also be achieved under a project on establishing the innovation Union, promoting innovation understood as precious, innovative ideas [2]. A stream of investments, therefore, should be channelled into those fields of science and industries bringing the highest added value. Special consideration should be given to small- and medium-sized enterprises encompassing 99.8% of all domestic companies and generating 68% of GDP. The aim of the foresight research conducted broadly in the recent decade in Europe and Poland, also in the field of materials science [3-9], is a quest for innovative areas deserving financial support. Technology foresight serving to identify the priority, innovative technologies and the directions of their strategic development was pursued also for materials surface engineering [10]. One of the 14 thematic areas analysed under such foresight research is the surface engineering of polymer surface layers.

The properties of surface layers (SL) of polymer materials including composites [11-14] are gaining increasing importance as the rapid growth of diverse applications of such materials has been seen [15, 16]. The formation of properties of materials' SL is the task of technology design, which, together with structural design and material design, represent three equivalent and inseparable elements of engineering design [17, 18]. The majority of polymer materials exhibit low reactivity, are hydrophobic by their nature and are characterised by low surface free energy (SFE) [19, 20]. Such characteristics to a large extent are limiting the adhesive properties of such materials and of polymeric and metallic layers deposited. Different physical methods of modifying such materials' SL are used to improve the adhesive properties of polymer materials, notably such as corona discharges, plasma method, laser method and electron-beam irradiation. The methods consist primarily in initiating reactions causing the formation of polar functional groups in this layer and in changing the geometric structure of the modified material's surface [21, 22]. The polymer materials SL modification methods analysed in this chapter permit to eliminate chemical products hazardous for the environment used in the methods of chemical modification of such layer.

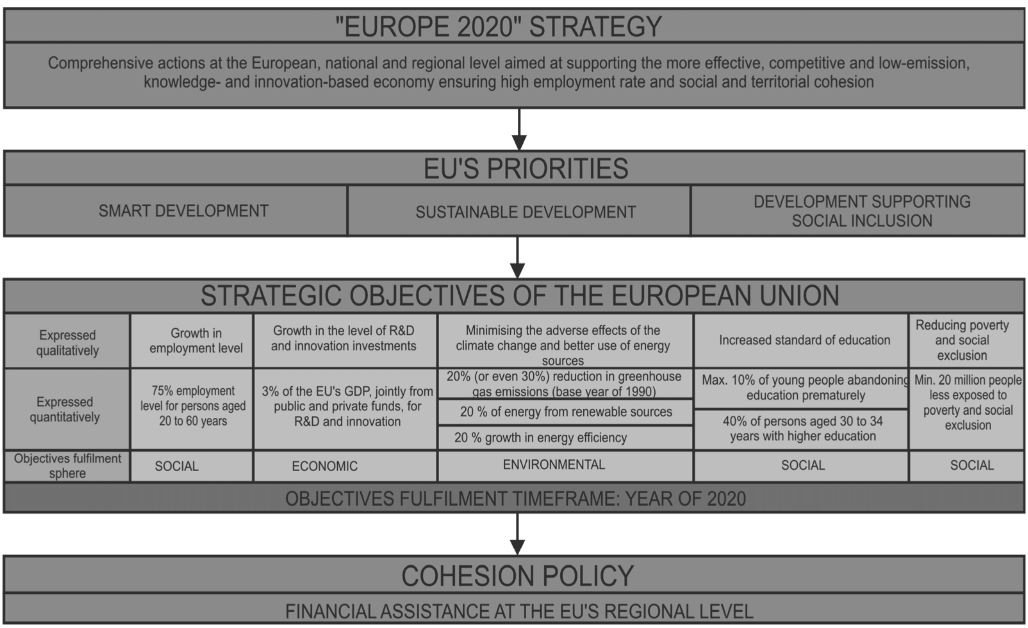


Figure 1. *EU main strategy, priorities and objectives. Custom presentation prepared on the basis of [1]*

2. Research scope and subject matter

The research efforts focussed on the selected technologies of modifying polymeric surface layers are of an interdisciplinary character. The foresight-materials science research methodology [23, 24] applied concerns technology foresight being part of the field of knowledge called organisation and management and of surface engineering forming part of the widely-understood materials science. A much broader prospective into the issues investigated had to be used at some stages, however. For this reason, methods and approaches deriving from other areas of specific knowledge had to be employed, i.e. computer science including information technology [25] and artificial intelligence [26] (neural networks joining with Monte Carlo methods); statistics; econometrics; operational studies; machine construction and operation; automation and robotisation of industrial processes; strategic, tactical and operational management; quality and environment management; accounting and finance. The following five homogenous groups were distinguished between for the purpose of the foresight and materials science investigations carried out under this task by adopting, as a criterion of grouping, physical processes the result of which is polymer material surface layer modification, i.e. respectively:

(A) Corona Treatment (CT);
(B) Remote Plasma Treatment (RPT);
(C) Low Pressure Plasma Treatment (LPTT);
(D) Laser Treatment (LT);
(E) Electron-Beam Irradiation (EBI).

3. Technologies descriptions, adopted methodology and materials science research results

This chapter provides an overview of five, selected technologies of polymeric surface layers modification induced by various physical processes. The methodology adopted and the results of own research in this area by means of corona discharges, laser light and electron-beam irradiation are also presented in relation to modification.

3.1. Corona Treatment

The corona treatment (CT) methods were described for the first time in patents [27-29], and the first industrial applications, primarily for modifying the SL of polyolefine foils, date back to the 60's of the last century. The current applications of the method are much more extensive and encompass not only flat plastic products such as foils and plates but also products with complex shapes (e.g. tubes and bottles) [30]. For this reason the CT method has become the most popular method of industrial SL modification of plastics.

Corona discharges occur due to differential potentials created between two activator electrodes, i.e. an HV discharge electrode and an earthed electrode in a space filled with gas (typically air) under atmospheric pressure [31]. The production of plasma is initiated by electrons staying within an interelectrode space. The electrodes are highly accelerated under the influence of an electromagnetic field. The electrons, while moving in an interelectrode space, collide with air molecules, causing their ionisation and hence an increase in the number of electrons and ions. Electric current starts to flow in the interelectrode space as a result of such phenomena. A stream of plasma falling on the SL of a product removes contaminations and changes the geometric structure of the surface. The molecules within plasma have a different kinetic energy. The most of the electrons in the interelectrode space of the activator have their kinetic energy of approx. 10 eV. The kinetic energy of electrons is higher than the energy of the basic bonds occurring in the macromolecules of polymer chains (e.g. energy of C - C, C - H, C - N bonds is smaller than 5 eV). The impact of molecules, atoms and ions on changes to the SL of the modified material is much smaller in the plasma generated during corona discharges than the impact of electrons [32]. Some chemical bonds of polymer chains are knocked out due to the kinetic energy of the electrons hitting against polymer chains. Radicals are formed that initiate chemical reactions modifying the SL of products (oxidisation processes are mainly at play). The radicals react with, in particular, oxygen, ozone, OH groups, water molecules, by forming polar compounds. The polar compounds forming in the SL as a result of chemical reactions, during which oxygen decomposition occurs, change the surface properties of the products modified, including the growth of SFE [33-34].

The subject matter of the own research was concerned with polyactide (PLA) 2002 D by Cargill Down LLC with the melt flow index of 4.2 g per 10 min. (2.16 kg, 190^{o}C) and density of d = 1.24 g/cm^3, in form of approx. 100 μm thick foil. An AF2 foil activator (Metalchem, Toruń) was used for modifying the surface layer of the PLA. The tested PLA samples were subjected to the activity of CT at ambient temperature (approx. 23ºC) under atmospheric pressure in the air. The surface layer of PLA was modified using a 0.25 m long, HV, single-point electrode. The parameters of the modification process are shown in Table 1.

Table 1. *Modification process parameters of the PLA surface layer, (E_j – unit energy of modification, P – capacity of corona discharges, v – foil displacement rate)*

E_j, kJ/m^2	P, W	v, m/min
0.5	200	96
1	400	96
1.5	400	64
2	400	48
3.5	400	27.4
5	400	19.2
7	400	13.7
10	400	9.6
20	400	4.8

The values of water (Akchem, Poland) and diiodomethane wetting angle (Sigma - Aldrich, Germany) for the PLA samples modified with the CT method in the air are shown in Fig. 2. As E_j is growing, the values of water or diiodomethane wetting angles are declining, whereas larger changes are seen for water. This is caused by the fact that polar forces take part in intermolecular interactions more intensively. Much higher growth in the interactions of polar liquid than dispersion liquid with modified PLA foil derives from the fact that polar groups are created in the surface layer of PLA that strongly interact with water molecules as a consequence of the modification process. Interactions of a dispersive character are of smaller importance here.

The surface free energy (SFE) of the tested samples was calculated with the Owens – Wendt method. Fig. 3 shows the values of a polar component (γ_s^p) and dispersive component (γ_s^d) of SFE and the result of SFE calculations for the samples modified in the air. The growth of the E_j value is accompanied by the growth of SFE of the PLA samples modified in the air. Growth in SFE is caused mainly by an increase in the polar component as polar groups are created strongly interacting with water molecules and improving wettability.

Investigations into the oxidisation degree of the surface layer of PLA were performed with X-ray photoelectron spectroscopy (XPS) using the Escalab 210 electron spectrophotometer

(VG Scientific, UK). An oxidisation degree (O/C) determined as a quotient of the number of oxygen and carbon atoms present in the tested SL (in percents) is shown in Fig. 4. As E_j increases, so increase the O/C values of the surface layer of PLA during modification in the air. Growth in O/C is a result of the higher share of oxygen groups the creation of which is initiated by free radicals reacting with oxygen from the environment and generated under the influence of energy of electrons colliding with the material being modified.

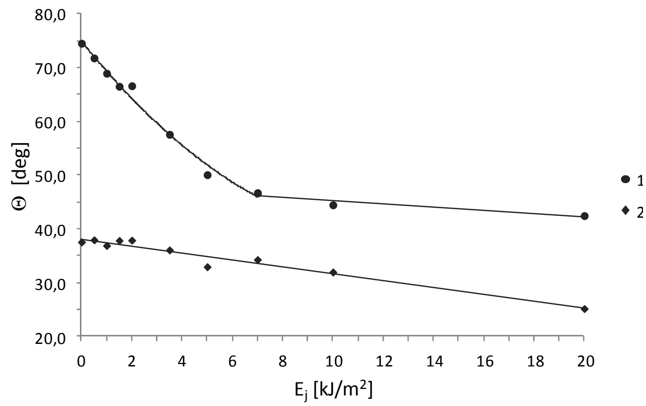


Figure 2. *Impact of unit value of modification energy (E_j) on water (θ_w) (1) or diiodomethane (2) wetting angle (θ_d) of samples modified in air*

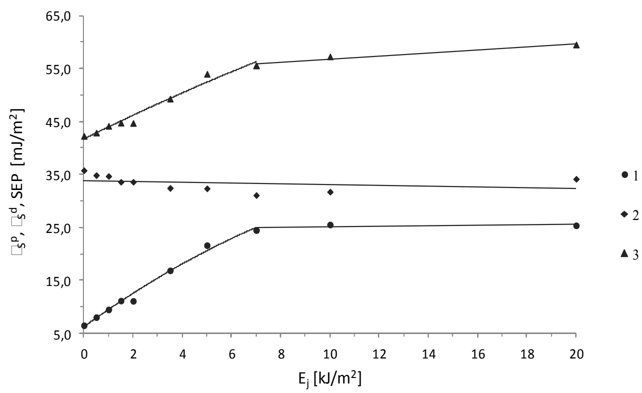


Figure 3. *Effect of unit value of modification energy (E_j) in the air of samples tested according to: (1) a polar component of surface free energy (SFE), (2) a dispersive component of SFE and (3) SFE values*

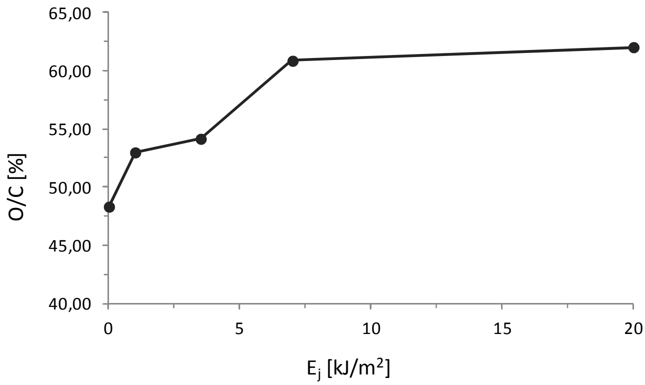


Figure 4. *Oxidisation degree (O/C) of the SL of the PLA samples modified in the air*

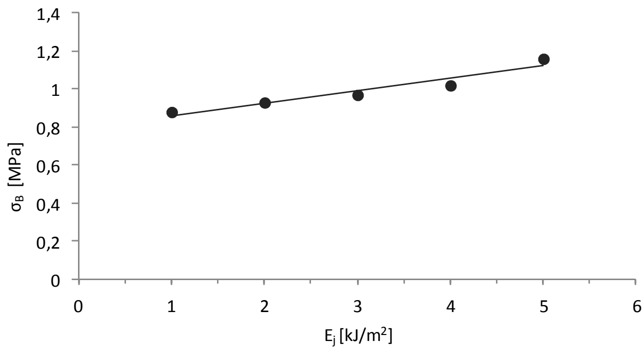


Figure 5. *Peel stress (σ_B) for joints of the PLA sample - Rapida F50RP paint modified in the air*

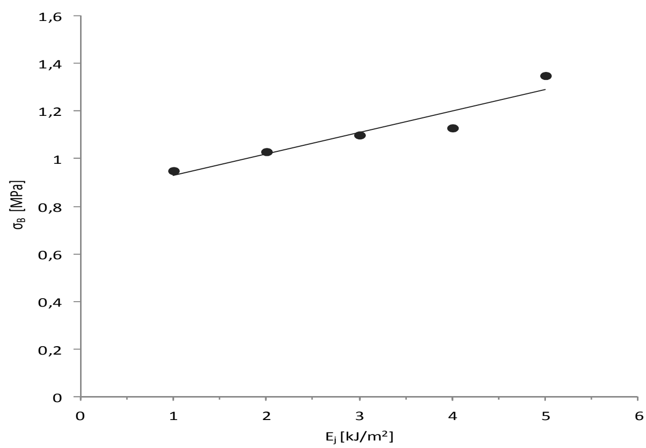


Figure 6. *Peel stress (σ_B) for joints of the PLA sample - Acrylic Enamel 2750 paint modified in the air*

Adhesion investigations were carried out with the method of detaching an adhesive joint by means of the PosiTest AT device and with the method of detaching with adhesive tape. Rapida F50RP paint (Michael Huber, Poland) and Acrylic Enamel 2750 (Bondex, Poland) paint and Araldite 2011 epoxy glue (Huntsman, Switzerland) were used for the test. The results of the investigations are presented in Figs. 5 and 6. Twelve peel stress measurements were made for each E_j value. Two extreme values were rejected and the arithmetic mean of the other ten results were used as the result. Paint was completely peeled off the tested samples at the entire area of the glued measuring stamp during the tests in both cases. The adhesive joints were fracturing between the paint and the tested substrate in all the tested samples. The strength tests of the PLA–paint joint with a method of peeling off with adhesive tapes did not exhibit any damaged printing after detaching the tape, nor any traces of paint on such tapes. This indicates that the adhesion of the tested samples and paint meets the requirements for printing technology and that the strength of their adhesive joints improves under the influence of CT.

3.2. Remote Plasma Treatment

The method of generating plasma outside the modification zone, as opposed to the corona treatment method, consists in modifying a polymer material outside a plasma generation zone. A device for modification with this method is shown in Fig. 7. High voltage and process gas are supplied to a unit of discharge electrodes with flexible conduits, whereas air may also be used as the gas. A stream of gas removes active plasma molecules produced during partial discharges and diverts the molecules onto the surface of the material being modified through a specially shaped nozzle. A special advantage of such devices is the ease of generating plasma with different properties, using different gases. The devices can also be constructed as small portable devices (used mainly for modifying items with complex shapes), including hand-held devices, and also devices mounted in the holders of robots. There are many constructional variants of such devices.

3.3. Low Pressure Plasma Treatment

Plasma is a partially ionised gas or a mixture of gases composed of the equal (approximately) number of electrons and neutral atoms and particles of relevant gases, as well as photons of electromagnetic radiation. Ions, atoms and neutral particles can occur in excited and/or basic conditions. In the natural conditions of the Earth plasma is produced during atmospheric discharges, and it is the most common state of matter in the universe, distinct, however, from the low-temperature plasma discussed here by its composition and temperature. The plasma existing there is called high-temperature (hot) plasma as its temperature is around 10^6 K, and its particles are completely ionised. On the other hand, the temperature of cold, low-temperature plasma discussed in this chapter is lower than 10^4 K and is generated during electrical discharges in gas [32, 35-39].

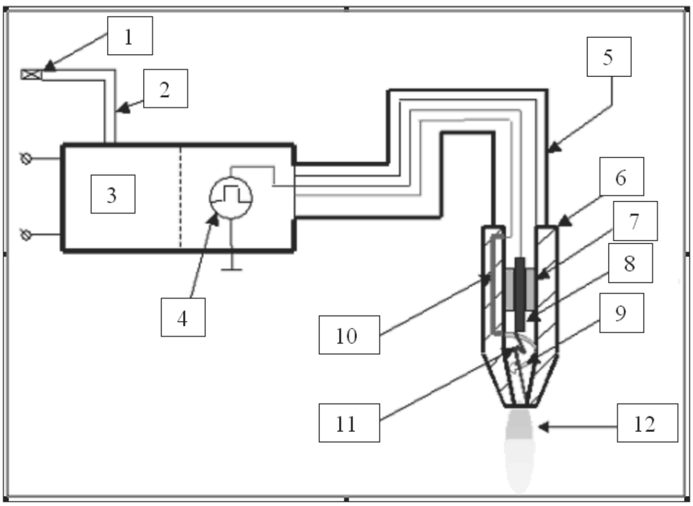


Figure 7. *Diagram of a plasma generating device outside the modification zone (1 - gas connection, 2 - gas supply, 3 - electricity and gas supply device, 4 - voltage generating set for supplying electrodes, 5 - flexible sheathing, 6 - earthed electrode, 7 - dielectric insulation, 8 - high voltage electrode, 9 - gas outlet, 10 - gas inlet, 11 - partial discharges, 12 - stream of plasma)*

Low-temperature plasma is formed under the influence of partial discharges taking place in a vacuum chamber where two electrodes are provided. The discharges are generated by a rapidly changing magnetic field in gas, most often in oxygen, nitrogen, helium, argon, chlorine and in the air [40, 41]. Low-temperature plasma, with its temperature lower than 10^4 K, interacts with the product being modified between several dozens of seconds to more than ten minutes. The pressure of gas in a vacuum chamber, called a discharge chamber, is low and is between 0.05 to 5 hPa. The average temperature does not exceed ambient temperature, whereas a concentration of electrons in plasma is between 10^{16} to 10^{18} electrons per cubic cm. A diagram of a device for modifying the SL of materials with plasma under a lower pressure is shown in Fig. 8.

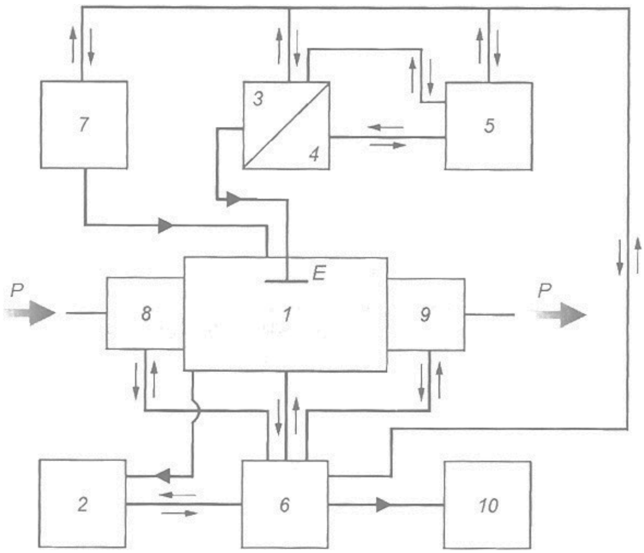


Figure 8. *Block diagram of a device for modifying the SL of materials: 1 – discharge chamber, 2 – unit generating lower pressure, 3 – generating set supplying discharge electrodes, 4 – impedance adjusting device, 5 – power control unit, 6 – automatic monitoring and control circuit, 7 – gas tank, 8 – supply unit, 9 – discharge unit, 10 – screen and printer, E – discharge electrode, P – stream of modified material [42]*

Plasma is generated according to the following principle: a rapidly changing electromagnetic field generated between two electrodes placed in a vacuum chamber induces the vibrations of the molecules present in the inter-electrode area. The vibrations are accompanied by such processes as: ionisation, dissociation and excitation of molecules and atoms of gas. Electrons in an electromagnetic field gain much higher speeds than ions, atoms or gas molecules (as their mass is much lower) and, therefore, their kinetic energy is higher. The kinetic energy of the molecules of gas, atoms and ions reaches the values of around 10^{-2} eV and is between 10^2 to 10^4 times lower than the kinetic energy of electrons. For this reason such energy is not of any major significance in the plasma generation process and does not cause marked changes to the SL of materials. A kinetic energy of electrons is usually between several to more than ten electronovolts and is sufficient for breaking convalescent bonds in polymer micromolecules. This leads to changes in the SL of the material and to the development of partial discharges [43]. The following processes occur while modifying the SL of materials with low-temperature plasma [44]:

- impurities are removed from the surface and SL of the material. The operating time necessary for plasma to clean the surface of material is tens of seconds, and unit power required for this purpose is several dozens of W/m^2;
- SL etching to remove part of the material (so-called weak boundary layer) and pieces of the amorphous phase. This process is especially important when glued joints are made, as surface roughness is increased as a result, thus the contact surface area between the material and paint or glue. A system with high adhesive strength is next created after anchoring them in the substrate;
- netting of macromolecules in the SL contributing to improved adhesive strength of two-phase systems. A structure strongly attached to the substrate is then formed and resistant to the activity of thermal energy, featuring high mechanical strength and inhibiting the breaking of adhesive joints;
- new chemical structures are created, in particular ketone, aldehyde, hydroxyl and carboxyl groups, under the influence of free radicals, oxygen and water steam. The groups change the chemical properties of the SL. In addition, plasma polymerisation can occur as a result of which a thin, insoluble, strongly netted layer of polymer is produced, acting as a good barrier for gases and liquids. The properties of the layer depend to a large extent on the type of gas in a discharge chamber.

The essential conditions of the SL modification process of materials by means of low-temperature plasma: (1) the time plasma acts on the material, (2) unit energy of partial discharges (J/m^2) and unit power of partial discharges (W/m^2), (3) temperature, pressure and type of gas in a discharge chamber, (4) electric voltage and frequency of partial discharges, (5) temperature of the modified material, (6) discharge chamber dimensions.

3.4. Laser Treatment

A group of the latest devices enabling to modify the SL of polymer materials is represented by lasers. Their special advantages can be used for the very accurate modification of small areas with complex shapes occurring for example when producing modern electronic circuits.

A separate area of applications for laser modification can be polymer materials used in medicine, where sterilisation is also needed apart from changing the properties of the SL. As opposed to the methods of modifying the SL of polymer materials known to date, however, laser modification is a new method, still in the phase of basic research [45]. The laser modification of the SL of polymer materials allows to change precisely different properties of this layer, mainly such as: wettability and SFE (by implementing polar function groups, chiefly in the oxidisation process), degree of polymer netting and the type of the geometric structure of the surface, without changing at the same time the properties of the material underneath this layer [46]. The laser modification process is simple, easy to control and environmentally secure. The physiochemical phenomena associated with the process are not fully identified, yet and intensive scientific research into this area is continued [47-50]. Considering the many types of lasers available at the market, excimer lasers are characterised by advantageous properties due to modification to the SL of polymers [51]. Excimer lasers operate within the range of ultraviolet radiation with short-lived impulses. This radiation is strongly absorbed by the majority of polymers. Ultraviolet radiation with the short duration of impulses is able to initiate photochemical reactions in a polymer material without causing heat damage to the material. Laser irradiation can be carried out using an energy higher or smaller than the energy of the ablation threshold of a polymer material. Radiation with an energy not causing material ablation is used to improve adhesive properties [52-54]. Table 2 presents our tests of wettability and free surface energy of samples made of PC (Lexan 143 R, GE Plastics, USA), PET (Elpet-A, Boryszew SA, Poland) and PS (Owispol 945 E, Dwory S.A. Oświęcim, Poland), whereas Fig. 9 presents changes to their geometric structure caused by radiation with the different number of ArF laser impulses.

An oxidisation degree (O/C) of the surface layer of polycarbonate (PC), poly(ethyl terephthalate) (PET) and polyesterine (PS) irradiated with the different number (N) of laser impulses is presented in Fig. 10. The degree of oxidisation is growing in all the tested samples. The character of such changes is similar in PET and PC, which can be described with exponential dependencies. Increase of O/C in PC is higher than in PET and the saturation level is reached faster. Increase in the value of O/C in PS is approximately linear.

Very intensive changes to the chemical and geometric structure of the irradiated polymer materials can be induced with an energy of laser impulses higher than the ablation threshold, and chemical reactions of additional components occurring in such materials can be initiated.

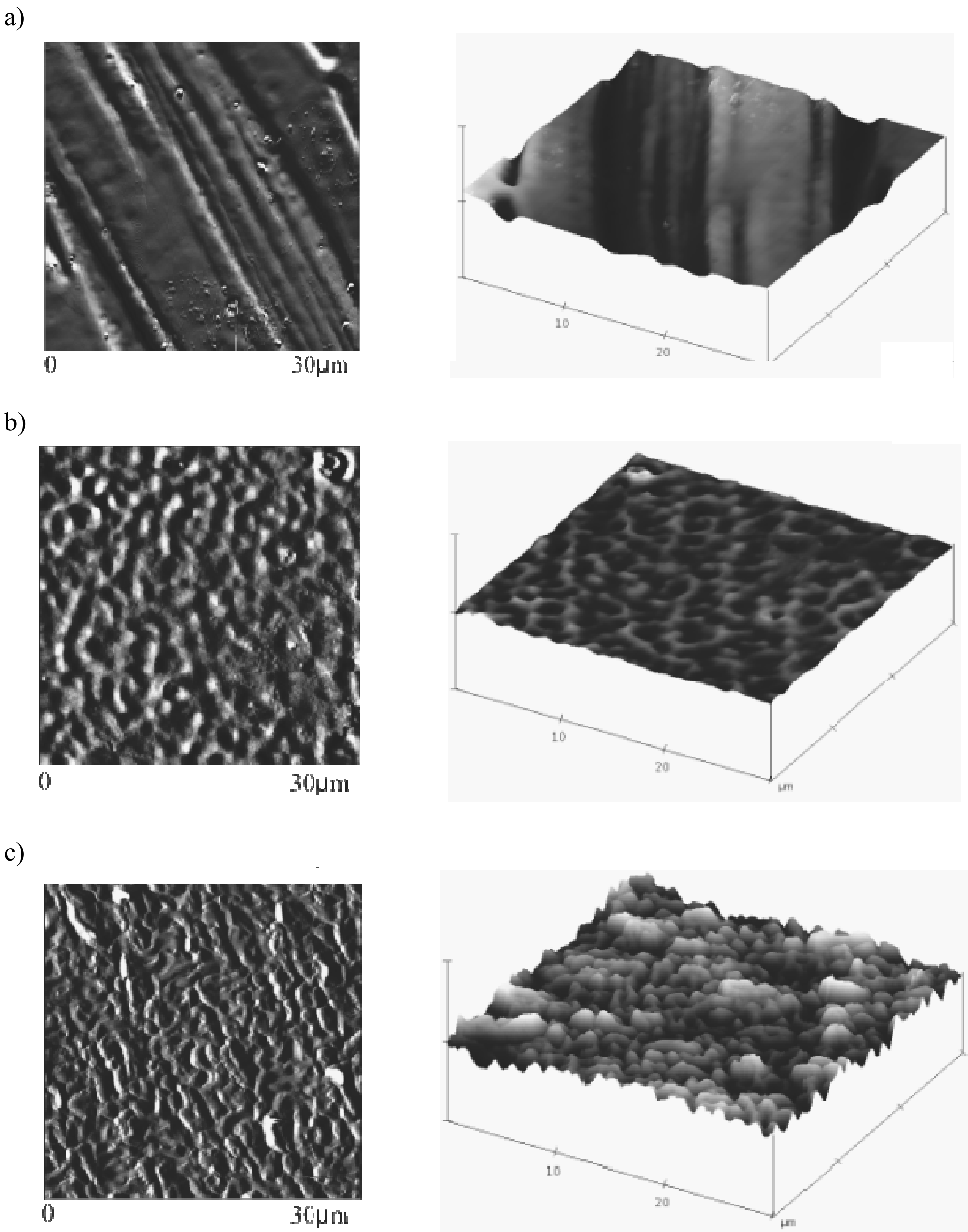


Figure 9. *Two- and three-dimensional images of samples surface: a) PC; b) PET; c) PS, irradiated with 2000 impulses of laser (E_j=6 mJ/cm^2)*

Such method of laser modification is used for electroless metallisation of polymer materials and enables to create differently shaped conductor tracks. Thermal degradation of polymer material is seen as a result of laser radiation acting on the surface and surface layer of the polymer material containing an appropriate precursor and metal clusters are produced (the source of which is the precursor), initiating and catalysing the autocatalytic process of such material's metallisation [55, 56].

Table 2. *Angle values of wetting with water (□$_w$), with diiodomethane (□$_d$), and surface free energy (SFE) of polycarbonate PC, poly(ethyl terephthalate) PET and polyesterine PS irradiated with the different number (N) of laser impulses with Ej = 6 mJ/cm^2*

N	□$_w$	□$_d$	SEP	Material
0	91.2	43.0	38.2	PC
10	90.1	42.1	38.7	
100	77.9	40.9	40.1	
500	74.1	34.2	43.8	
1000	73.3	33.5	44.4	
2000	67.1	33.1	46.2	
0	79.1	50.3	35.8	PET
10	78.6	50.0	36.0	
100	78.0	47.1	37.8	
500	74.4	43.3	40.3	
1000	70.1	36.1	44.2	
2000	66.9	32.9	46.2	
0	104.0	77.2	19.1	PS
10	101.1	71.0	22.3	
100	100.3	66.9	24.6	
500	103.9	33.2	46.8	
1000	103.3	33.0	46.8	
2000	107.0	29.4	50.9	

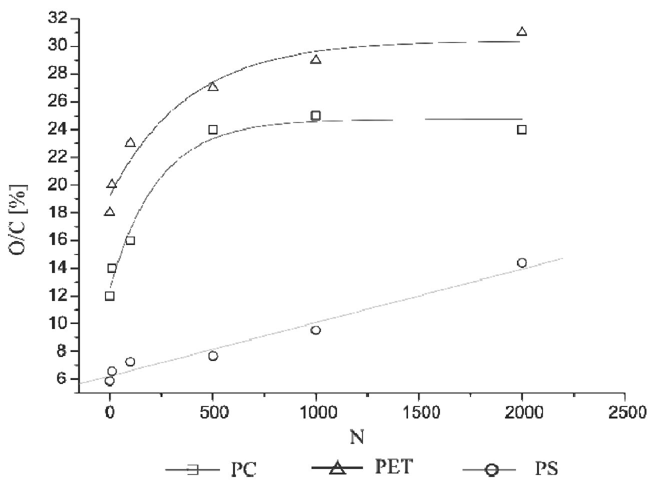


Figure 10. *O/C dependence of the surface layer of PC, PET and PS samples irradiated using the different number of laser impulses (E_j = 6 mJ/cm^2)*

While seeking new polymer materials for the autocatalytic metallisation, metallisation precursors (other than used so far) were introduced as part of own works at the stage of polymer material processing. A new polymer composite has been established as a result of the research with a polyamide 6 matrix containing two metallisation precursors: copper acetylacetonate (II) $Cu(acac)_2$ and copper oxide (II) CuO. Excimer laser ArF (λ = 193 nm) was used to modify the SL and activate the surface of a new composite. Impulses with the unit energy (E_j) of irradiation of, respectively, E_{j1} = 40 mJ/cm^2 or E_{j2} = 120 mJ/cm^2 were used when modifying such composite. The composite was modified with the different number of impulses of: 5, 10, 50, 100 and 500.

SEM investigations revealed that the surface of the composite is undergoing marked changes as a result of laser modification. Fig. 11 shows a clear boundary of laser impulses activity. An unmodified surface of the sample is characterised by small roughness with visible scratches being a replica of the injection moulding walls (Fig. 12a). The surface of the irradiated sample is largely modified. Any surface irregularities being a replica of the injection moulding surface disappear and new cone-like structures typical for the ablation process are formed (Fig. 12b).

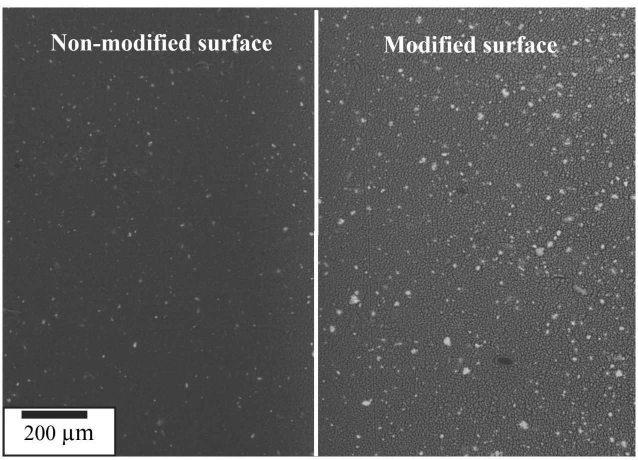


***Figure 11.** SEM image of the surface of the new composite not irradiated and irradiated with 500 impulses of laser with the energy of 120 mJ/cm^2*

A thickness of the modified SL depends on the number of laser impulses (Fig. 13). Growth in the number of impulses is leading to the deeper ablation of the SL of a new composite.

A thickness of the modified layer can be estimated based on the images of fractures of the samples. The thickness for 50 laser impulses is approx. 4 µm, whereas for 500 impulses it is approx. 25 µm. The comparison shows that if the number of impulses is increased, this has a decisive effect on increasing the thickness of the modified layer and on increasing surface roughness.

a)

b)

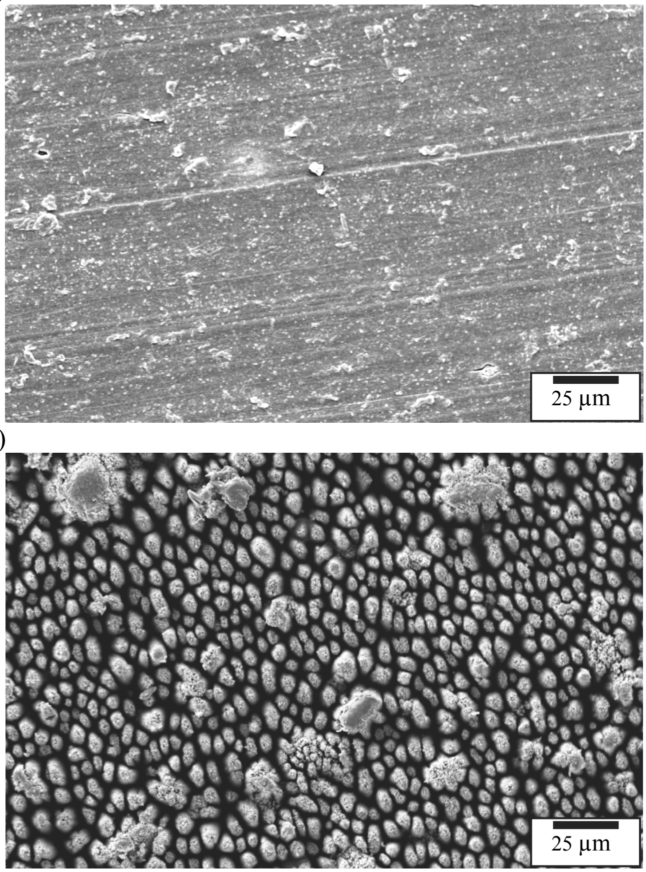


Figure 12. *SEM image of a) unmodified and b) modified surface of new composite*

The laser irradiation of a composite developed contributes to the decomposition of metallisation precursors and metallic copper clusters are produced. An image of a quantitative EDS analysis of the new composite (Fig. 14) confirms this process. Copper atoms contained in the SL of the new composite initiate and catalyse its metallisation process. This enables to deposit a layer of metallic copper on the surface of this composite, and such layer is deposited already after 5 laser impulses (Fig. 15).

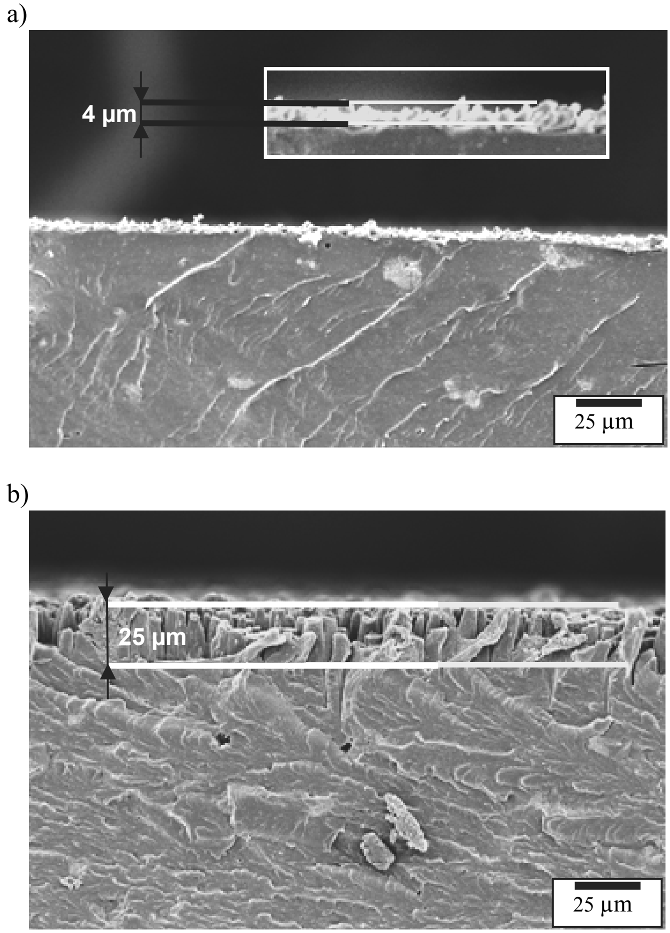


Figure 13. *SEM images of fractures of the new modified composite: a) 50 or b) 500 laser impulses with the energy of 120 mJ/cm^2*

The further advancement of laser SL modification methods for the purpose of polymer materials metallisation will contribute to the miniaturisation of electronic components. Functional components that may be designed and produced using laser methods will reduce the number of individual parts and shorten their manufacturing process.

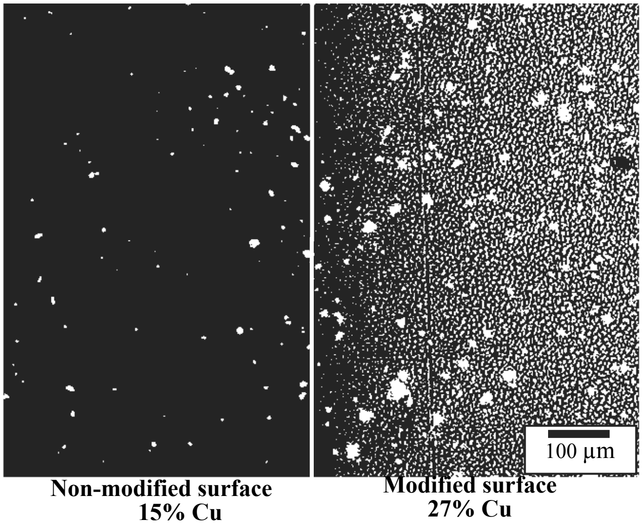


Figure 14. *Image of a quantitative analysis of Cu atoms content in the surface layer of a new composite partially irradiated with 500 laser impulses with energy of 120 mJ/cm*^{2} *(line marks a laser impulses activity boundary)*

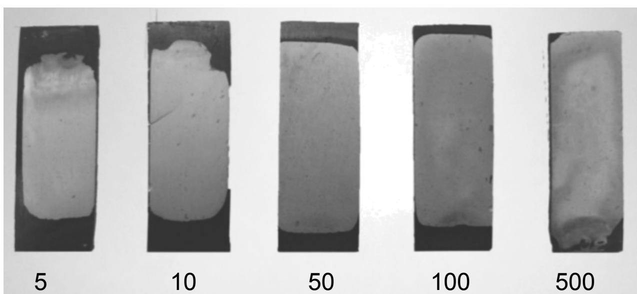


Figure 15. *Effects of metallising a new composite irradiated with the unit energy of 120 mJ/cm*^{2} *and with the different number of impulses (the number of impulses is identified with a digit under each of the samples presented). Scale 1:1*

3.5. Electron-Beam Irradiation

Irradiation has been used for over 50 years for modifying the volumetric and surface properties of polymer materials [57-60]. The impact of irradiation, mainly electron-beam one, on changes to the surface properties of polymer materials was a subject of extensive scientific research [61, 62]. When a polymer material is irradiated, its SL is oxidised in the air and this increases wettability and growth of the SFE, thus improving the adhesive properties of this material. Such irradiation can also be performed in the atmosphere of reactive gases, in particular such as O_2 or NH_3, to produce polar functional groups in the surface layer of the modified material.

The subject of the research presented in [62] were composites differing both, in the composition of polymers used for manufacturing them as well as in mass fraction of compatibilisers. Low density polyethylene (LDPE) Malen-E FABS 23-D0022 (Basell Orlen Polyolefins Płock, Poland), high density polyethylene (HDPE) Hostalen ACP 5831 D (Basell Orlen Polyolefins Płock, Poland), isotactic polypropylene (PP) Malen P F 401 (Basell Orlen Polyolefins Płock, Poland), polystyrene (PS) Owispol 945 E (Dwory S.A. Oświęcim, Poland), amorphous poly(ethylene terephthalate), (PET) Elpet-A, (Boryszew SA, Oddział Elana Toruń, Poland) and a compatibiliser in form of trimethylolpropane triacrylate (TMPTA) (Sigma Aldrich, Germany) were used for producing them. 2 types of composites were obtained by extruding such polymers: (a) a three-component composite, mass fraction of 33.4% LDPE, 33.3% HDPE and 33.3% PP, hereinafter called composite A, containing, respectively, 1% (A1), 2% (A2) or 3% (A3) by weight of TMPTA and (b) a five-component composite, mass fraction of 24% of LDPE, 23% HDPE, 21% PP 15% PS and 17% PET, hereinafter called a composite B containing, respectively, 1% (B1), 2% (B2) or 3% (B3) by weight of TMPTA.

The tested samples were irradiated at the Institute of Nuclear Chemistry and Technology, Warsaw, using an UELW-101-10 accelerator (NPO TORYJ, Russia). The samples of the mixtures produced were placed on a conveyor moving with a finely adjusted speed. A dose of electron irradiation absorbed by the samples was dependent on such speed. The samples were radiated with the doses of 25, 50, 100 and 300 kGy. The values of the doses were controlled with the calorimetric method. The average values of wetting angles for particular components are presented in Table 3. The data presented shows that that a wetting angle is decreasing monotonically as the irradiation doses of the tested components are growing. The effect of an electron irradiation dose on changes to the SFE of particular components is shown in Table 4. SFE changes occurring under the influence of electron irradiation are similar for all components. The dose is growing monotonically within the whole range of the doses applied, with the highest increase seen within the range of doses up to 50 kGy, and the slowest increase within the range of 50 to 100 kGy, and much slower for doses above 100 kGy. An increase in wettability and SFE occurring due to electron irradiation is caused mainly by the implementation of polar oxygen groups in the surface layer of the radiated composites. Table 5 provides the oxidisation degree values of the investigated composites, irradiated with the different doses of energy. The data shown in Table 5 provides that the TMPTA compatibiliser is substantially influencing the growth of the oxidisation degree of the SL of the irradiated composites. This influence derives from the chemical structure of TMPTA containing double bonds that are breaking under the influence of electron irradiation. The radicals produced as a result react with the oxygen present in the SL and increase the oxidisation degree of this layer.

Table 3. *Value of wetting angle with water of composites irradiated with different doses of energy*

Type of composite	Irradiation dose, kGy				
	0	25	50	100	300
A	106.6	102.4	97.3	90.8	87.3
A1	105.7	95.2	85.1	80.9	78.1
A2	104.2	86.8	84.0	80.8	77.4
A3	101.2	84.0	82.2	71.1	66.9
B	104.0	101.3			
B1	108.5	94.1	92.2	87.6	82.5
B2	105.3	91.5	85.8	86.5	83.4
B3	104.5	96.0	89.1	84.4	80.2

Table 4. *SFE values (mJ/m^2) of composites irradiated with different doses of energy*

Type of composite	Irradiation dose, kGy				
	0	25	50	100	300
A	19.1	21.4	24.5	28.4	30.4
A1	20.2	30.6	32.1	34.0	36.0
A2	19.6	30.6	31.8	34.3	36.3
A3	22.6	32.3	33.7	40.2	42.5
B	20.7	22.3	26.4	27.6	29.7
B1	17.8	26.4	27.7	30.4	33.6
B2	19.9	28.3	31.7	31.3	33.1
B3	20.2	25.2	29.4	32.7	35.1

Table 5. *O/C (%) values of composites irradiated with different doses of energy*

Type of composite	Irradiation dose, kGy				
	0	25	50	100	300
A	0.3	1.6	2.0	4.3	7.3
B	1.9	3.9	4.3	5.2	6.4
A2	1.7	5.1	10.1	11.6	19.0
B2	3.9	5.7	7.1	8.4	11.2

4. Development perspectives of polymer surface layers modification

4.1. Polymer surface layers modification versus surface engineering development

The anticipated development and strategic position of polymer surface layers modification technologies against surface materials engineering was determined using the reference data acquired whilst performing technology foresight for materials surface engineering [23, 10]. Over 300 independent domestic and foreign experts representing scientific, business and public administration circles have taken part in the foresight at different stages of the efforts. The experts have completed approx. 650 multi-question surveys and held thematic discussions during 10 expert panels. An analysis of development prospects has been performed in the initial phase of research for approx. 500 groups of detailed technologies including a state-of-the-art review, technological review and a strategic analysis with integrated methods. The following scientific and research methods were used for this purpose: trends extrapolation, environment scanning, STEEP analysis, SWOT analysis, expert panels, brainstorming, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and statistical analysis. 10 critical technologies were selected in 14 thematic areas as a result of the works performed. A group of 140 critical technologies were analysed in detail for three iterations of the e-Delphix method according to the e-foresight concept [25]. The polymer surface layers modification technologies were one of 14 thematic areas analysed under this foresight research.

Investigations with the e-Delphix method with the sample size of 198 have revealed the robust strategic position of polymer surface layers modification technologies among other materials surface engineering technologies. 38% of the surveyed claim that the technologies have good prospects of industrial applications. 41% of the respondents maintain that numerous scientific and research works will be devoted to such technologies in the nearest 20 years. 43% of the surveyed point out that the thematic area of “Surface engineering of polymers” is crucial and its significance should be absolutely on the rise so that an optimistic scenario of the country's/Europe/World development – "Race won" – can come true. The scenario provides that the potential available is exploited adequately for fulfilling the strategic development objectives; people are, statistically, better off; social attitudes are optimistic and prospects for the coming years bright. 63% of the surveyed think that the importance of laser technologies in relation to other materials surface engineering technologies will be growing, whereas 37% maintain it will remain on the same level, and no one said that this role would diminish over the nearest 20 years. The promising outcomes of technology foresight, elaborated based on the reference data, point, therefore, to the predicted important role of polymer surface layers modification technologies for the development of materials surface engineering overall (mezo scale) and for the development of the entire national/European/global economy (macro scale). The discussed results of technology foresight, established according to the opinions of the experts expressed during investigations with the e-Delphix method, presenting the position of polymer surface layers modification technologies versus overall materials surface engineering, is shown in Fig. 16.

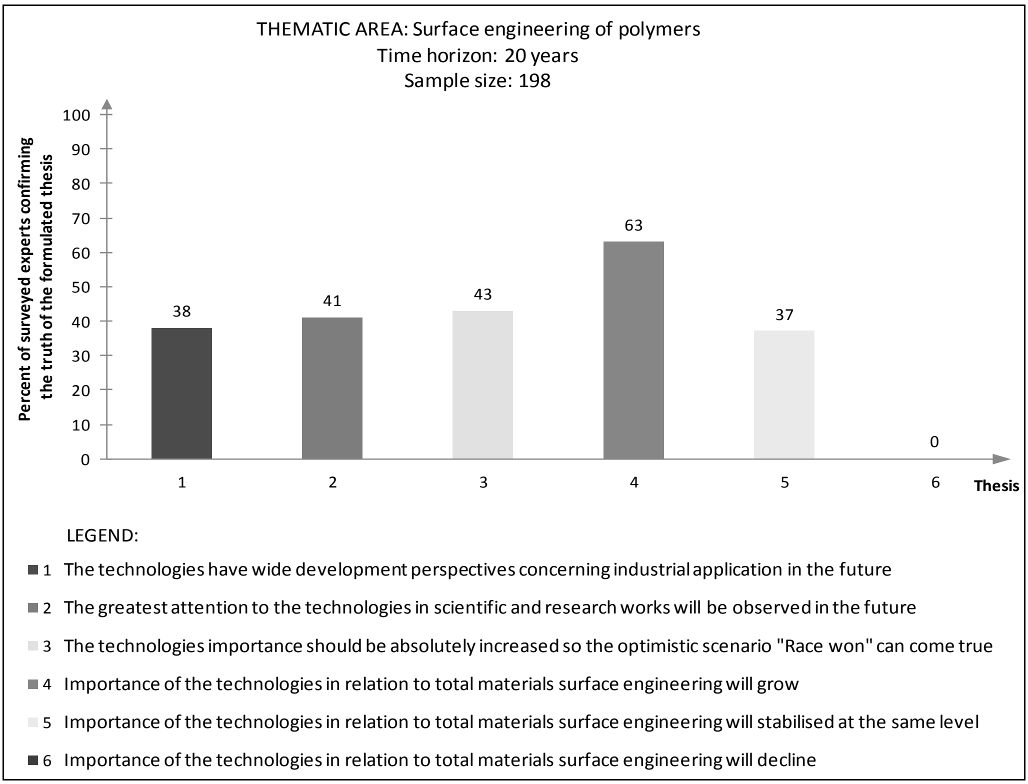


Figure 16. *Strategic position of polymer surface layers modification technologies versus other materials surface engineering technologies*

4.2. Strategic position of the selected polymer surface layers modification technologies

The results of the foresight research described in this chapter include the assessment of the potential and attractiveness of the analysed technologies against a micro- and macro-environment. The assessment was performed based on key experts' opinions expressed with a universal scale of relative states consisting of ten points and a recommended strategy of managing a relevant technology together with the predicted strategic development tracks resulting from such assessment.

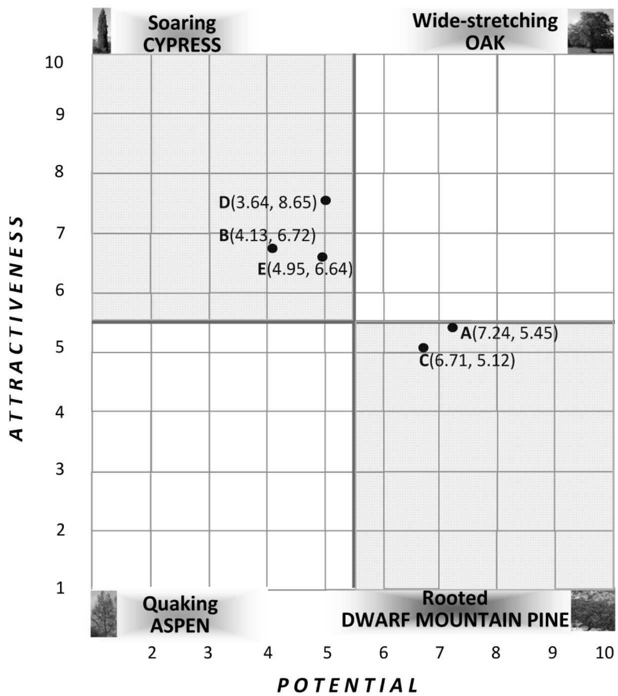


Figure 17. *The dendrological matrix of technology value prepared for, respectively: (A) Corona Treatment, (B) Remote Plasma Treatment, (C) Low Pressure Plasma Treatment, (D) Laser Treatment, (E) Electron-Beam Irradiation*

The individual technologies have been evaluated by the experts for their: business, economic, humane, natural and system attractiveness as well as for their: creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and a result obtained for the individual groups of technologies was entered into the dendrological matrix of technologies value (Fig. 17). The base technology of corona discharges (A) and the mature technology of polymer surface layer modification by means of low-temperature plasma in the conditions of lower pressure (C) were found in the field of rooted dwarf mountain pine representing a high potential and limited attractiveness. A prototype technology of laser modification of polymer surface layers (D) with very promising development prospects, especially in the electronic and computer industry, the evolving technology of polymer materials surface layer modification with plasma generated outside the modification zone (B) and a prototype technology of modification of a polymer materials surface layer with a high-energy electron beam (E) was found in the soaring cypress field corresponding to attractive technologies with a limited potential, so additional investigations strengthening such technologies and representing groundwork for future, broad industrial applications are necessary.

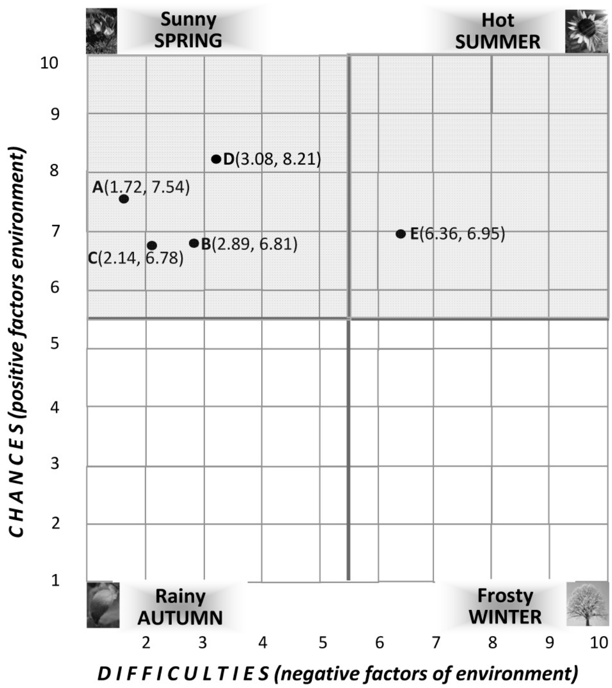


Figure 18. *The meteorological matrix of environment influence prepared for, respectively: (A) Corona Treatment, (B) Remote Plasma Treatment, (C) Low Pressure Plasma Treatment, (D) Laser Treatment, (E) Electron-Beam Irradiation*

Positive and negative environment influence on the relevant groups of technologies is presented graphically with a meteorological matrix of environment influence. The results of a multi-criteria analysis of the experts' scores acquired in the survey-taking process were entered into the matrix, as shown in Fig. 18. The environment of most of the investigated technologies (A-D) is unusually supportive and provides ample opportunities and very few difficulties, therefore was placed in the sunny spring field. The technology (E) consisting in the modification of the polymers surface layer with a high-energy electron beam is in a stormy environment, as it is accompanied by numerous opportunities such as new applications and opportunities to enter new markets, as well as by difficulties related to high investment costs, necessity to employ highly qualified staff, a strong position of buyers and suppliers in the supply chain, as well as a high level of specialisation and applicability in relation to a narrow group of products.

Using the pre-defined mathematical relationships, the specific numerical values provided in the dendrological and meteorological matrix [2x2] were moved to the technology strategy matrix dimensioned [4x4]. The matrix presents graphically the position of the investigated polymer surface layers modification technologies according to their values and intensiveness of environment influence, by indicating the appropriate action strategy (Fig. 19). The development prospects of the technology of (A) corona discharge and (C) polymers surface layer modification by means of low-temperature plasma in the conditions of lower pressure was rated 9 points in a universal scale of relative states consisting of 10 points and were found in the field of dwarf mountain pine in spring. It is recommended in relation to these technologies to exploit good market conditions while enhancing the attractiveness of a high potential technology, especially in terms of modernising, automating, computerising and promoting it intensively to maintain competitive edge. The technology D (4.7, 8.8) of laser modification of polymer surface layers was rated equally high (9 points), and 8 points were assigned to the technology B (4.3, 8.6) corresponding to the modification of the polymers surface layer with plasma generated outside the modification zone. Both technologies were found in the cypress in spring field, with the development strategy envisaging investigations, improvements and additional investments for an attractive technology taking advantage of the robust market circumstances. The development prospects of the technology (E) of modifying the surface layer of polymer materials by means of a beam of high-energy electrons were rated as moderate (6 points) due to a stormy environment bringing both, many opportunities but also difficulties. The cypress in summer strategy, appropriate for the technology (E), consists of strengthening the potential of an attractive technology in uncertain environment conditions, of individual risk assessment and, depending on the outcome, fighting fiercely for customers or phasing out the technology slowly from the market if difficulties outpace opportunities offered by the environment.

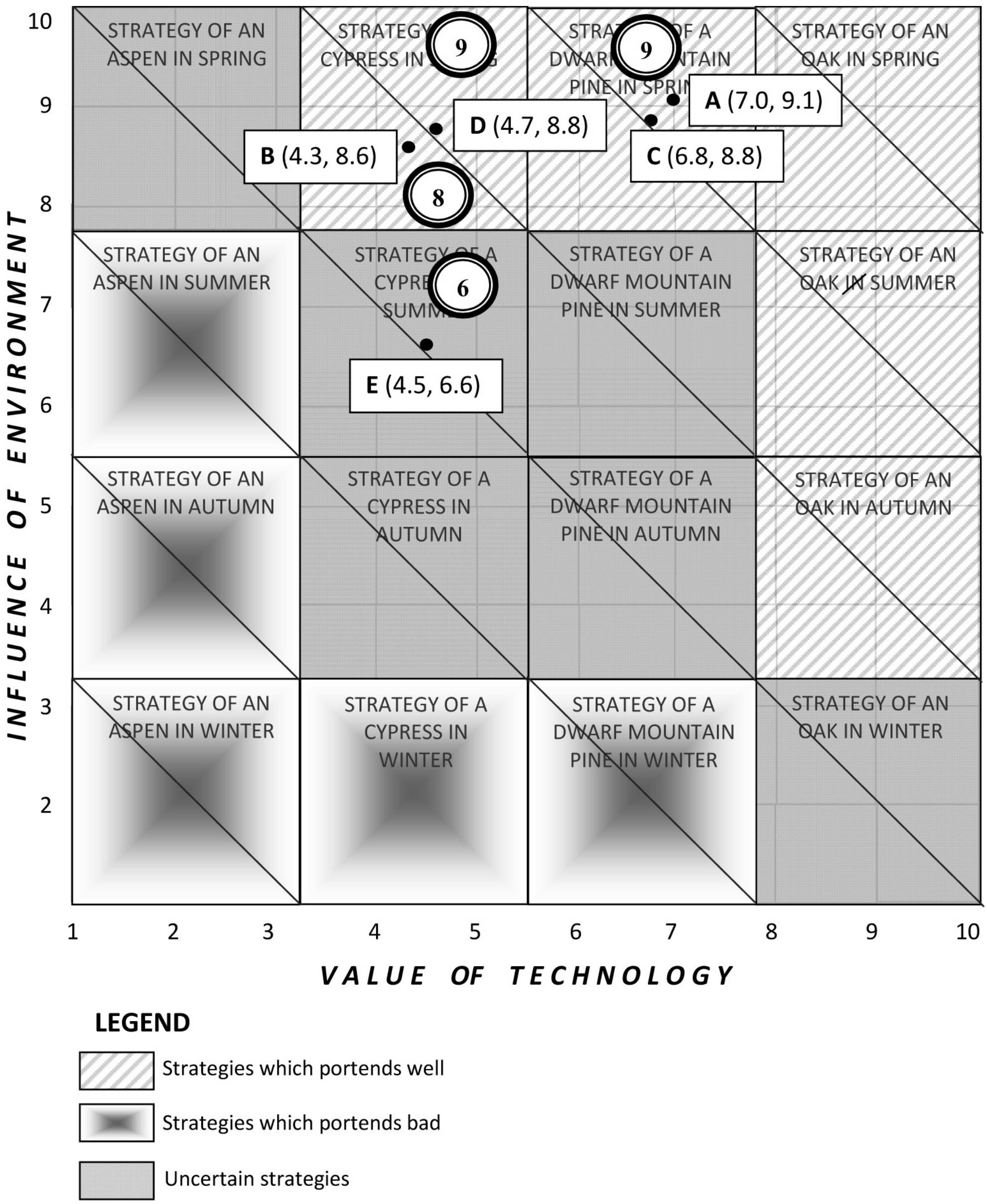
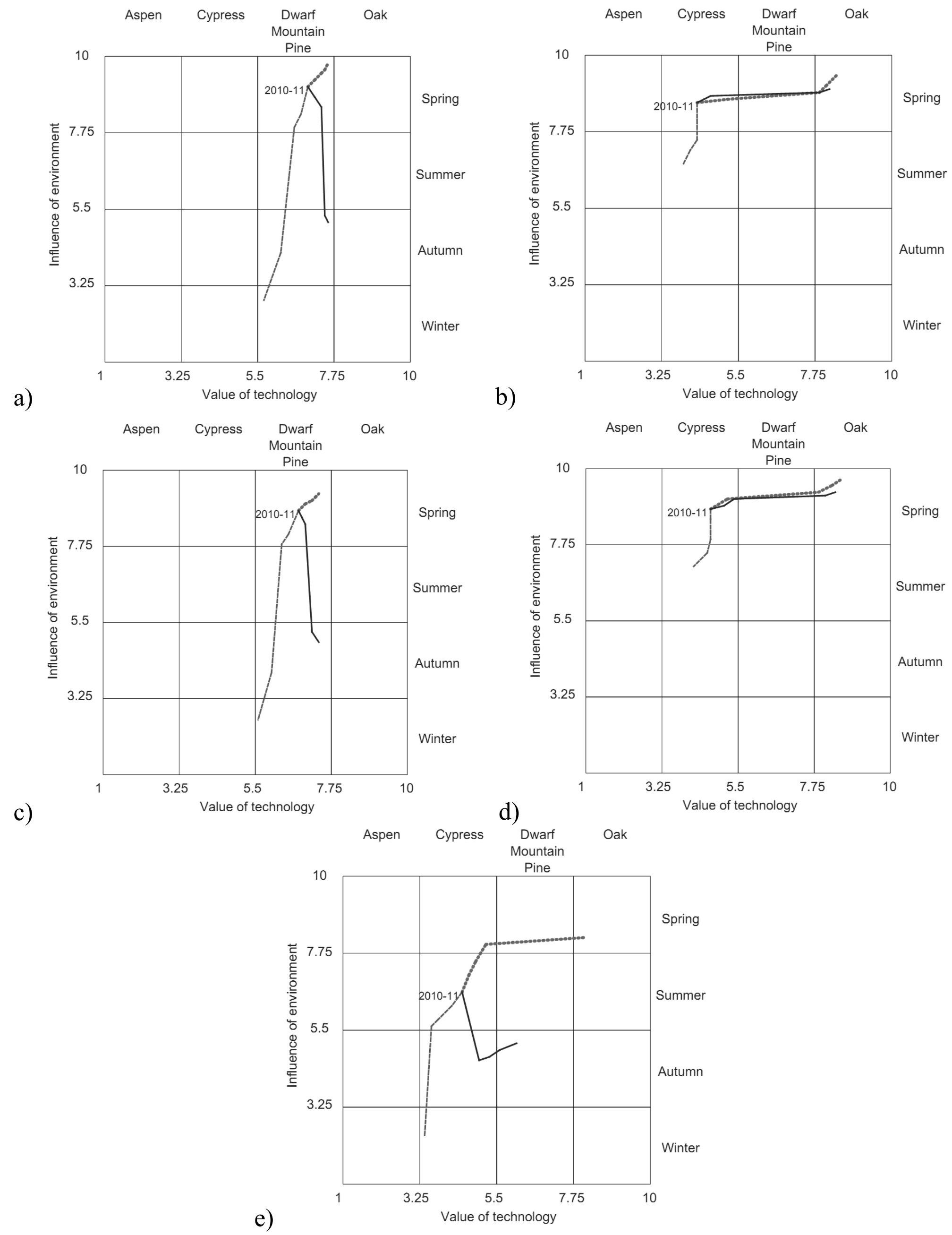


Figure 19. *The matrix of strategies for technologies prepared for, respectively: (A) Corona Treatment, (B) Remote Plasma Treatment, (C) Low Pressure Plasma Treatment, (D) Laser Treatment, (E) Electron-Beam Irradiation*



Types of strategic development tracks:
Optimistic Neutral Pessimistic

Figure 20. *Strategic development tracks prepared for, respectively: (A) Corona Treatment, (B) Remote Plasma Treatment, (C) Low Pressure Plasma Treatment, (D) Laser Treatment, (E) Electron-Beam Irradiation*

Strategic development tracks for the individual specific technologies representing a forecast of their development for the years of 2015, 2020, 2025 and 2030 according to the three variants: optimistic, pessimistic and the most probable one, were next entered into the matrix of strategies for technologies. Simplified charts presenting the results of all the investigations carried out for the five analysed groups of technologies corresponding to polymer surface layers modification using different physical processes are shown in Figs. 20a-e.

5. Technology Roadmaps

Technology roadmaps are a useful tool of a comparative analysis for the particular technologies according to the materials science, technological or economic criterion adopted [63-65]. Three time intervals for, respectively, 2010-11, 2020 and 2030, are given on the axis of abscissa of a technology roadmap created with a custom concept [23, 24], and a time horizon for the overall results of the research provided on the map is 20 years. Seven main layers are provided on the axis of coordinates responding, respectively, to the following more and more detailed questions: When? Why? What? How? Where? Who? How much? Each layer of the main maps is split into more detailed sub-layers. An advantage of technology roadmaps is their flexibility and, if needed, additional sub-layers can be added or expanded for the maps according to the industry, size of enterprise, scale of the company's business or an entrepreneur's individual expectations. Technology information sheets, containing technical information very helpful in implementing a specific technology in the industrial practice, especially in SMEs not having the capital allowing to conduct own research in a given field, are detailing and supplementing the technology roadmaps. Technology roadmaps were prepared using reference data such as the results of materials-science and foresight research for all the five analysed technologies and, as the data presented therein is diverse, a decision was made to present them fully in, respectively, Figs. 21-25.

6. Summary

The chapter presents a comparative analysis of the five selected technologies of polymer surface layers modification by using physical processes resulting in changes to the structure of

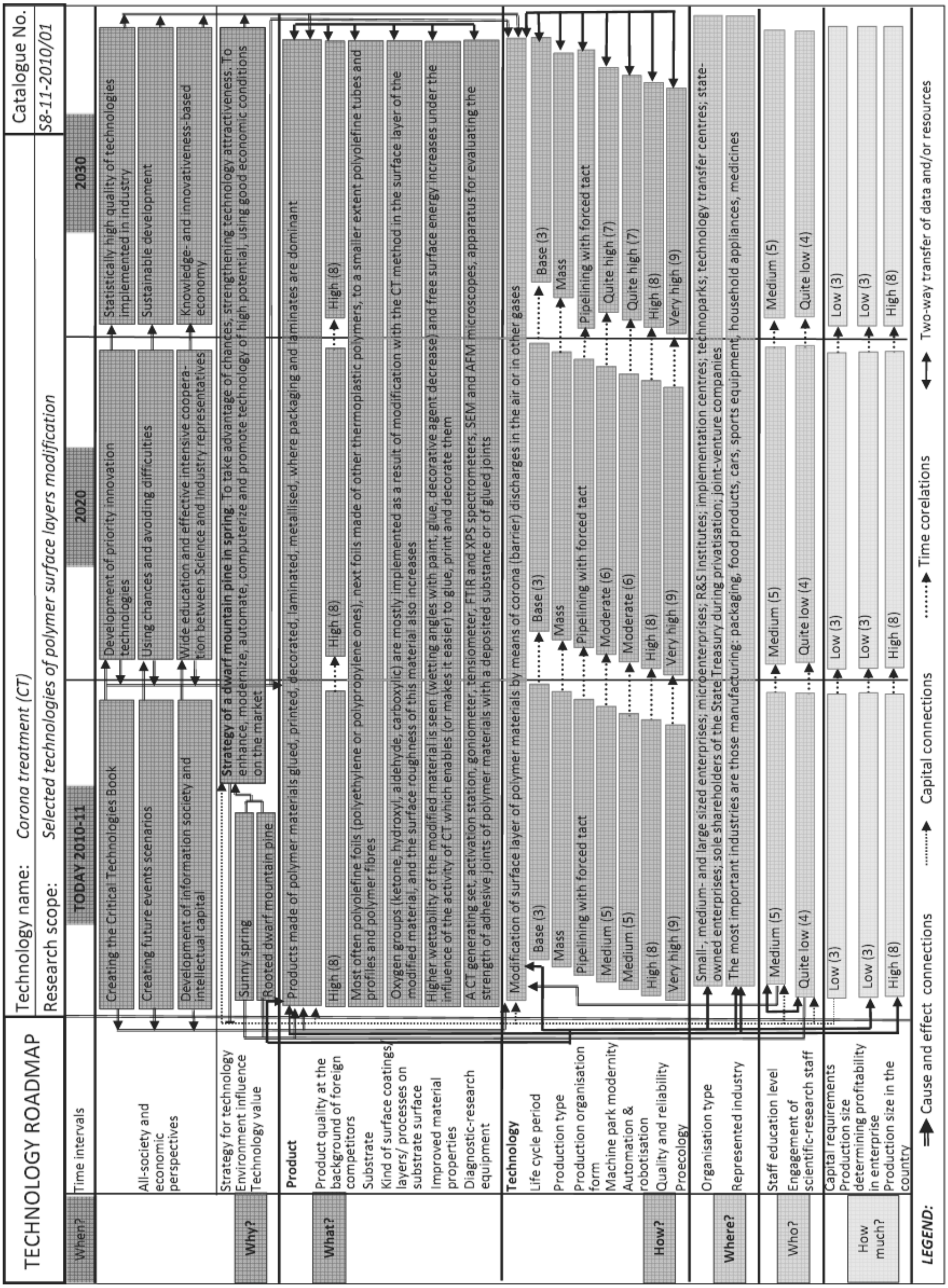


***Figure 21.** The technology roadmap prepared for prepared for the (A) technology, i.e. Corona Treatment*

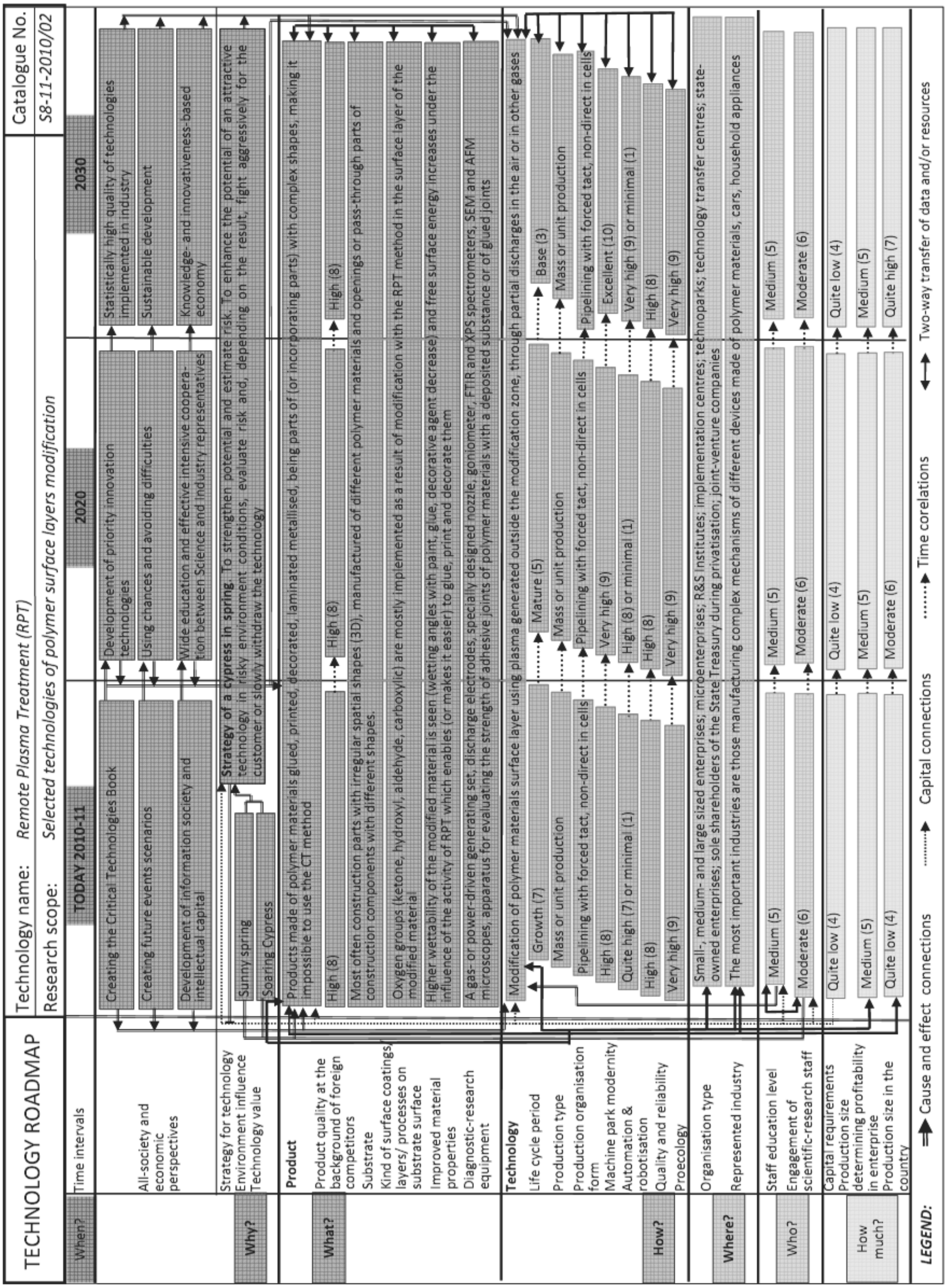


Figure 22. *The technology roadmap prepared for the (B) technology, i.e. Remote Plasma Treatment*

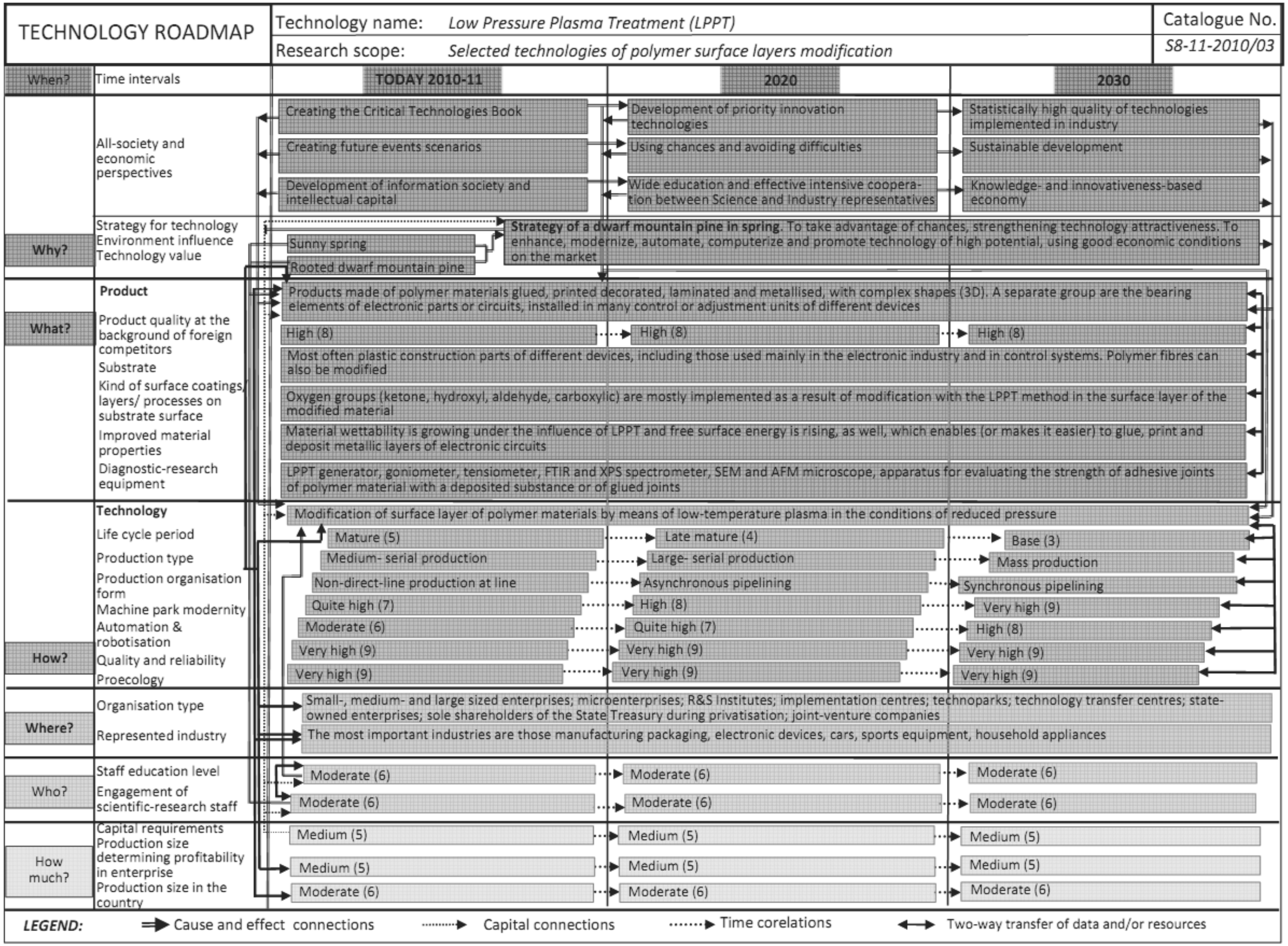


Figure 23. *The technology roadmap prepared for the (C) technology, i.e. Low Pressure Plasma Treatment*

Figure 24. *The technology roadmap prepared for the (D) technology, i.e. Laser Treatment*

Figure 25. *The technology roadmap prepared for the (E) technology, i.e. Electron-Beam Irradiation*

surface and properties of polymers as a criterion of classification. The materials science and foresight research pursued represents part of a larger project aimed at selecting and characterising, using a cohesive pool of analytical tools, the priority, innovative materials surface engineering technologies, as discussed by the following series of publications, notably: [66-72].

The analysis made has revealed very high (9 points out of a scale of relative states comprised of ten points) development prospects of the base technology of modification of polymers surface layer by means of corona (barrier) discharges in the air and in other gases (A) that is currently widely used in the industry, especially in the packaging, food, automotive, pharmaceutical, sports equipment and household appliances sector. Oxygen groups are implemented in the polymers surface layer as a result of modification using corona discharges and its roughness increases leading to higher wettability of material and increased surface free energy, enabling to or making it impossible to glue, print and decorate modified polymers. The anticipated progress and unthreatened strategic position of corona discharges in the nearest 20 years stems from the fact that the technology is relatively simple, highly effective, not requiring large expenses at the stage of investment and in further operation, and no highly skilled personnel is required. The disadvantages include a possibility of damaging very thin foils less than 15 μm thick, material shrinkage during the process and the necessity to neutralise or evacuate ozone. The further development of corona discharges will surely relate to better constructions of activators and measuring apparatuses and automatic process control circuits. A new domain of future uses is a sterilisation function for different micro-organisms, and this can be used in the food, pharmaceutical and medical industry.

A strategic position of the technology (C), i.e. polymer materials surface layer modification with low-temperature plasma in the conditions of low pressure (0.05-5 hPa) was also rated 9 points in the ten-point scale, which corresponds to a very high level. This relatively simple technology allows to modify thin polymeric foils and also parts with the similar shape (3D) very uniformly and gently. A separate group of products manufactured this way are the carriers of parts or electronic circuits installed in control or adjustment units for different equipment. The technology of polymers modification with low-temperature plasma in the conditions of lower pressure is used most often for manufacturing packaging, electronic equipment, sports equipment and household appliances and in the automotive industry. The anticipated directions of technology improvement will include shortening the modification time that currently is several dozens of minutes and is causing limited process efficiency; improved construction of discharge chambers with high required tight sealness and the progress of the process itself to eliminate its cyclicity for the sake of ensuring continuous modification.

The development prospects of polymers surface layers modification using different types of lasers, including excimer lasers (D) were also highly valued (9 points). This technology is currently at its prototype stage of life cycle and allows to modify precisely the selected pieces of a given material's surface layer, including narrow conductive layers designed earlier, as well as other areas with their complex shapes. The technology should hence be used in the electronic industry in the future, especially for producing computers and in the machine and packaging industry. The potential of polymer surface layers laser modification will most apparently be reinforced in the nearest future by improving the design of lasers, measuring apparatuses and automatic process control units. A current limitation for the technology is the fact that areas with large surface areas cannot be modified as well as relative high consumption of electricity supplied to the laser per a unit of field of the area modified, and the characteristics will surely be investigated and improved further over the nearest 20 years.

The development prospects of the currently evolving polymer materials surface layer modification technology using plasma generated outside the modification zone through partial discharges in the air or in other gases (B) was rated high (8 points). Regardless the constraints of the technology, i.e. relatively small efficiency of the modification process and the fact that the process at the current stage of development must be carried out manually, it has a crucial advantage as compared with a classical corona discharges method (A), i.e. elements can be modified having complex, irregular spatial shapes (3D) and openings and pass-through parts of construction components. The technology enables to generate plasma with varied properties, in different gases, also using small portable devices or such mounted in the holders of robots, which is currently used mainly for manufacturing complex mechanisms of different devices made of polymer materials and for manufacturing cars and household appliances. The future development trends will surely relate to improved efficiency of the plasma generator and process automation.

The most uncertain development prospects (6 points), for the technologies subjected to foresight and materials science research discussed in this chapter, are characteristic for the technology of polymer materials modification using high-energy electron-beam irradiation (E), being in a stormy environment. The environment of the technology brings both, many opportunities such as new applications at attractive markets with a high capital potential (power industry, construction), as well as difficulties associated with high investment and operational costs (energy-intensive process), highly skilled staff needs to be employed, a position of buyers and suppliers in the delivery chain is strong, and the level of specialisation is high and applicability is true for a narrow range of products. If the safeguard system is used improperly or fails, a radiation hazard may occur, which is a disadvantage considering the environmental friendliness of the process. Energy-intensive electron-beam irradiation is used primarily for modifying deeper layers of the material up to 40 mm thick such as usually the insulating sheaths of power cables or polyethylene tubes and plates or structural materials containing different polymers. The purpose of modification causing the netting of the surface layer of the material is to improve mechanical strength, resistance to degradation processes and improved hydrophilicity next allowing to glue, print and/or decorate it. The predicted development of the technology (E) will be aimed at improving the modification devices and optimising the process parameters, and a surprise development scenario for the technology, both an optimistic and pessimistic one, is not out of question.

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13 Neural networks aided future events scenarios presented on the example of laser surface treatment

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Abstract

Purpose: *The purpose of the chapter is to present a methodological concept allowing to demonstrate the development directions of materials surface engineering according to the level of generality and the intensity of the phenomena analysed on other phenomena.*

Design/methodology/approach: *A set of analytical methods and tools was used to present the development directions of materials surface engineering at the three levels analysed, i.e.: a macro-, meso- and microlevel. The analytical methods and tools comprise the scenario method, artificial neural networks, Monte Carlo method, e-Dephix method, statistical lists as bar charts, foresight matrices together with technology development tracks, technology roadmaps, technology information sheets and the classical materials science methods.*

Findings: *A research methodology allowing to combine a presentation and description of the forecast future events having a varied level of generality and capturing the cause and effect relationships existing between the events.*

Research limitations/implications: *The methodological concept discussed, implemented with reference to materials surface engineering, has a much broader meaning, and can be successfully applied in other technology foresights, and also in industrial and thematic foresights after minor modifications.*

Practical implications: *The outcomes of the research conducted may be and should be used in the process of creating and managing the future of materials surface engineering and, within the time horizon of 20 years, may and should influence positively the development of the economy based on knowledge and innovation, sustainable development and the statistical level of the technologies used in industry, especially in small- and medium-sized enterprises.*

Originality/value: *An own methodological concept constitutes an original way of presenting the development directions of the investigated field of knowledge. The use of neural networks represents an innovative and experimental approach unseen in foresight methodology to date.*
Keywords: *Scenario method; Neural networks; Foresight; Laser surface treatment*

This chapter has been also published as:
A.D. Dobrzańska-Danikiewicz, J. Trzaska, A. Jagiełło, E. Jonda, K. Labisz, Neural networks aided future events scenarios presented on the example of laser surface treatment, Archives of Materials Science and Engineering 51/2 (2011) 69-96.

1. Introduction

The contemporary techniques of building scenarios were used for the first time in the 50's of the last century and their widespread use in the public and business area has started since the seventies [1]. According to the foresight technology, the scenarios reflect the future opportunities and are developed in a systematised way and their task is to capture the holistic sense of particular conditions [2]. The building of scenarios consists of describing the events in the area investigated and indicating their logical and chronological sequence including the macroeconomic factors influencing positively or negatively the forecast development of the events, thus brings specific opportunities and risks. The extensive research [3] who has analysed altogether 860 foresight projects, pursued globally, indicates that the creation of scenarios of future events is the third most often used method. The method was applied in 43% of the projects analysed with only a literature review (55%) and expert panels (51%) being ranked higher. This result translates directly into the applicability of the scenario method in the particular regions of the World. The applicability of the scenario method in Northwest Europe, Eastern Europe, North America and Asia ranges between 40 to 50%. The method has been used less often in Northern Europe (approx. 30% of projects) and North America (approx. 20% of projects) [4]. Evaluative investigations held for 40 foresight projects that had already been implemented or have been implemented in Poland [5] point out that the scenario method enjoys greater popularity in Poland as compared to the global average. The method has been used in almost the three-third of the projects and ranked first among all the methods applied, staying ahead of the Delphi method and the expert panels method that ranked just behind. The building of scenarios of future events is also found among the tasks planned for implementation as part of technology foresight concerning the priority innovative technologies and directions of strategic development of material surface engineering [6]. This chapter presents methodology assumptions for the research works conducted as well as the application examples of the scenario method at the different levels of generality of the issues discussed.

2. The scenario creation custom idea

The references [7-11] indicate that there is no one correct and generally accepted method of creating the scenarios of future events or a management algorithm recommended for implementation in the scenario creation process. In fact, the algorithm is created each time from the scratch by the practicians implementing a specific project [1]. The same refers to building the scenarios presenting the forecast future of materials surface engineering where a methodological challenge exists of combining skilfully the presentation and description of the phenomena characterised by varied generality and to capture the cause and effects relationships existing between them. In order to solve the so formulated research task, all the analysed phenomena are divided into the three groups:

- A **macrogroup** with all the single critical phenomena of general nature characterised by strong interaction with the other phenomena;
- A **mesogroup** with a limited number of phenomena interacting moderately with the other phenomena;
- A **microgroup** comprising numerous specific phenomena highly sensitive to the interaction of other phenomena.

The classification adopted is presented graphically in Fig. 1. The approach shown allows for a two-fold method of deductive reasoning, i.e. analysis or synthesis. The analytical approach consists of determining what macroscenario will occur in the future for the specific combination of the current micro- and mesofactors. Deductive reasoning by way of a synthesis, adopted for the undertaken research concerning the forecast development of materials surface engineering, forces us to seek such a combination of micro- and mesofactors that would contribute, with

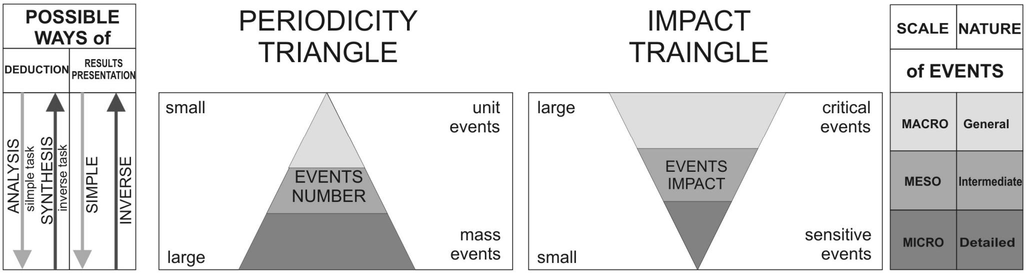


Figure 1. *Overview of the phenomena subjected to investigations with the scenario method*

a specific probability, to the occurrence of each of the three possible macroscenarios in the future. The presentation of research outcomes that is unrelated to the method of deductive reasoning is another issue. This chapter uses a simple method of presenting the research results and the phenomena investigated are presented in the individual sub-chapters starting with the highest level of generality (macro), through an indirect level (meso) ending with the most detailed notions (micro). According to the periodicity triangle shown in Fig. 2 created for the materials surface engineering research conducted, the number of the phenomena considered increases along with the growing level of specificity. 3 scenarios of future events are considered at the macrolevel: optimistic, neutral and pessimistic with their overview presented in Table 1. The mesolevel is grouping 16 key factors influencing the development of materials surface engineering and 14 thematic areas analysed under the foresight research. The micro-level is represented by 140 groups of critical technologies. Specific technologies with an unknown *n* number can be distinguished between for them, often differing in details only. They can, however, substantially condition the development prospects of a particular technology and its applicability in the industrial practise. Considering the scale of the phenomena described, the full results of the research cannot be included in one chapter only. A representative group of laser technologies in surface engineering was, therefore, chosen (mesoscale) with special focus on laser remelting and alloying, especially with reference to hot-work alloy tool steels (microscale) and the scenario creation concept developed is presented by using such example. The concept is much more far-reaching and has been applied with reference to all the thematic areas, groups of critical technologies and specific technologies analysed for the research work conducted i.e. [12-20]. The analytical methods and tools for creating the scenarios of future events for materials surface engineering are entered into the periodicity triangle (Fig. 2).

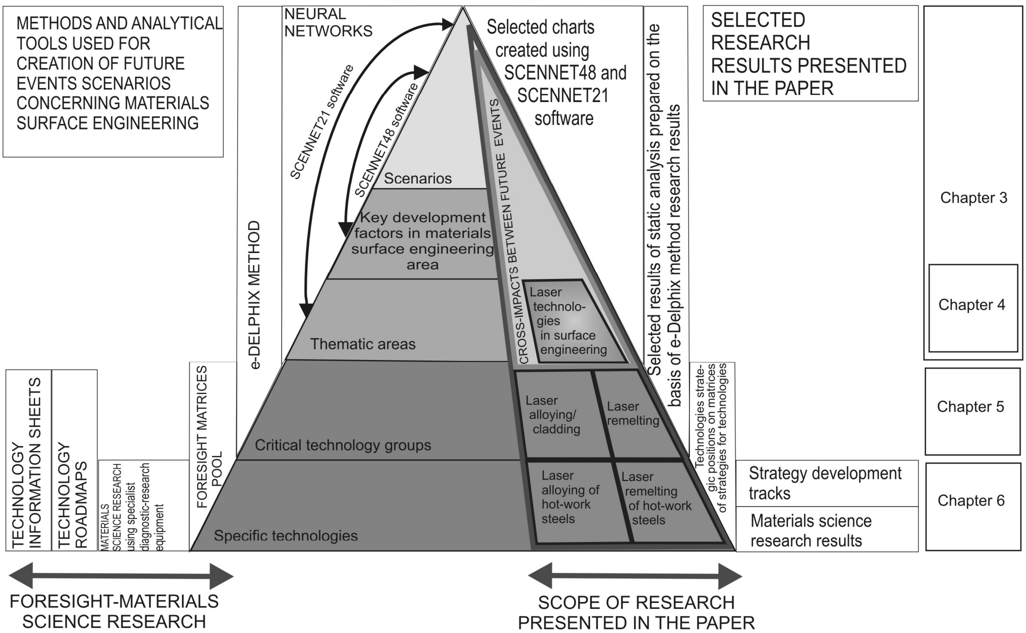


Figure 2. *Analytical methods and tools for creating the scenarios of future events for materials surface engineering and the selected research results presented in the chapter*

Table 1. *Overview of three alternative macroscenarios of events development*

Forecast Macroscenarios	Overview of forecast macroscenario type
Optimistic: Race won	The global economic crisis has been prevented and economic growth is experienced based on peaceful co-operation and international integration. The competitive position of the European Union is growing among the world economies. Numerous reforms are being successfully implemented in Poland having social approval the purpose of which is the actual transformation of economy supporting the sustainable development of the knowledge-based economy. Poland is skilfully combining endogenic growth factors with foreign investments and the effective use of EU funds. The consequence of widespread actions planned is the gradual improvement of the society's education, the wide-scale application of innovative and environmental friendly technologies in many thriving small and medium-sized companies (SMEs) and large corporations operating more and more often in high-tech industries, effective use of Poland's agricultural resources, and also the development of modern transport and ICT infrastructure. The economic, system, technological, financial and social potential available is used adequately to put into life the strategic development goal, statistically people are better off, social attitudes are optimistic and prospects for the coming years bright.
Neutral: Progress achieved	The world economic crisis has been prevented and the World is slowly returning to the growth path in the paradigm of sustainable growth based on co-operation and international integration, although the fear of terrorism and local wars still exists which, in unfavourable circumstances, may spread to many countries. The

Forecast Macroscenarios	Overview of forecast macroscenario type
	European Union needs to fight hard for its position among global economies, especially with regard to China and India emerging as world powers. There are efforts made in Poland, with different outcomes, to tackle reforms aimed at economy transformation and the reforms are often opposed by the society and people's reluctance towards change. Poland is trying to use the EU funds, but not all the money is managed effectively. The introduction of a knowledge-based economy and sustainable development brings results such as the growing education level of the society and its environmental awareness. The SME sector is developing at a constant but slow rate, and the level of implementing the innovative and environmental technologies leaves still much to wish for. Large corporations operate mainly in medium and low and medium and high technologies. The country is constantly facing problems in public finance, agriculture and healthcare, and the modern transport and ICT infrastructure is developing steadily but relatively slowly. The economic, system, technological, financial and social potential is only partly used to achieve strategic development goals, statistically people are slightly better off but social attitudes are mixed. Theoretically good development prospects for the coming years depend on the circumstances in the European and world economy, wise management of public funds in long term and on how quickly the relevant reforms are introduced supported with the society's involvement.
Pessimistic: Inclined plane	The global economic crisis has been slowed down to some degree only. The world is facing terrorism, growing oil prices, consequences of disasters and local wars spreading to more and more countries. The European Union stays behind other global economies, especially China and India emerging as global powers. Usually unsuccessful attempts are made in Poland to tackle reforms serving to transform economy that face social disapproval and strong reluctance towards changes. The EU funds allocated to Poland are smaller and smaller every year, and most of the money is used to save the current economy, whereas the level of investments is slowing down. The implementation of the knowledge-based economy and sustainable development concepts, initially boding well, is now weakening. The MSE sector is developing sluggishly, and innovative environmental technologies cannot be usually applied due to the lack of investments and the low availability of credits. Large corporations operate in medium and low and medium and high technologies, and many of them go bankrupt and move their head offices to Asia. The country is constantly facing problems in public finance, agriculture, healthcare, education and transport infrastructure. The economic, system, technological, financial and social potential is weakly utilised for fulfilling strategic development goals with the goals being, apparently, wrongly formulated. Statistically people are worse off which is accompanied by social unrests. Development prospects for the coming years are weak and Poland will be heading for a disaster if a sudden breakthrough is not experienced.

In addition, the results of the research chosen for presentation in the individual sub-chapters of the chapter are presented graphically. Sub-chapter 3 provides an overview of the methodological assumptions and the examples of the practical implementation of neural networks used for analysing cross impacts in order to identify how the key mesofactors of materials surface engineering development and the specific thematic areas condition the occurrence of each of the three alternative macroscenarios. Sub-chapter 4 includes a development forecast of laser technologies in materials surface engineering established based on the results of the e-Delphix method differing from the classical Delphi method in that experts are surveyed using in an electronic way and in that the level of generality for the questions asked to the experts is growing along with the subsequent iterations of the research. Sub-chapter 5 of this chapter discusses the strategic position of the relevant groups of critical laser surface treatment technologies against the thematic area of "Laser technologies in surface engineering" together with a statistical list presenting, in percents and as forecast by the experts, the values of growth, stabilisation and decrease in the importance of the individual critical technologies. At last, sub-chapter 6 contains the results of the materials science-foresight research concerning laser remelting and cladding of hot-work alloy tool steels with special emphasis laid on the results of materials science investigations and the strategic position of the specific analysed technologies presented in a matrix of strategies for technologies together with the forecast strategic development tracks. The scenarios of future events established concerning materials surface engineering can be characterised, according to the references [9, 21, 22], in a number of ways according to the various criteria of classification Fig. 3 shows an overview of the scenarios prepared for the research followed against the overview of optional scenarios according to the classification criteria adopted in an arbitrary manner.

3. Cross-impacts analysis made using neural networks

3.1. Methodological assumptions

Neural networks were used in a novel and experimental manner to cross impacts analysis. The analysis serves to identify how the key mesofactors of surface engineering development (e.g. collaboration between science and industry, number of specialised laboratories and R&D institutions, continuous improvement and high quality of technology, transparent and friendly legislation, international co-operation and EU funds) and the relevant thematic areas analysed (e.g. laser technologies, thermochemical technologies, nanotechnologies) may influence the occurrence of each of the macroscenarios. A data set elaborated according to the results of survey investigations was divided randomly into the three sub-groups: learning, validation

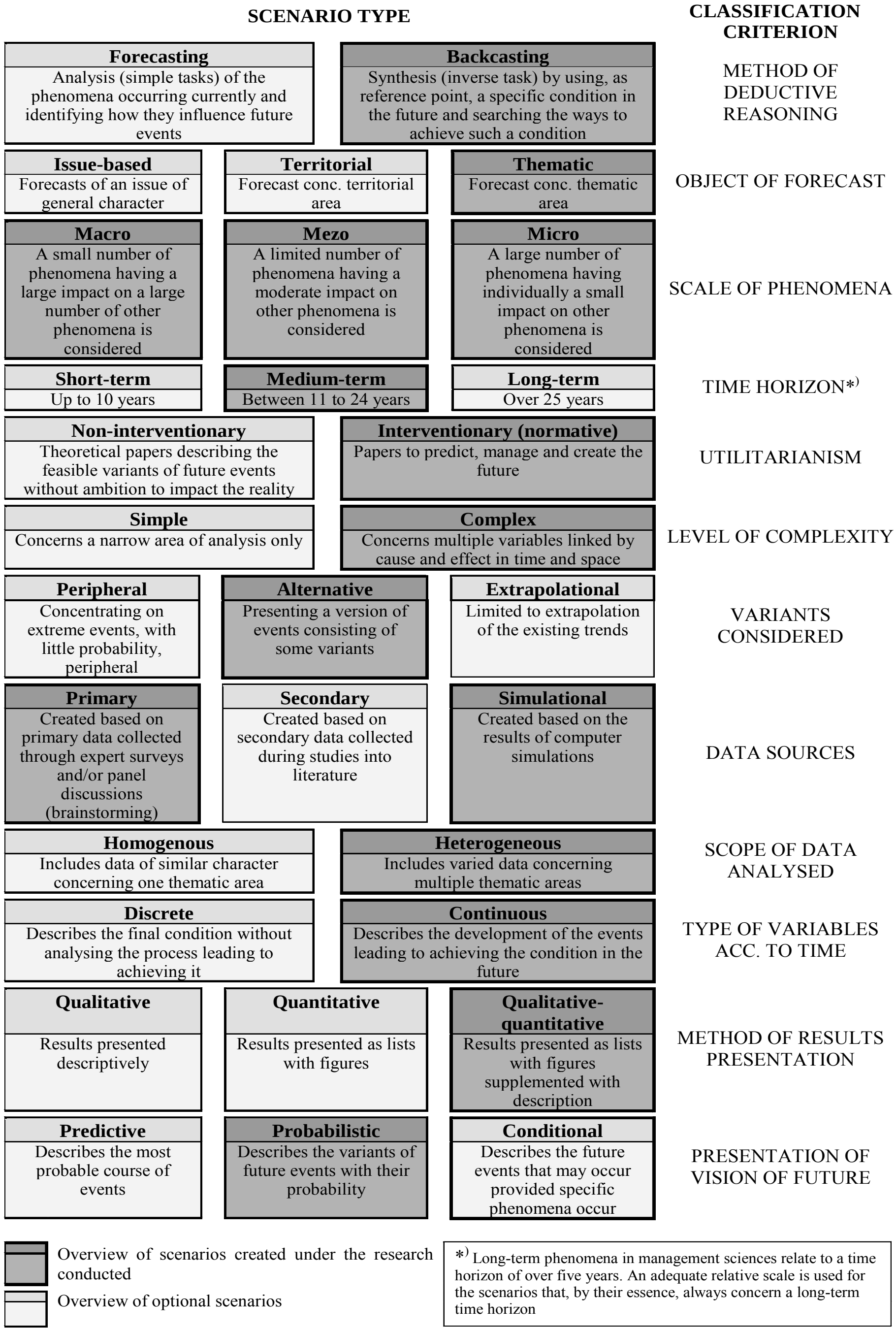


Figure 3. *Overview of the scenarios created under the research conducted against the overview of optional scenarios*

and testing sub-group. The data from the learning set was used for modifying network importance in the learning process and the data from the validation set was used for network evaluation in the learning process. The remaining part of the data, as a test set, was used to determine, independently, network efficiency after completing fully the network development procedure. The following values were used as the basic indicators of model quality evaluation: an average absolute error of network forecast, a standard deviation of the network forecast error, R Pearson's correlation coefficient for the value set and for the value obtained at the neural network output. The quality evaluation indicators of artificial neural networks were calculated for each of the separated sets. The similar values of the average error, standard error deviation and correlation coefficient confirm the generalisation ability of the network, i.e. an ability to generalise the knowledge acquired in the learning process.

9 models were created altogether using artificial neural networks by adopting, as dependent (input) variables, the probabilities of the occurrence of a growth trend, stabilised trend and/or declining trend determined for the key mesofactors conditioning the development of materials surface engineering and for the individual thematic areas for the research domain of M (Manufacturing) and P (Product). The first research field (M) reflects a manufacturer's point of view and encompasses the production processes determined by the state of the art and a machine park's manufacturing capacity. The second research field (P) is determined by the expected functional and usable properties resulting from the client's demands and concentrates on the product and the material it is made of. The experts were evaluating the occurrence probability of the relevant scenarios by dividing the total value of probability (of 100%) by the three possible variants of future events. The (output) dependent variables represent a probability that each of the three macroscenarios considered, i.e. optimistic, neutral and pessimistic, occurs. The types of the scenarios created and their links to neural networks are presented in Fig. 4.

A project of artificial neural networks and their numerical simulation was prepared with Statistica Neural Networks software, 4.0F version. The following parameters were defined to create a calculation model using an artificial neural network: neural network types, neural network structures, error functions, activation functions, postsynaptic potential (PSP) functions, training methods and parameters, variables scaling methods. The type of a neural network is defined with a mathematical neuron model and also with the characteristic arrangements of neurons in the network and also with the method of links between neurons, as discussed in the following publications [23-27]. General regression neural networks (GRNN)

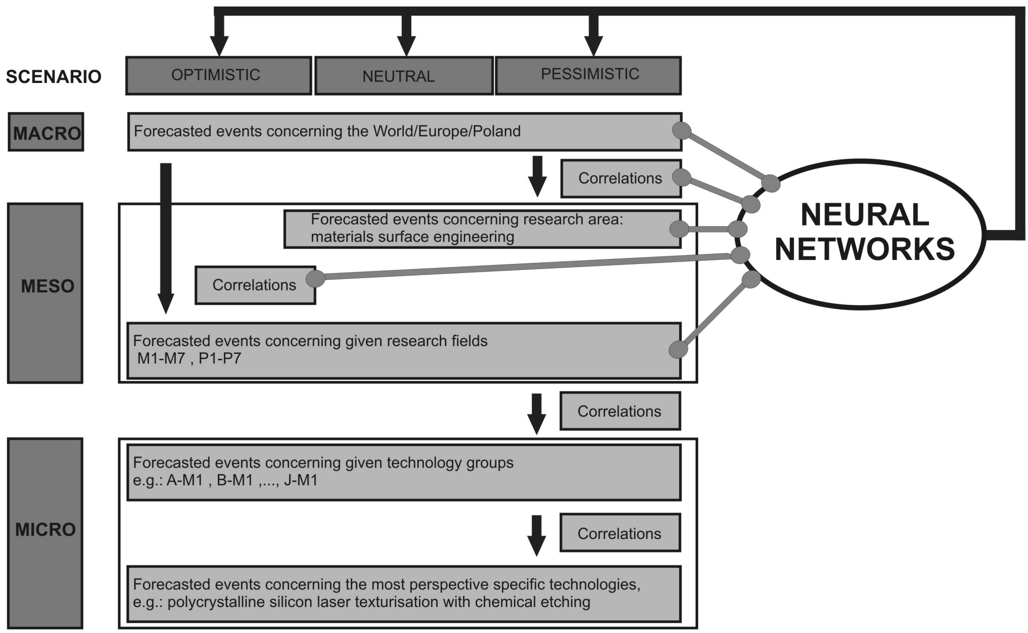


Figure 4. *The types of the scenarios created versus neural networks*

are made up of four layers: an input, radial, regression and output layer. Radial neurons, the number of which equals the number of patterns, represent the centres of concentrations existing in the training set. The regression layer, made up of linear neurons, has one neuron more than the output layer. The neurons of this layer fulfil two tasks: the first task – fulfilled by the neurons the number of which corresponds to the number of the network outputs, calculate conditional regression for each output variable, the second task – fulfilled by a single neuron, comes down to calculating the density of probability [28, 29]. Each of the neurons of the output layer designates a quotient of conditional regression calculated for a neuron of the preceding layer and for the density of probability. Neural networks with radial base functions (RBF) have three layers: an input layer, a hidden layer with radial neurons and an output layer with neurons having a linear characteristic. Linear neural networks (LNN) have two layers only: an input and output layer. Information is processed in the output layer only. The output layer has a linear PSP function and a linear activation function. The most popular type of neural networks is a multilayer perceptron (MLP). A linear postsynaptic potential function and usually a nonlinear activation function are used for such type of neural networks. Determining the number of hidden layers and the number of neurons in such layers is essential for designing the structure of multilayer perceptron [30, 31]. Table 2 lists the types of neural networks analysed in the chapter and their corresponding characteristic values of future parameters.

Table 2. *The parameters optimised when designing neural networks*

Network type	Training method	Activation function	PSP function	Error function
LNN	pseudoinversion	Linear	linear	sum of squares
RBF	k-average, k-nearest neighbours, pseudoinversion	linear, linear saturation, exponential	linear, radial	
GRNN	sampling	exponential, linear saturation	quotiential, radial, linear	
MLP	reverse error propagation, conjugated gradients, quasi-Newton, Levenberg-Marquardt, fast propagation, delta-bar-delta	logistic, linear with saturation, hyperbolic	linear	

Artificial neural networks allow for the building of relations between the investigated values without defining a mathematic description of the problem analysed. It is essential to prepare a representative set of experimental data. The special cases of neural networks analysed in the training process should be distributed equally across the whole domain of the function approximate [32]. An important thing is to define the variability range of the data analysed, thus defining a space in which a neural network can be used. Extrapolation beyond the range of training data may lead to significant prediction errors. The fact that single values exist only within certain ranges of input variables does not allow to make an assumption that the neural model prepared will be correctly predicting the value of a dependent variable in the area defined by the minimum and maximum values of the relevant independent variables. Whilst analysing the results of the survey investigations, special attention was paid to untypical, rare data. Those answers of the experts identifying the occurrence probability of a growth trend, stabilised trend or falling trend, clearly differing from the information provided in the other surveys, were analysed. The evaluation was made using the size tables prepared for independent variables. 5 and 95 percentiles were determined for each variable input. The range of independent variables was thus designated for which a neural model can be used. A few dozens of neural networks differing by their type (GRNN, RBF, MLP, LNN), structure, error and activation functions, training method and parameters were analysed for each model. Information on the neural networks characterised by the most favourable values of the indices used for the evaluation is listed in the table. Table 3 gives an example of such a list prepared for an optimistic scenario applying to the research field (P).

Table 3. *Overview of artificial neural networks: research field (P), optimistic scenario*

Network symbol		**3.2P_1**	**3.2P_2**	**3.2P_3**	**3.2P_4**	**3.2P_5**
Type of network / number of neurons in layers: input-hidden-output		MLP/ 21-5-1	MLP/ 21-6-1	**MLP/ 21-7-1**	MLP/ 21-9-1	MLP/ 21-11-1
Training method / number of training epochs		BP/50, CG/2	BP/50, CG/130	**BP/50, CG/132**	BP/50, CG/33	BP/50, CG/273
Average absolute error, %	training set	7.5	6.3	**6.3**	7.0	5.7
	validating set	7.5	6.2	**6.4**	6.7	5.2
	testing set	7.9	7.0	**6.5**	6.8	5.5
Standard error deviation, %	training set	9.8	8.4	**8.0**	9.4	7.3
	validating set	10.2	8.9	**9.8**	9.1	8.0
	testing set	9.7	8.9	**7.9**	8.5	7.1
Correlation coefficient	training set	0.45	0.63	**0.68**	0.51	0.74
	validating set	0.43	0.62	**0.6**	0.59	0.71
	testing set	0.48	0.59	**0.69**	0.63	0.78

Explanations:
Amount of data in the training / validating / testing set: 150 / 35 / 35
Error function: sum of squares
Activation function in the input/ hidden/ output layer: linear saturation function/ logistic function / linear saturation function
Post Synaptic Potential (PSP) function: linear function
BP: Error back propagation method
CG: Conjugate gradient method
(shaded) Neural network chosen for further analysis

After completing the stage of designing and of numerical verification of artificial neural networks including calculations for a test set, a computer simulation of the impact of the trend change occurrence probability in the analysed thematic area on the macroscenario forecast was performed. The neural models developed were also used to calculate the values of independent variables for which each of the macroscenarios considered should assume the defined value. As there was no mathematical model describing the process examined and as there was relatively large space for potential solutions, it was decided that random activity will be the appropriate approach to solve the issue analysed. According to the general definition, any techniques employing random variables to solve a problem are called Monte Carlo methods [33]. If the issue is analysed in more detail, the Monte Carlo name often refers to a group of methods having the following common characteristics: they analyse a specific and finite space of considerations, they determine points randomly from the area of input data and for each of them. In addition, due to clear calculation procedures, partial results are obtained and they determine the ultimate result by aggregating partial results [34]. The adopted calculation method of independent variables, for which each of the considered macroscenarios should assume a set value, has the characteristics mentioned above. It should therefore be said that

Monte Carlo methods have been used to solve the research problem. The random sampling of the area in many cases improves a chance of obtaining a suboptimum solution which is a sufficient outcome from the user's prospective. As the number of tests increases, so increases the probability of designating a vector of input variables for which the concept examined assumes optimum values. The detailed stages of implementing neural networks in e-foresight research are shown in Fig. 5.

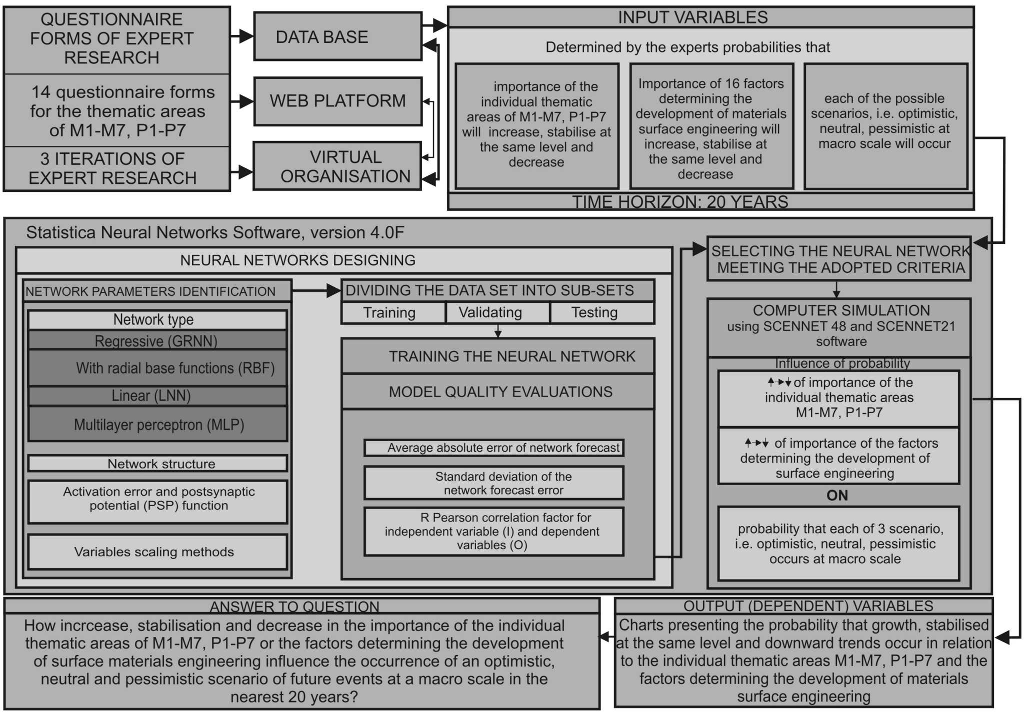


Figure 5. *Detailed stages of implementing neural networks in e-foresight research*

3.2. Computer simulations

The custom-designed SCENNET software available in two versions: 21 and 48, is an IT tool used for the determination of cross relationships and cause and effect relationships between the events likely to occur in the future at a macro- and mesoscale using neural networks. SCENNET21 enables to simulate the influence of development of the individual thematic areas of materials surface engineering on each of the three scenarios at a macroscale using neural networks and to present the simulation results in a table and in a graphical manner. SCENNET48 enables to simulate the influence of the key mesofactors conditioning the future development of materials surface engineering on each of the possible macroscenarios. A simulation based on neural networks is performed also in this case and the simulation results are generated as tables with numbers and as charts showing graphically a probability that the relevant trends of the analysed factors occur.

The task of SCENNET21 software is to find optimum input parameters in order to obtain the set output value for the selected neural networks. The programme was created using C++ language with the C++Builder XE2 Professional packet. It enables to search a maximum output value, minimum output value and a value set by the user and to find optimum values for one of 6 neural networks prepared with Statistica Neural Networks 4.0F software. The networks were implemented using C++ language and were incorporated into the software as functions. Each of the functions assumes as input parameters a table consisting of 21 elements representing input variables of the selected neural network. A number representing an output variable of the neural network is the result of each of such functions. A set of 21 input parameters is divided into seven groups each containing three elements. Each of the groups corresponds to a single analysed thematic area, respectively between M1 to M7 and between P1 to P7, depending on the network selected. Three elements inside the group mean, respectively: a probability that a growth trend occurs, a probability that a stabilised trend occurs at the existing level and a probability that a falling trend occurs for the individual groups of technologies in relation to the overall surface engineering technology. Each input parameter of the function may assume values ranging between 0 to 100. The programme enables to set a minimum and maximum search value individually for each input variable. A sum of three probabilities of the occurrence of the individual trends must equal 100. Approx. 5000 combinations meeting the condition of the sum of three probabilities equal to 100 is obtained for the range of 0 to 100 for each thematic area. Due to a very high number of possible combinations a random search method (Monte Carlo) was applied. It takes on average 200 ms to check the 10^4 combinations. An increase in computation time according to the number of iterations is presented on the chart (Fig. 6). Time measurements were made with a computer equipped with an Intel Core i5 M560 2.67Ghz CPU and 4 GB RAM memory. A collection of solutions approximate to optimum solutions is obtained after checking about

10^7 iterations. The subsequent iterations have a minimum impact on the improvement of the results obtained, therefore it is groundless to perform them considering that the time necessary for performing them is rising exponentially.

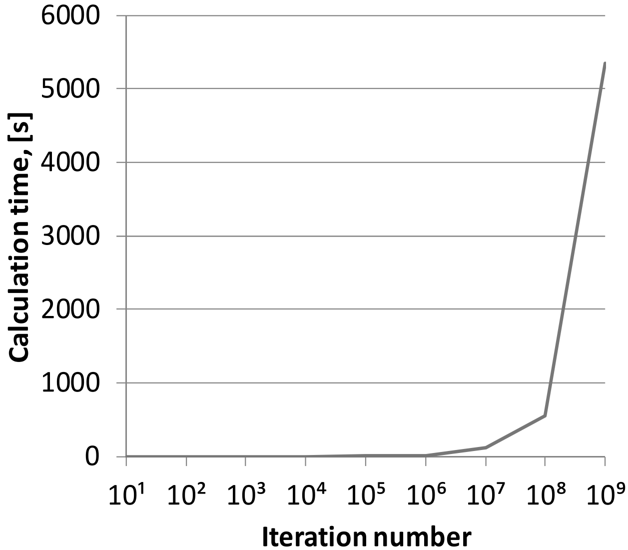


Figure 6. *Chart illustrating dependency between computation time and the number of iterations performed*

The working principle of SCENNET21 software is shown in a flow chart presented in Fig. 7. The user sets search parameters in the first place, including: the number of solution search iterations, the network to be the objective function, the ranges of input variables and the value expected for a network output variable. The programme, using the input data entered, calculates the width of search ranges and then all the possible combinations of probabilities for each thematic area giving the sum of 100. The simulations are performed by assuming that the search area for the values of decision variables will be limited to the scope of changes defined in the constraints of neural models. Another step is to sample seven combinations of probabilities recorded in the previous step. A function representing a neural network is next activated where the input parameters are the seven combinations of the numbers sampled in the previous step. A network output value is recorded in the "store" table. The worst value is then removed from the "store" table. The previous four stages are repeated until the loop counter equals the number of iterations pre-defined by the user. Once the calculations are completed, the results are shown as tables. The results can be exported to a spreadsheet (a file with csv extension) and can be presented as charts recorded as a graphical file with svg extension. The programme additionally features a function of generating chart labels in Polish and English and is recording charts in the colour and monochromatic version.

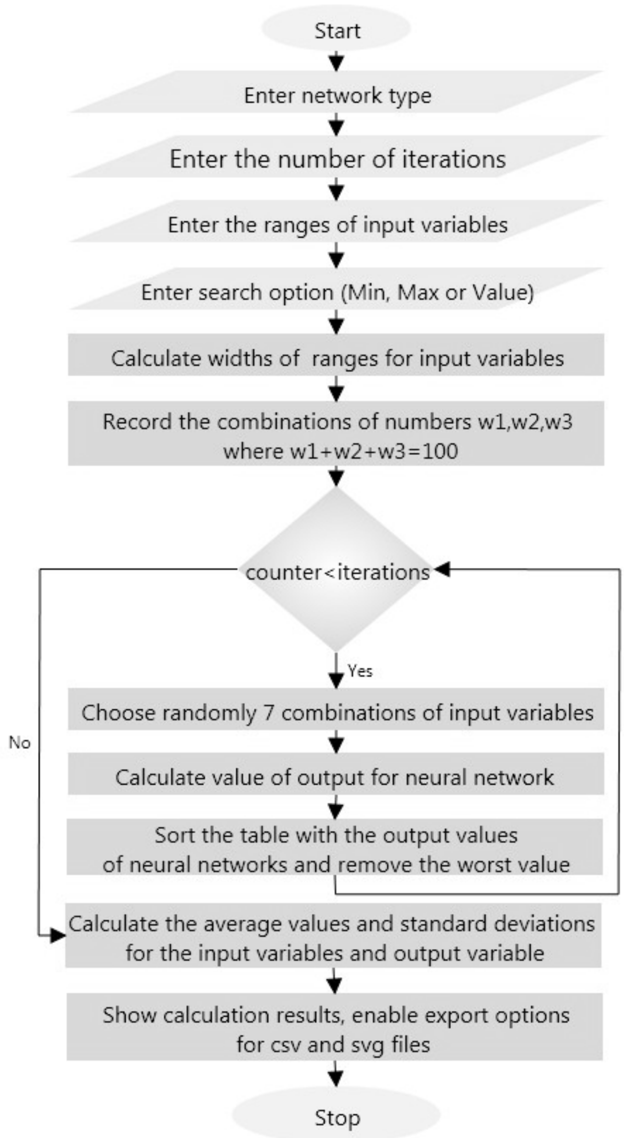


Figure 7. *SCENNET21 software flow chart*

A modification of SCENNET21 software is SCENNET48 software using its motor. SCENNET48 software calculates optimum values for the subsequent three neural networks to identify which of the mesofactors determining the future development of materials surface engineering (e.g. collaboration between science and industry, the number of specialised laboratories and R&D institutions, continuous improvement and high quality of technology, transparent and friendly legislation, international co-operation and EU funds). The subgroup elements, similar as in SCENNET21 software, specify the subsequent probabilities that the relevant trends occur, the sum of which needs to be equal to 100. The programme working algorithm differs in the number of the combinations of the probabilities sampled. The results of calculations are presented as a table. SCENNET48 software features the same export options as its predecessor. Fig. 8 shows an example of a window of SCENNET48 showing parts of calculations carried out for an optimistic scenario with a 20% probability defined by the user and the number of iterations equal to 100 000.

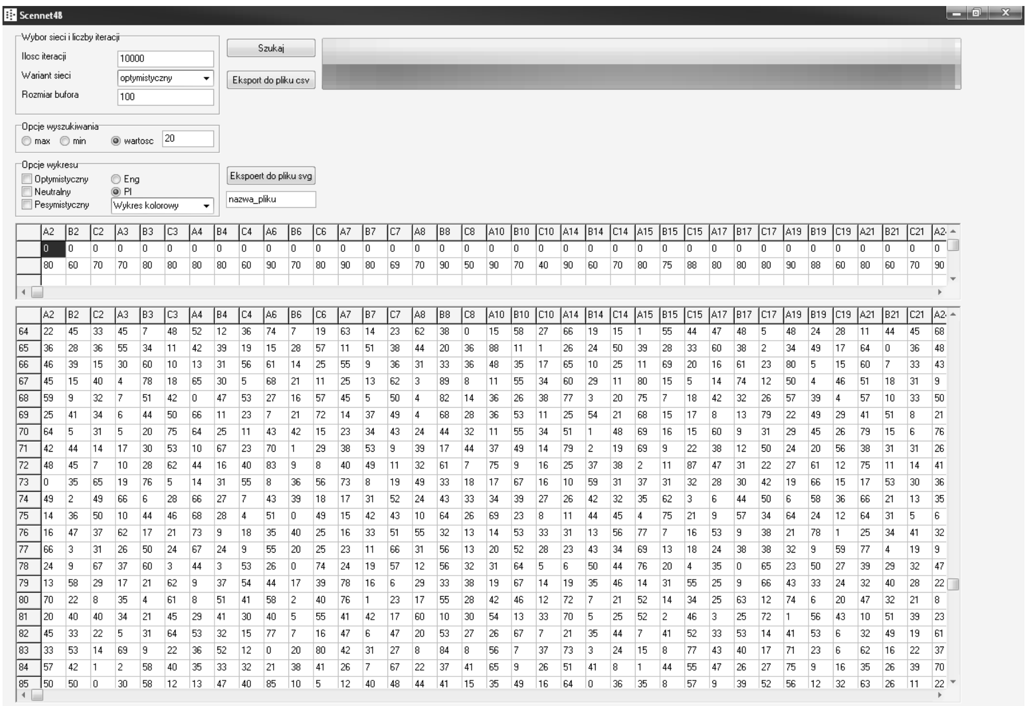


Figure 8. *Examples of SCENNET48 software window presenting the results of a simulation made using neural networks*

The purpose of the computer simulations performed using SCENNET21 software was to present to what degree the development of the individual thematic areas forming part of the research field (M) Manufacturing and (P) Product influences the occurrence, with a specific probability, of one of the three scenarios. A representative chart for the research field (M) generated by SCENNET21 software for an optimistic scenario which, according to the experts, can occur with a probability of 30%, is shown in Fig. 9. Probability that a growth trend, a trend stabilised at the current level and a falling trend occurs was determined on the axes of abscissa in percents. The relevant, analysed thematic areas are provided on the axis of coordinates, i.e., respectively: (M1) Laser technologies in surface engineering, (M2) PVD technologies, (M3) CVD technologies, (M4) Thermochemical technologies, (M5) Polymer surface layers, (M6) Nanostructural surface layers, (M7) Other surface engineering technologies. The results presented of a computer simulation performed using neural networks indicate that the following, respectively, has a substantial positive impact on the development of materials surface engineering in general and the implementation of an optimistic macroscenario likely to occur with a 30% probability: Nanostructural surface layers (M6), Laser technologies in surface engineering (M1), PVD technologies (M2) and the Polymer surface layers (M5). The same results for the second research field of (P) Product are shown in Fig. 10. The following thematic areas are provided on the axis of coordinates, respectively: (P1) Surface engineering of biomaterials, (P2) Surface engineering of structural metallic materials, (P3) Surface engineering of structural non-metallic materials, (P4) Surface engineering of tool materials, (P5) Surface engineering of steels used in automotive industry, (P6) Surface engineering of glass, micro- and optoelectronic and photovoltaic elements, (P7) Surface engineering of polymers. The results of the simulation made indicate that the following have the largest positive impact on the development of materials surface engineering in general and on the implementation of the optimistic macroscenario that occurs with a 30% probability, respectively: Surface engineering of, respectively: (P6) glass, micro- and optoelectronic and photovoltaic elements, (P1) biomaterials and (P4) tool materials.

The simulations aimed at determining to what degree the selected critical mesofactors impact the occurrence, with a specific probability, of one of the three macroscenarios, were carried out with SCENNET48 software. The results of the computer simulations carried out with neural networks point out that the following factors have mainly a considerable, positive impact on the development of materials surface engineering in general and on the implementation of the optimistic macroscenario that may occur with a 30% probability. The factors include: collaboration between science and industry and the growing importance of nanomaterials and graded materials in relation to other materials surface engineering technologies; the number of specialised laboratories and R&D institutions; continuous improvement and high quality of technology; transparent and friendly legislation; international co-operation and EU funds as well as the rising significance and strengthening of the technologies ensuring mechanical, tribological and anticorrosive properties.

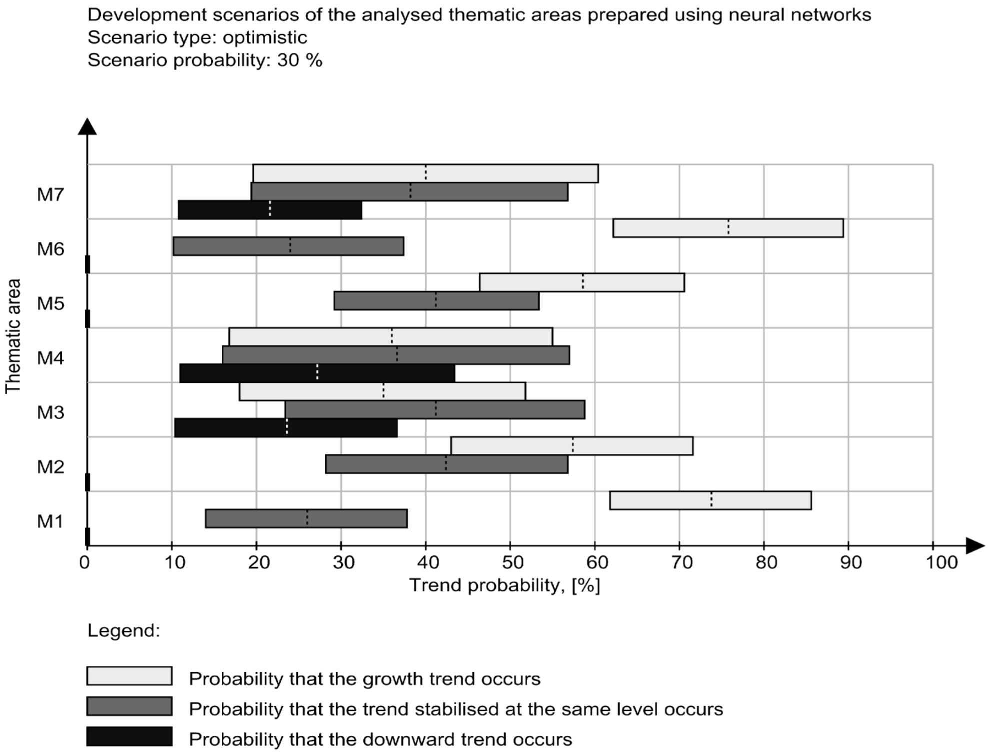


Figure 9. *Results of simulations that use neural networks, presenting the probability values for the occurrence of an increase, stabilised and decrease trend for the thematic areas of the (M) research field if the optimistic scenario takes place with a 30% probability*

The experiments made have shown that it is substantiated to use neural networks for analysing cross impacts between events when creating the multi-variant probabilistic scenarios of future events. By using this simulation tool, a dependency can be identified between the occurrence, with a specific probability, of each of the considered alternative macroscenarios and the variants of changing the individual mesofactors while taking into account that within the defined time horizon they can increase, remain stable or decrease. All the difficulties encountered by pioneers have been related to the experimental and innovative idea of implementing neural networks for creating the scenarios of future events. The chief challenge was the specificity of the input data in form of expert opinions expressed quantitatively with the universal scale of relative states. The phenomena were assessed differently because such assessment tends to be subjective, which is typical for expert investigations. In particular the experts, most likely unintentionally, on one hand were making an attempt to represent the interests of their own circles and on the other hand were frequently viewing those phenomena having a high level of generality (macro- and mesoscale) through the prospective of their own, much narrower, specialisation.

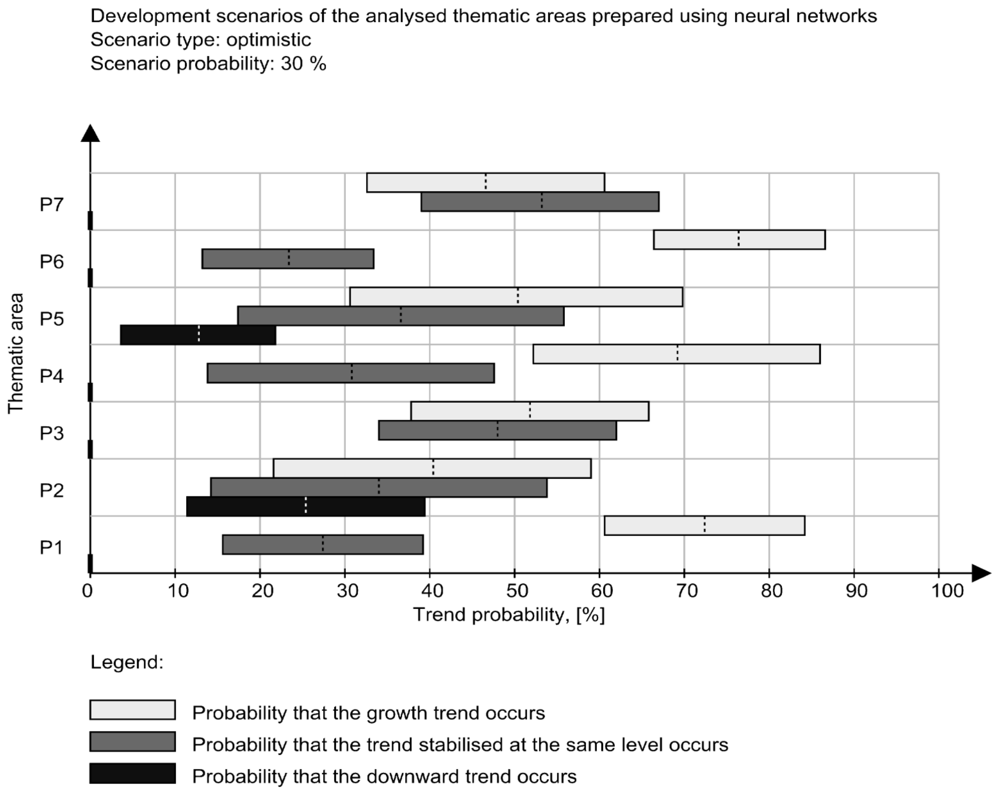


Figure 10. *Results of simulations that use neural networks, presenting the probability values of the occurrence of an increase, stabilised and decrease trend for the thematic areas of the (P) research field if the optimistic scenario takes place with a 30% probability*

4. Forecasted laser surface treatment progress

The anticipated development and strategic position of laser technologies in respect of materials surface engineering was identified using the reference data acquired whilst performing technology foresight for materials surface engineering [6]. Over 300 independent experts from many countries representing scientific, business and public administration circles have taken part in the FORSURF technology foresight. The experts have completed approx. 650 multi-question surveys and held thematic discussions during 10 expert panels. A collection of 140 critical technologies, 10 for each thematic group, was selected for the above 14 thematic groups from the initially inventoried approx. 500 specific technology groups. The scientific and research methods of evaluating the state of the art for a particular concept, technology review and a strategic analysis with integrated methods were used for this purpose, including: extrapolation of trends, environment scanning, STEEP analysis, SWOT analysis, expert panels, brainstorming, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and static analysis. 10 critical technologies were selected within the group of 14 thematic areas as a result of the efforts undertaken. A collection of 140 critical technologies was thoroughly analysed according to three iterations of the e-Delphix method performed according to the idea of e-foresight [35]. Laser technologies in surface engineering was one of the 14 thematic areas analysed as part of the foresight research.

Foresight investigations with the sample size of 198 have revealed a very robust strategic position of laser technologies among other materials surface engineering technologies. The experts found that that laser technologies have the best industrial application prospects in the group of all the analysed materials surface engineering technologies in the nearest 20 years. 78% of the surveyed held such a view. Nearly a three fourth of the respondents (73%) maintain that numerous scientific and research studies will be devoted to such technologies in the analysed time horizon. 70% of the persons surveyed claim that the thematic area of “Laser technologies in surface engineering” is crucial and its importance should be absolutely rising so that an optimistic scenario can come true of the country’s/Europe/World development, i.e. “Race won” assuming that the potential available is adequately utilised to fulfil the strategic objectives of development and so that people, statistically, are better off, social attitudes are optimistic and the prospects for the coming years bright. 81% of the surveyed persons argue that the significance of laser technologies in relation to other materials surface engineering technologies will be growing, whereas 18% maintain it will remain on the same level with only

3 individuals asserting that the role will diminish over the next 20 years. The excellent results of technology foresight elaborated based on the reference data point to, therefore, the anticipated key role of laser technologies for the advancement of the overall materials surface engineering (mesoscale) and for the development of the entire domestic/ European/ global economy (macroscale) [18] The described technology foresight outcomes prepared based on the opinions of the experts expressed during the research with the e-Delphix method presenting a position of laser technologies against materials surface engineering in general are shown in Fig. 11.

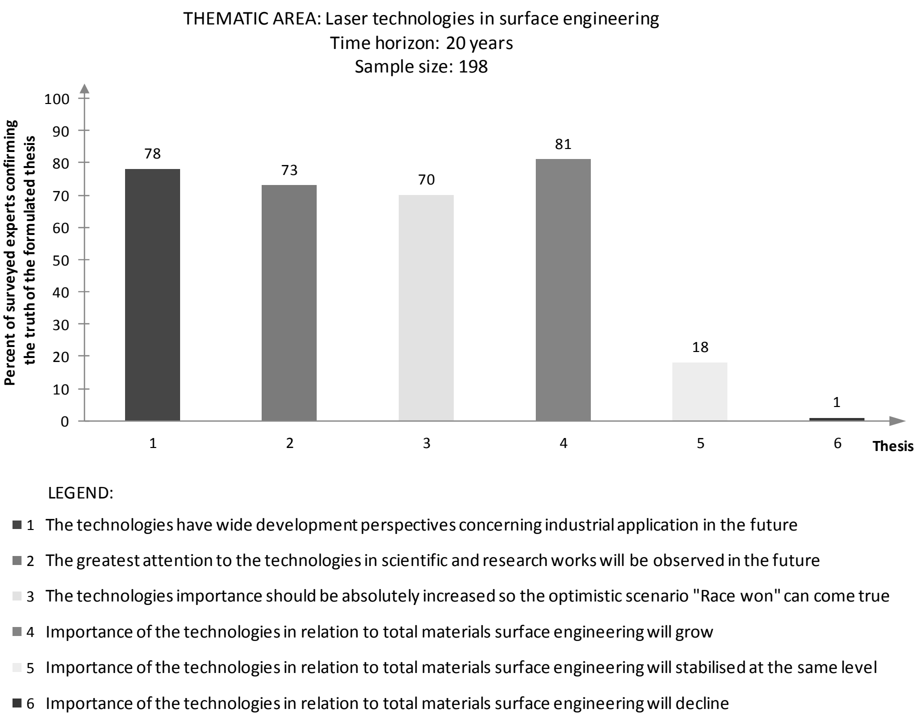


Figure 11. *Strategic position of laser technologies versus other materials surface engineering technologies*

5. Strategic position of critical laser technologies in surface engineering

A state of the art analysis undertaken in the initial stage of the foresight research including a diagnosis of the state of the art, technology review and a strategic analysis with integrated methods enabled to choose 10 critical technologies for each of the 14 thematic areas analysed, including the area of "Laser technologies in surface engineering". Critical technologies should be understood as priority technologies with the best development outlooks and/or of key significance for the industry within the analysed time horizon of 20 years. The data required for determining development tendencies and a strategic position of the individual groups of critical technologies for laser surface treatment was acquired via electronic surveys completed by the experts. The surveys were prepared using the e-Delphix method according to the guidelines of e-foresight [35]. A selected group of the key experts being specialists in the field of laser technologies assessed the relevant groups of critical technologies according to a universal scale of relative states where 1 is a minimum rate and 10 an extraordinarily high one. A set of unprocessed primary data was analysed. The analysis results were shown graphically using a pool of foresight matrices [36]. The following groups of critical technologies of laser surface treatment were examined in particular:

(A) Laser heat treatment,
(B) Laser remelting,
(C) Laser alloying / cladding,
(D) Laser cladding,
(E) Laser additive manufacturing (e.g. LENS),
(F) Laser Chemical Vapour Deposition (LCVD),
(G) Laser Assisted Physical Vapour Deposition (LAPVD),
(H) Laser treatment of functional materials (e.g. polycrystalline laser texturing in photovoltaics),
(I) Pulsed Laser Deposition (PLD),
(J) Laser treatment of biomaterials.

According to the methodology established, a strategic position of each of the analysed critical technologies of laser surface treatment is presented graphically using the matrix of strategies for technologies made up of sixteen fields. The matrix presents, graphically, a position of each group of technologies according to its value and environment influence intensity and identifies a recommended action strategy. The matrix contains the results of the expert investigations visualised with the dendrological and meteorological matrix transformed by means of software created for this purpose. The methodological structure of the both matrices is referring to the portfolio methods commonly known in management sciences, and first of all to the BCG matrix [37], enjoying its unique popularity due to a reference to simple associations and intuitive reasoning, becoming an inspiration when elaborating methodological assumptions for the both matrices. A four-field dendrological matrix of the technology value includes the expert assessments for the relevant technologies according to the potential being the actual objective value of the specific technology group and according to attractiveness reflecting the subjective perception of the relevant technology group by its potential users. A four-field **matrix of environment influence** presents, in a graphical manner, the results of how the external positive (opportunities) and negative (difficulties) factors influence the technologies analysed [36].

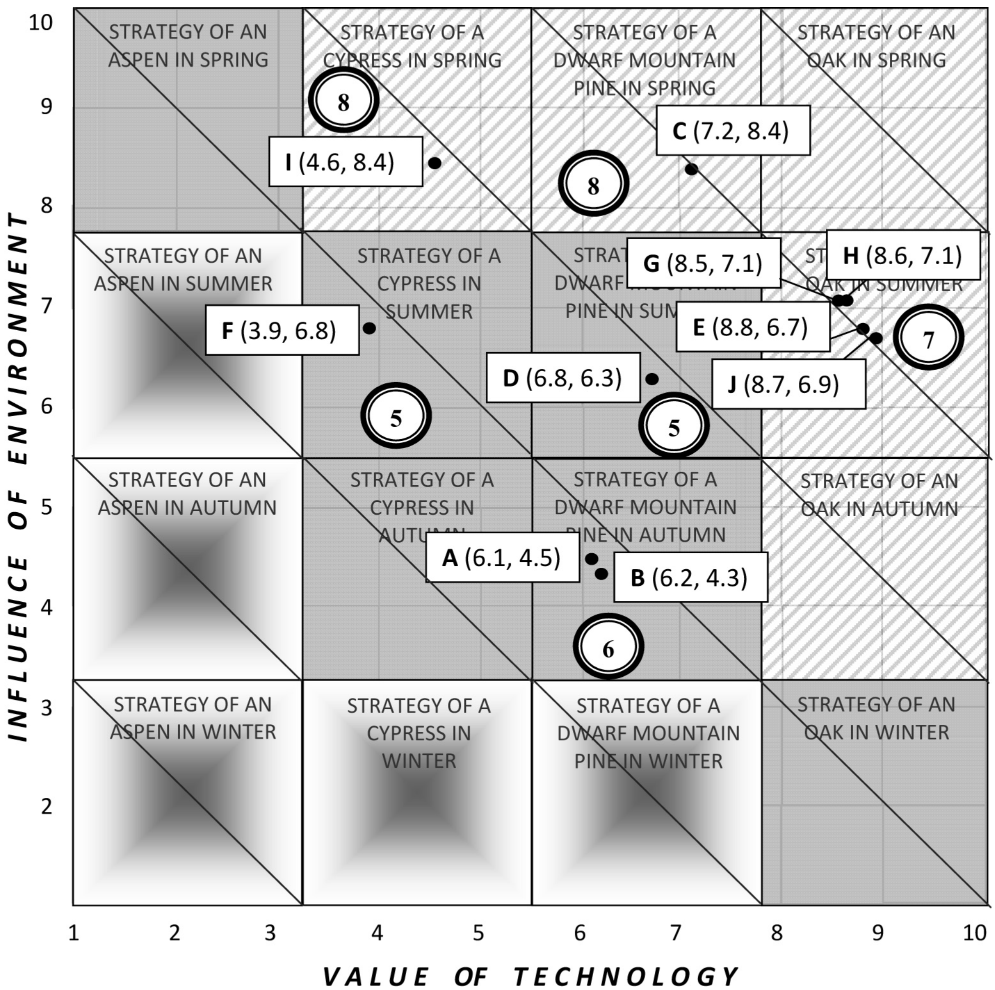


Figure 12. *A matrix of strategies for technologies prepared for the thematic field of "Laser technologies in surface engineering"*

A matrix of strategies for technologies developed for the thematic area of "Laser technologies in surface engineering" is provided in Fig. 12. The circles mark the strategic development prospects of a given group of technologies expressed in numbers using the universal scale of relative states.

The development outlooks of the technology group (C) corresponding to laser alloying / cladding was highly evaluated in a ten-degree scale, by awarding 8 points. The technology group (C) was placed in the dwarf mountain pine in spring field signifying its high potential and limited attractiveness. It is hence recommended for the group to make the technology more attractive, more modern, to automate it, to computerise it and to promote it using the strong market conditions. Pulsed Laser Deposition (I) also was awarded 8 points and was placed in the cypress in spring field meaning that this technology group is highly attractive and its potential needs to be strengthened. For this purpose, the technology has to be researched, improved and invested in further in the strong market conditions. As regards the very promising experimental or prototype technologies that were given 7 points, i.e. laser treatment of functional materials (H) and biomaterials (J) and Laser additive manufacturing (E) and also Laser Assisted Physical Vapour Deposition (G) being in their early-mature phase of the lifecycle, the oak in summer strategy is recommended. The strategy provides that the technology's attractiveness and potential should be exploited in the risky environment of fierce global competition. In addition, opportunities should be sought for and difficulties avoided and the technology should be intensively promoted with such measures being preceded by marketing research in order to tailor the product to a customer's demands as far as possible. The development prospects of the base groups of technologies, i.e. (A) laser heat treatment and (B) laser remelting were found to be moderate (6). It was recommended that profits should be derived from production conducted in a stable, predictable environment using a robust technology that should be modernised and intensively promoted to enhance its appeal. The group of the base technologies (D) of laser cladding was placed in the field of the dwarf mountain pine in summer, and the action recommended for the technology is to make this technology, having a large potential, more attractive and modern and to tailor the technology to a client's requirements according to the results of marketing research. Laser Chemical Vapour Deposition (F) was placed in the cypress in summer field implying that the potential of this attractive technology group should be enhanced in the risky conditions of the environment and that the risk should be appraised and, depending on the outcome, either a customer should be aggressively fought for or the technology should be phased out from the market slowly. The environment of the base group of technologies (D) and of the early-mature group of technologies (F) is a stormy one, therefore, one should not preclude that either positive or negative surprise scenarios of their development should take place.

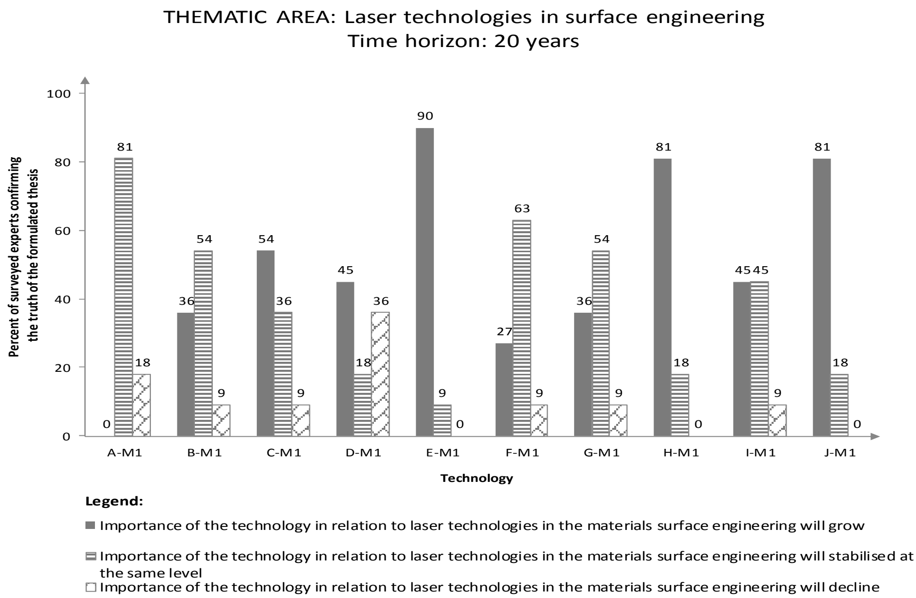


Figure 13. *Expert opinions presenting the expected increase, stabilisation and decrease in the importance of the individual groups of critical laser surface treatment technologies in relation to the thematic area of "Laser technologies in surface engineering"*

The experts surveyed, as part of the research conducted with the e-Delphix method, also identified the forecast development trends of the relevant critical technologies of laser surface treatment against the thematic area of "Laser technologies in surface engineering". Laser additive manufacturing (E), the importance of which against other laser surface treatment technologies should be rising according to 90% of experts, was ranked highest. Pulsed Laser Deposition (I) and laser treatment of biomaterials (J) also enjoyed high rates. 81% of the experts claim that the importance of those groups of technologies will be growing over the nearest 20 years. The statement presented shows that the future of laser cladding (D) is most uncertain as 36% of the experts maintain that the role of this technology group will be dwindling, 45% of them think the role will be on the rise and 18% assert that the role will maintain at the same level. Fig. 13 lists in detail all the expert opinions reflecting the anticipated growth, stabilisation and decline in the importance of the relevant groups of critical laser surface treatment technologies with respect to the thematic area of "Laser technologies in surface engineering".

6. Hot-work steels laser treatment today and in the future

6.1. Materials and methodology

The long-run development prospects of hot-work alloy tool steels subjected to laser treatment using the custom methodology [36] were identified based on interdisciplinary research including materials science experiments, notably light and scanning microscopy, X-ray phase qualitative analysis and investigations into mechanical and functional properties (hardness, microhardness, roughness, wear strength, thermal fatigue strength) as well as expert studies. The samples of hot-work alloy tool steel X40CrMoV5-1 and 32CrMoV12-28 with their chemical composition given in Table 4 were used for own investigations. The samples underwent typical heat treatment, i.e. they were quenched and tempered twice, and then remelted (without using powders) and laser alloyed. Carbide powders with their properties listed in Table 5 were deposited onto the samples prior to laser alloying. Fig. 14 shows a photo of one of the powders used for alloying, i.e. tungsten carbide WC. A ROFIN SINAR DL 020 High Power Diode Laser (HPDL) was applied for the laser remelting and alloying of the steels with carbide powders. The technical specifications of the laser are given in Table 6. Six homogenous groups were distinguished between for the analysed technologies by using the type of powder deposited onto the surface or the lack of such powder as a comparative analysis criterion, including, respectively:

(K) Remelting of hot–work alloy tool steels (without powders),
(L) Alloying of hot–work alloy tool steels using the NbC niobium carbide,
(M) Alloying of hot–work alloy tool steels using the TaC tantalum carbide,
(N) Alloying of hot–work alloy tool steels using the TiC titanium carbide,
(O) Alloying of hot–work alloy tool steels using the VC vanadium carbide,
(P) Alloying of hot–work alloy tool steels using the NbC WC tungsten carbide.

Table 4. *Chemical composition of the examined hot-work alloy tool steels*

Steel grade	Mass concentration of elements, %								
	C	Mn	Si	P	S	Cr	W	Mo	V
X40CrMoV5-1	0.41	0.44	1.09	0.015	0.010	5.40	0.01	1.41	0.95
32CrMoV5-1	0.308	0.37	0.25	0.020	0.002	2.95	–	2.70	0.535

Table 5. *Selected properties of powders used for laser treatment*

Coating type	Hardness HV, GPa	Melting point, °C	Density, g/cm^3	Thermal expansion coefficient α, $10^{-6} \cdot K^{-1}$
WC	2400	2730-2870	15.77	23.8
NbC	1800	3480-3610	7.6	7.6
VC	2600	2650-2830	5.81	7.5
TiC	3200	3065-3180	4.94	8.3
TaC	1600	3780-3985	14.5	7.8

Table 6. *Technical data of HPDL ROFIN DL 020 diode laser*

Parameter	Value
Laser wave length, nm	940 ± 5
Power, W	100-2300
Focus length of the laser beam, mm	82/32
Power density range of the laser beam in the focus plane, kW/cm^2	0.8-36.5
Dimensions of the laser beam, mm	1.8-6.8 with 82 mm focus length

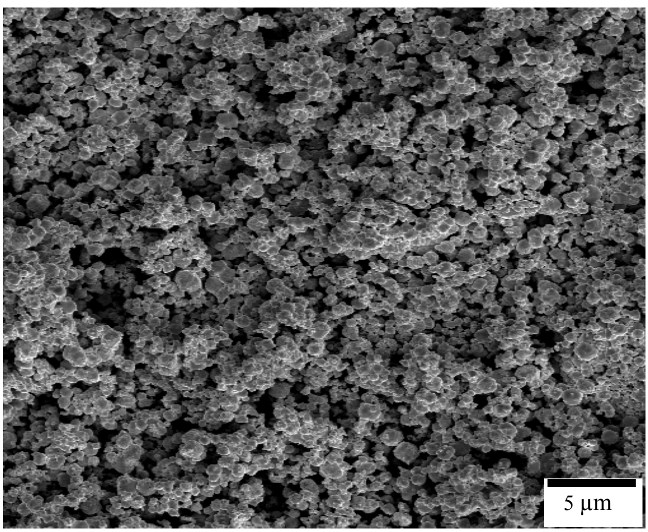


Figure 14. *Topography of the WC tungsten carbide powder used for steel alloying (SEM)*

The structure of the tested steels was observed with a Leica MEF4A light microscope with the magnification of: 25-1000 x and with a DSM-940 electron scanning microscope by Opton with the accelerating voltage of 20 kV. The structures were photographed with a Leica – Qwin computer-aided image analysis system and the following was measured: the depth of the remelted zone (RZ), of the heat-affected zone (HAZ) and the width of the bead appearance. Hardness measurements were taken with the Rockwell's method with a Zwick ZHR 4150TK hardness tester. The microhardness of the samples remelted and/or alloyed with laser was measured with the Vickers method with a DUH 202 Shimadzu ultramicrohardness tester. R_a, μm, roughness was measured with a Surtronic 3+ contact profilometer by Taylor - Hobson. The tests of abrasive wear resistance with the metal – ceramic material method were carried out using a specially constructed stand. Four remelting and/or alloying beads were made on each sample within the laser power range of 1.2-2.3 kW. Fig. 15 shows an example of a X40CrMoV5-1 steel view after alloying with tungsten carbide [38].

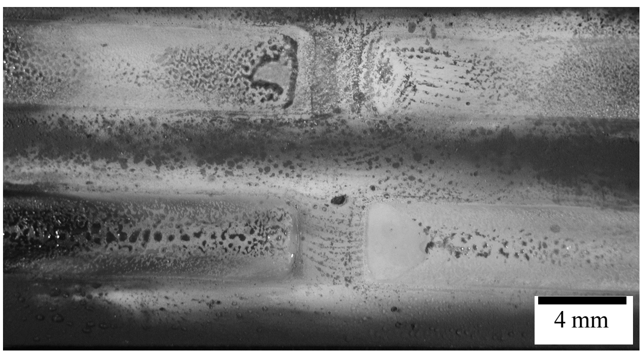


Figure 15. *View of the X40CrMoV5-1 hot-work alloy tool steel after alloying with the WC tungsten carbide, laser power of 1.2-2.3 kW*

The results of the strategic position evaluation for the relevant analysed technologies made according to the opinions of the key experts expressed with the universal scale of relative states (1 - min., 10 - max.) is presented in the graphical version with a set of foresight matrices [30]. The individual technologies, differing in the type of the powder deposited onto the substrate or the lack of it, were assessed for their potential and attractiveness. The results were placed into the dendrological matrix of the technology value. The results of the evaluation for the influence of external positive factors (opportunities) and negative factors (difficulties) on the technologies analysed were entered into the metrological matrix of environment influence.

The results of expert investigations were at the next stage of the works entered into the matrix of strategies for technologies presenting graphically the place, expressed in numbers, of each technology group considering its values and environment influence intensiveness and by indicating a recommended action strategy. Strategic development tracks according to three variants reflecting the predicted position of a specific technology for individual time intervals were also entered into the matrix. A group of technology roadmaps was established at the last stage of the works, based on the results of foresight-materials science research, being a graphical comparative analysis tool enabling to choose the best technology in terms of the materials science, technological or economic criterion selected.

6.2. Structure and properties of leaser treated hot-work steels

A remelted zone (RZ), heat-affected zone (HAZ) as well as transition boundaries between the remelted zone and the heat-affected zone and between the heat-affected zone and the native material are formed in each remelted and/or alloyed surface layer of the examined steels [39-41]. The examples of surface layers after alloying X40CrMoV5-1 steel with tungsten carbide with the laser power of 1.2 kW and 32CrMoV12-28 steel with the laser power of 2.0 kW are shown in Fig. 16 and Fig. 17. The thickness of the remelted zone and the heat-affected zone depends on the laser beam power. If the rate of alloying and the thickness of the alloying layer is constant, zone thickness increases along with higher laser power [42, 43]. The surface layer formed in the remelting process is characterised by lower roughness. An increase in surface and roughness irregularity was, however, found for the steel alloyed with carbide powders.

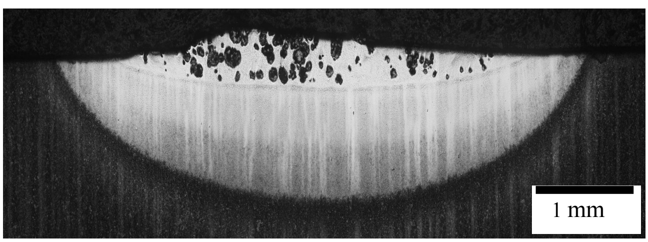


Figure 16. *Surface layer of the X40CrMoV5-1 steel after alloying with WC, the laser power of 1.2 kW*

Figure 17. *Surface layer of the 32CrMoV12-28 steel after alloying with WC, the laser power of 2.0 kW*

a)

b)

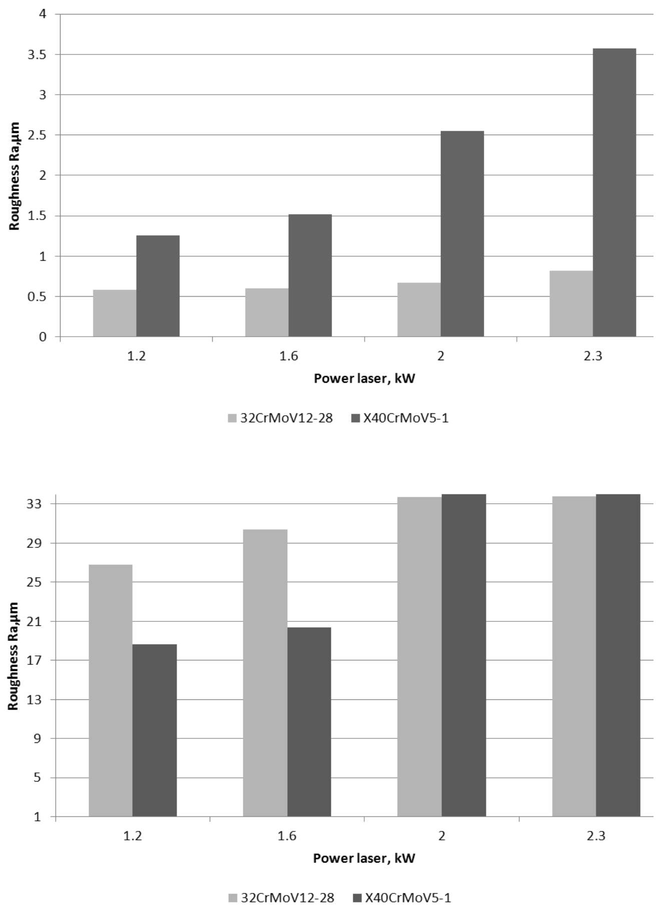


Figure 18. *Effect of laser power on the roughness of the X40CrMoV5-1 and 32CrMoV12-28 surface layers a) remelted with laser, b) alloyed with laser with the WC tungsten carbide; laser power of 1.2-2.3 kW*

This stems from the fact that the alloying material is fluctuating due to the varied surface tension of the material being remelted and by the laser radiation energy being absorbed by the alloying material. Fig. 18a shows how laser power influences the roughness of the remelted surface layers of the X40CrMoV5-1 and 32CrMoV12-28 steels, and Fig. 18b shows the same for those alloyed with tungsten carbide WC.

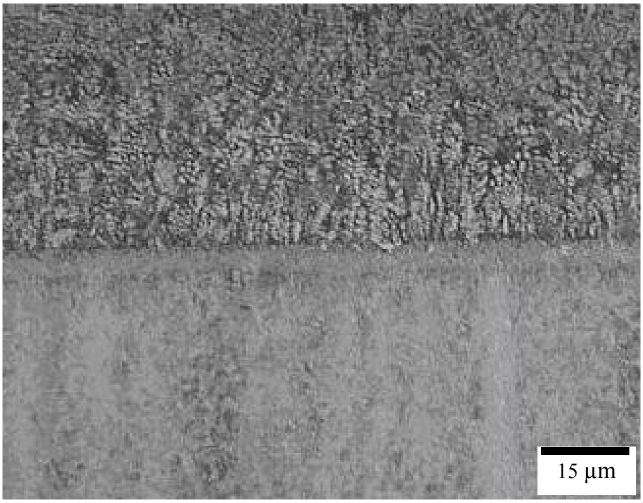


Figure 19. *Remelting toe of the X40CrMoV5-1 surface layer after the NbC niobium carbide alloying, laser power of 2.3 kW*

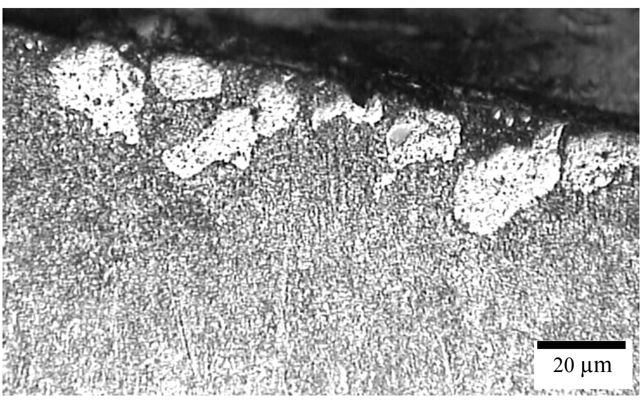


Figure 20. *Remelting limit of the 32CrMoV12-28 surface layer after the TaC tantalum carbide alloying, laser power of 1.6 kW*

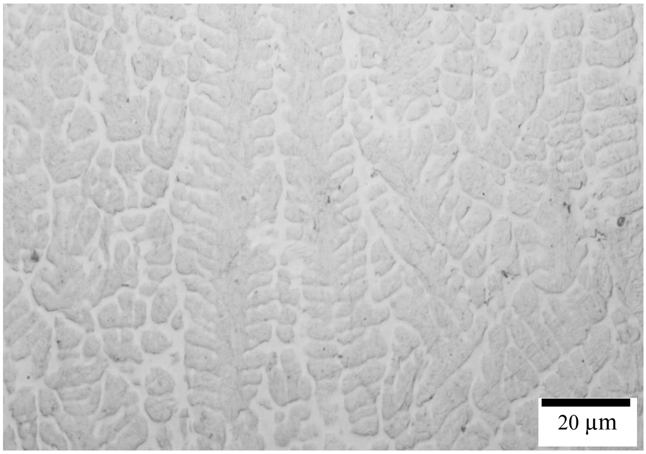


Figure 21. *Central zone of the X40CrMoV5-1 remelted surface layer after the TiC titanium carbide alloying, laser power of 1.2 kW*

It was found based on observations with a light microscope that the structure of steel after remelting and after laser alloying is characterised by the occurrence of areas with a highly varied morphology connected with material solidification. Heat in the central area of the remelted zone is evacuated in all directions and the structure thus formed is made of fine equiaxial crystals with a lattice of carbides [44-47]. Figs. 19-22 shows some examples of the remelting toe and the central zone of surface layer remelting for each of the steel grades analysed.

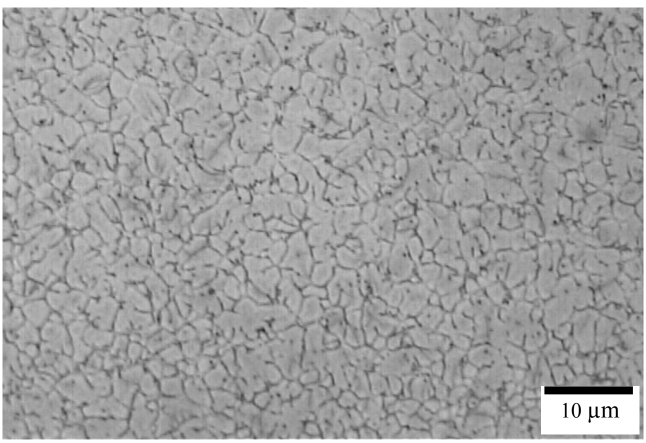


Figure 22. *Central zone of the 32CrMoV12-28 remelted surface layer after the VC vanadium carbide alloying, laser power of 2.0 kW*

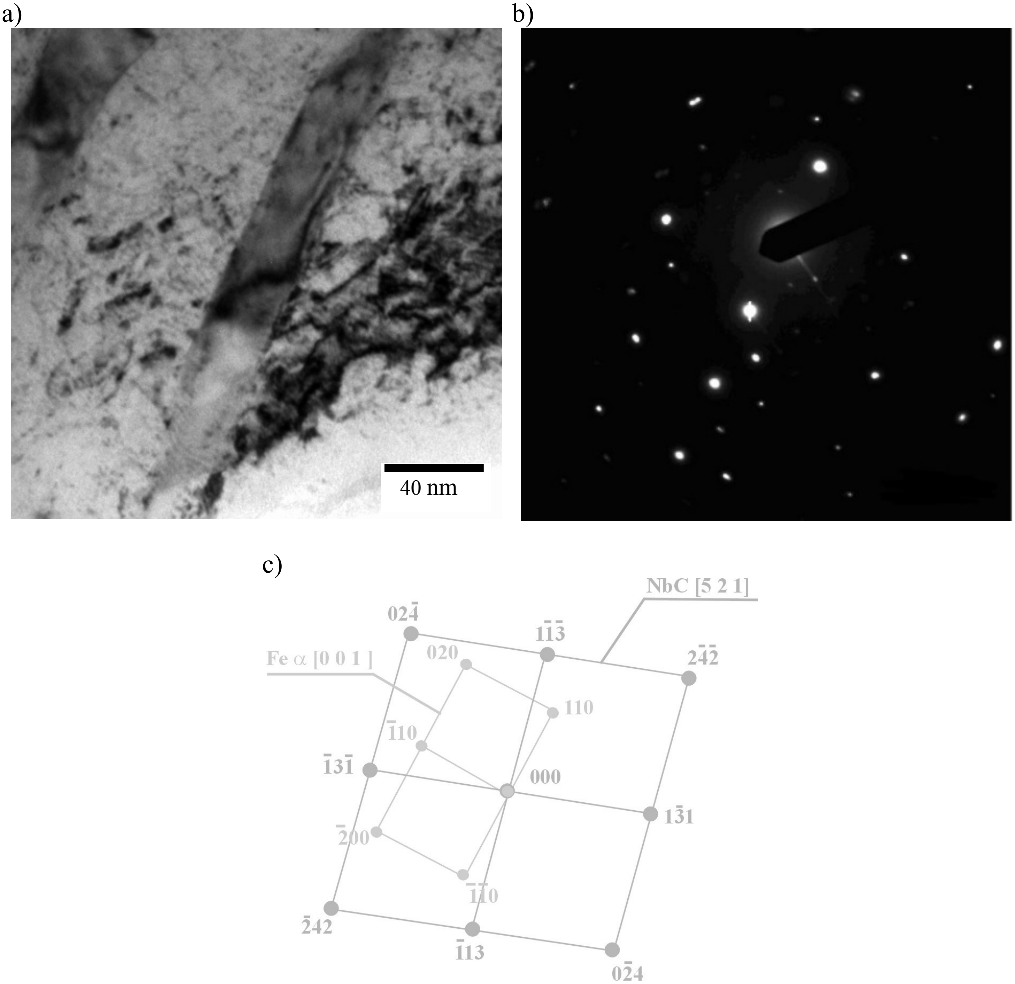


Figure 23. *Structure of thin foil made of the X40CrMoV5-1 steel after alloying with the NbC niobium carbide, laser beam power of 1.2 kW; a) image in bright field, b) diffraction pattern from the area as in figure a), c) diffraction pattern solution for figure b)*

It was found on the basis of investigations carried out for thin foils made of the X40CrMoV5-1 surface layer alloyed using laser with carbide powders that the relevant carbides used for alloying occur on the limits of grains (Fig. 23). Lath martensite with a high dislocation density constitutes the surface layer matrix after alloying [48, 49]. Fine carbides such as the M_3C or the M_7C_3 identified with the electrons diffraction method are also found in the martensite of the surface layer of the steel alloyed with laser [50, 51]. The investigations of thin foils made of the 32CrMoV12-28 laser-alloyed steel with carbide powders reveal the presence of the dispersive lattice of carbides, at the limits of grains mainly. They form as fine dispersive carbides in some cases inside the grains (Fig. 24).

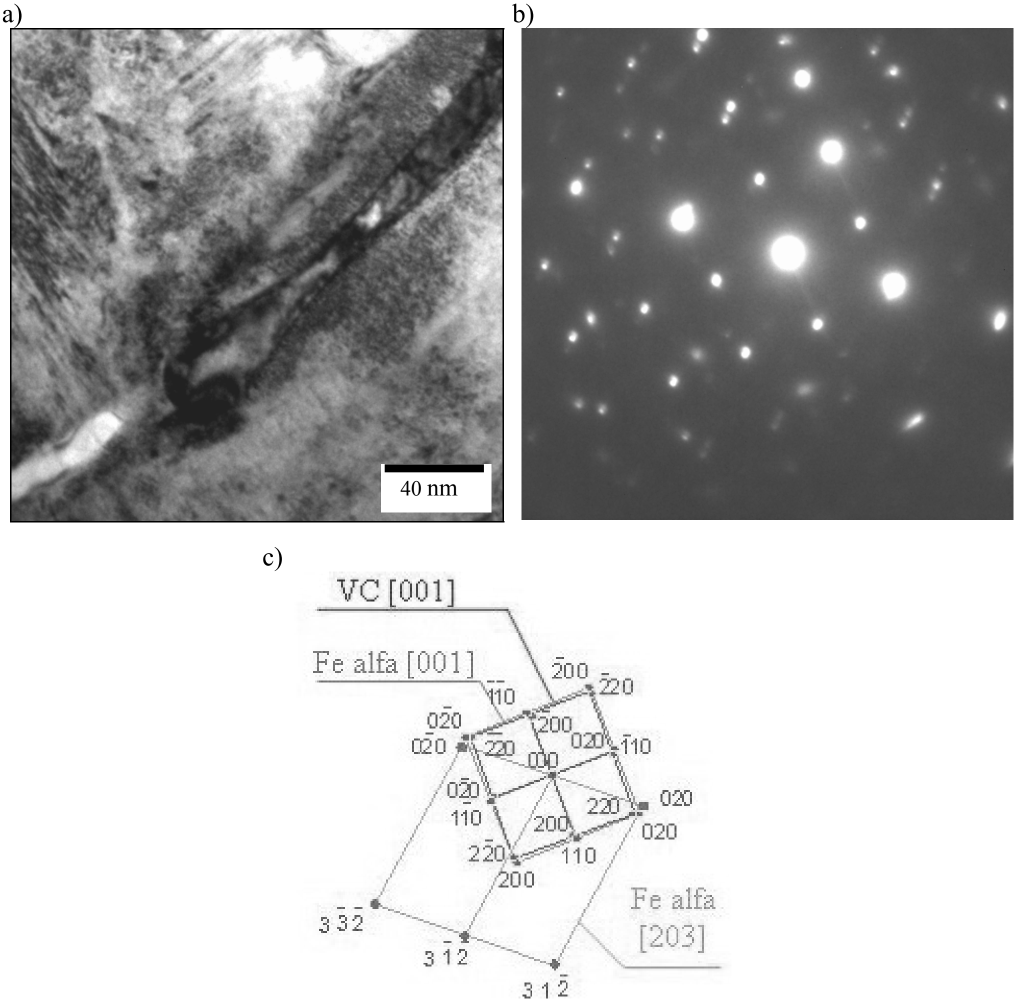


Figure 24. *Structure of thin foil made of the 32CrMoV12-28 steel after alloying with VC vanadium carbide, laser beam power of 1.6 kW, a) image in bright field, b) diffraction pattern from the area as in figure a), c) diffraction pattern solution for figure b)*

The selected mechanical and functional properties of hot-work tool steel subjected to laser remelting and alloying were also examined in the course of the materials science research. Their hardness, microhardness, roughness, wear resistance and thermal fatigue strength was examined in particular [52]. Laser treatment in the majority of cases improves the hardness of

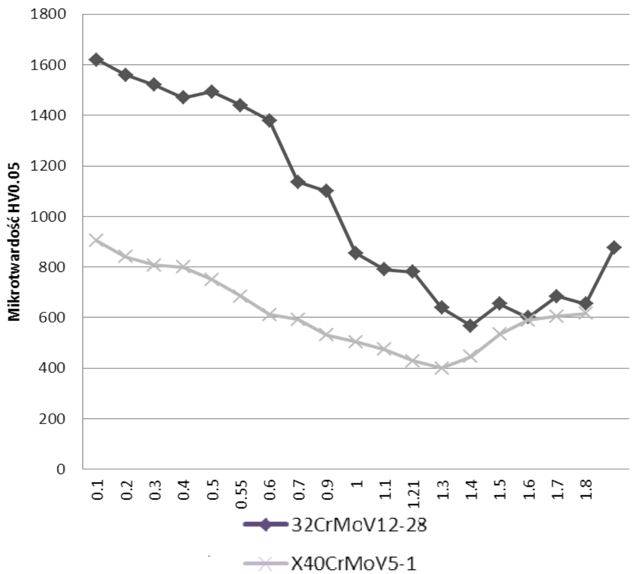


Figure 25. *Variations to the surface layer microhardness of the X40CrMoV5-1 and 32CrMoV12-28 hot-work alloy tool steels alloyed with laser using the TaC tantalum carbide; laser power of 1.6 kW*

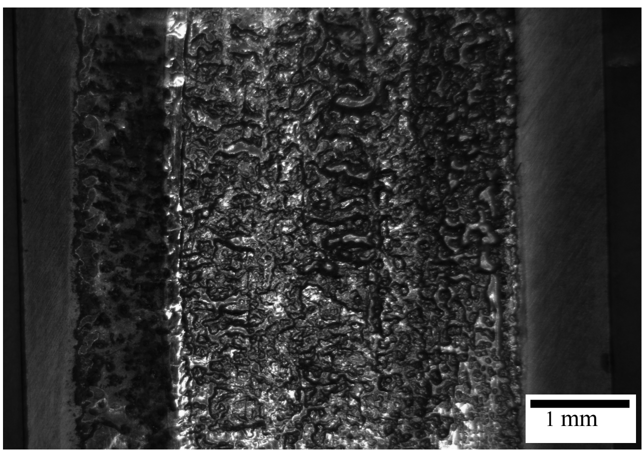


Figure 26. *Wear trace of the surface layer after an abrasion test acc. to ASTM G65 for the X40CrMoV5-1 steel alloyed with the WC tungsten carbide powder, laser power of 1.2 kW*

the investigated steels. The hardness of the surface layer alloyed with carbide powders is growing along with the higher power of laser used for alloying [53, 54]. The results of microhardness measurements at the lateral section of the surface layer according to distance from the surface of samples indicate that microhardness is growing in the majority of cases of laser remelting and/or alloying using carbide powders. An area was also identified in all the microhardness measurements of the surface layer of the heat treated, remelted and/or laser-alloyed steel where hardness is clearly decreasing. Such area is present along the whole width of the heat-affected zone limit and native material [55]. Such lower hardness is seen as a consequence of steel tempering during laser treatment when steel is heated to a temperature higher than the tempering temperature [56] (Fig. 25). The tribological properties of steel are rising along with the growing surface layer hardness after laser alloying. Figs. 26 and 27 illustrate the

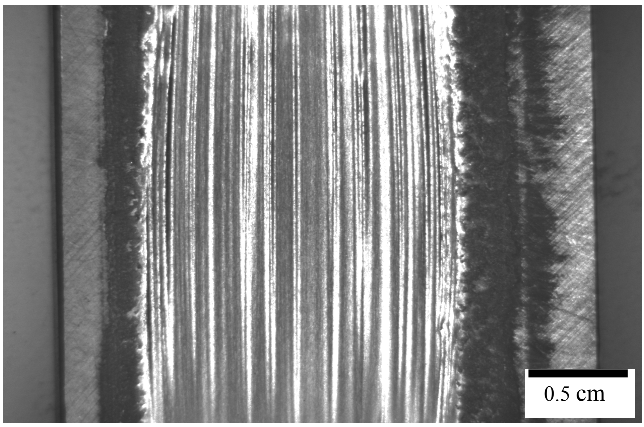


Figure 27. *Wear trace of the surface layer after an abrasion test acc. to ASTM G65 for the 32CrMoV12-28 steel alloyed with the TiC titanium carbide powder, laser power of 2.0 kW*

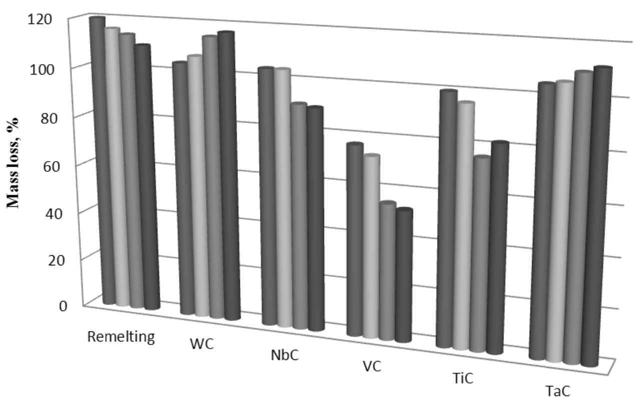


Figure 28. *A relative loss of mass measured when testing the wear resistance of the 32CrMoV12-28 steel remelted and alloyed by laser with carbide powders within the laser power range of, respectively: 1.2, 1.6, 2.0 and 2.3 kW*

wear traces of the surface layers of tool steels, respectively the X40CrMoV5-1 and the 32CrMoV12-28, after testing abrasibility acc. to ASTM G65. Wear resistance was also tested for the steels subjected to remelting and alloying with carbide powders [57-59]. Figs. 28 and 29 show a relative loss of mass measured when testing the wear resistance of X40CrMoV5-1 steel. The detailed research results for the mechanical and functional properties made for the X40CrMoV5-1 and the 32CrMoV12-28 steel remelted and alloyed with different carbide powders using laser with different power is listed in Table 8.

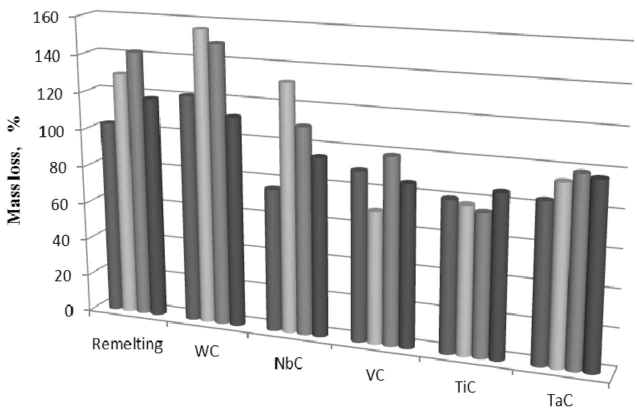


Figure 29. *A relative loss of mass measured when testing the wear resistance of the X40CrMoV5-1 steel remelted and alloyed by laser with carbide powders within the laser power range of, respectively: 1.2, 1.6, 2.0 and 2.3 kW*

Table 8. *Research results of mechanical and functional properties of hot–work alloy tool steels remelted and alloyed using HPDL*

Substrate material	Laser power of, kW	Surface layer remelting depth, mm	Roughness R_a, µm	Hardness HRC	Microhardness expressed in universal scale of relative states	Wear resistance *) (relative mass loss of sample), %	Thermal fatigue strength / Average depth of cracks, µm
(K) The laser remelting of hot-work alloy tool steels (without powders)							
X40CrMoV5-1	1.2	0.56	1.26	54.51	6	64	42
	1.6	1.03	1.52	56.00	3	78	39
	2.0	1.47	2.55	56.76	5	85	32
	2.3	1.67	3.57	57.71	6	70	28
32CrMoV12-28	1.2	0.52	0.58	53.20	1	120	76
	1.6	0.92	0.60	51.50	1	116	74
	2.0	1.13	0.67	44.63	2	114	63
	2.3	1.67	0.82	41.10	2	110	6

Substrate material	Laser power of, kW	Surface layer remelting depth, mm	Roughness R_a, µm	Hardness HRC	Microhardness expressed in universal scale of relative states	Wear resistance *) (relative mass loss of sample), %	Thermal fatigue strength / Average depth of cracks, µm
(L)	The laser alloying of the NbC niobium carbide powders in the surface of hot-work alloy tool steels						
X40CrMoV 5-1	1.2	1.43	12.13	58.37	8	72	no data available
	1.6	1.90	14.82	55.70	7	78	no data available
	2.0	2.64	23.18	56.64	9	67	no data available
	2.3	3.32	25.90	58.37	10	71	no data available
32CrMoV1 2-28	1.2	1.28	6.40	55.20	6	104	67
	1.6	1.74	9.80	56.10	7	104	54
	2.0	2.45	11.20	60.73	8	91	62
	2.3	2.61	18.20	60.66	9	90	51
(M)	The laser alloying of the TaC niobium carbide powders in the surface of hot-work alloy tool steels						
X40CrMoV 5-1	1.2	1.62	4.94	56.71	6	52.5	24
	1.6	2.33	5.40	58.82	8	56	25
	2.0	3.00	5.64	58.39	8	61	19
	2.3	3.52	8.66	60.26	9	55	18
32CrMoV1 2-28	1.2	0.99	6.80	65.06	9	105	no data available
	1.6	1.87	9.40	65.46	10	106	no data available
	2.0	2.56	9.30	67.26	10	110	no data available
	2.3	2.79	14.40	67.13	10	112	no data available
(N)	The laser alloying of the TiC niobium carbide powders in the surface of hot-work alloy tool steels						
X40CrMoV 5-1	1.2	1.42	2.46	55.34	10	49	24
	1.6	1.66	5.34	56.53	8	47	24
	2.0	2.21	6.14	57.55	8	46	20
	2.3	2.56	8.40	62.09	9	52	16
32CrMoV1 2-28	1.2	0.85	7.80	53.20	1	100	18
	1.6	1.39	11.10	51.50	1	96	15
	2.0	1.78	12.70	44.63	2	76	1
	2.3	2.13	12.90	41.10	2	82	8
(O)	The laser alloying of the VC niobium carbide powders in the surface of hot-work alloy tool steels						
X40CrMoV 5-1	1.2	1.32	9.20	55.68	8	53	24
	1.6	1.62	9.60	61.78	7	45	24
	2.0	2.18	9.80	62.57	7	58	23
	2.3	2.40	10.82	62.56	8	50	22
32CrMoV1 2-28	1.2	1.30	9.60	57.16	5	77	16
	1.6	1.55	10.60	56.66	5	73	14
	2.0	1.93	11.60	57.30	5	55	7
	2.3	2.27	19.20	58.36	6	53	4
(P)	The laser alloying of the WC niobium carbide powders in the surface of hot-work alloy tool steels						
X40CrMoV 5-1	1.2	1.46	18.62	55.62	5	76	61
	1.6	1.79	20.38	57.53	1	94	50
	2.0	1.98	23.82	57.93	1	94	46
	2.3	2.12	36.74	58.66	5	67	47
32CrMoV1 2-28	1.2	0.81	26.80	53.20	5	104	no data available
	1.6	1.27	30.40	51.50	5	107	no data available
	2.0	1.39	33.70	44.63	6	115	no data available
	2.3	1.91	33.80	41.10	6	117	no data available

*) 100% mass loss of sample has been assumed for the 32CrMoV12-28 steel laser treated with TiC within the laser power range of 1.2 kW.

The remelted zone (RZ) and the heat-affected zone (HAZ) can be distinguished between in the surface layer as a result of the remelting of the tested steels with their thickness depending on the laser power used for remelting. If steel remelting is carried out without inserting alloying additives such as carbide powders into the liquid metal pool, then a small increase in the properties of the surface layers of the steels examined is seen as compared to their respective properties obtained in conventional heat treatment, depending on the power of the laser beam used for remelting [60]. Likewise, the surface roughness of the steels alloyed with carbide powders is growing as the power of the laser beam increases within the entire range. This is caused by the presence of strong convective currents in liquid steel induced by the high power of the laser beam and fast crystallisation connected with the impact of a stream of shielding gas [61]. If the low laser beam power rating is applied, the remelting structure is relatively homogenous with its bottom being flat. The waving of the remelting bottom increases along with high higher laser beam power. The hardness of the surface layer of the steel examined achieved as a result of remelting is growing insignificantly as compared to the hardness of steel achieved after conventional heat treatment [62]. Along with an insignificant improvement of the surface layer hardness of the steels examined as a result of remelting, wear resistance is improving insignificantly as compared to the surface layers formed after alloying with carbide powders. Thermal fatigue strength for the steels subjected to remelting only is slightly higher than the strength achieved after standard heat treatment [63]. During laser alloying with powders containing WC, NbC, VC, TiC or TaC, they can be partially dissolved in a liquid metal pool or the carbides remain unsolved thus forming conglomerates as the unsolved grains of carbide powder are melted into the melted metal substrate. Abrasive wear resistance grows compared to steel resistance following standard heat treatment. An improvement in tribological properties is related to an improvement in steel hardness which, in turn, is caused by the structure being refined [64-66]. Thermal fatigue strength in case of alloying with carbide powders is also growing versus the resistance of the surface layers of the steels treated conventionally [56, 67, 68]. The correctly selected conditions of alloying such as laser power and scanning rate permit to achieve the high quality of surface layers free of cracks and featuring a regular, flat shape of the remelting face. The carbon phases of the powders used for alloying are present in the surface layer structure of the steel subjected to laser alloying, including: the WC tungsten carbide, the VC vanadium carbide, the NbC niobium carbide, the TaC tantalum carbide and the TiC titanium carbide. The investigations performed have evidenced that the surface layers produced in the laser remelting and/or alloying using carbide powders of the X40CrMoV5-1 and the 32CrMoV12-28 steel using a high power diode laser (HPDL) are characterised by higher mechanical and functional properties as compared to steels undergoing conventional heat treatment.

6.3. Strategic development directions of surface laser treatment of hot-work steels

A strategic position of the relevant, specific technologies of the laser remelting and alloying of alloy hot-work tool steels with carbide powders was identified by means of the matrix of strategies for technologies. A dendrological matrix of technology values was developed to determine the value of the relevant, specific technologies analysed. The matrix considers technology potential and attractiveness. The evaluation of positive and negative environment influence on the relevant, specific technologies was visualised with a meteorological matrix of environment influence [13]. The results obtained were entered into the matrix of strategies for technologies (Fig. 30) using software. As regards the N technology (8.79, 7.86) of laser treatment of hot-work tool alloy steels using the TiC titanium carbide powder and the O technology (8.61, 7.80) corresponding to the use of vanadium carbide VC powder, it is recommended to apply the oak in spring strategy. The strategy consists in developing, strengthening and implementing an attractive technology with a high potential in the industrial practise in order to achieve a spectacular success. The oak in summer strategy should be employed for the technology L (8.13, 6.15) of laser treatment of hot-work alloy tool steels using the NbC niobium carbide powder and for the technology M (8.22, 6.44) corresponding to the use of the TaC tantalum carbide powder. The strategy provides for that the attractiveness and potential of the technology shall be used in a risky environment and the difficulties that may arise will be avoided while matching a product to the customer demands preceded with thorough marketing research. The oak in autumn strategy needs to be implemented for the laser treatment of steel hot-work alloy tool steels using the WC tungsten carbide powder marked with the symbol P (8.40, 3.63). Such way of acting is connected with achieving successes with an attractive, stable technology at the predictable market and is combined with seeking new markets, customer groups and the products than can be produced with the technology. As far as the (K) laser remelting of hot-work alloy tool steels is concerned, it is recommended to use the dwarf mountain pine in summer strategy that recommends that a technology with a high potential should be made more attractive and modernised and to conduct marketing research and customise a final product to the customer's demands.

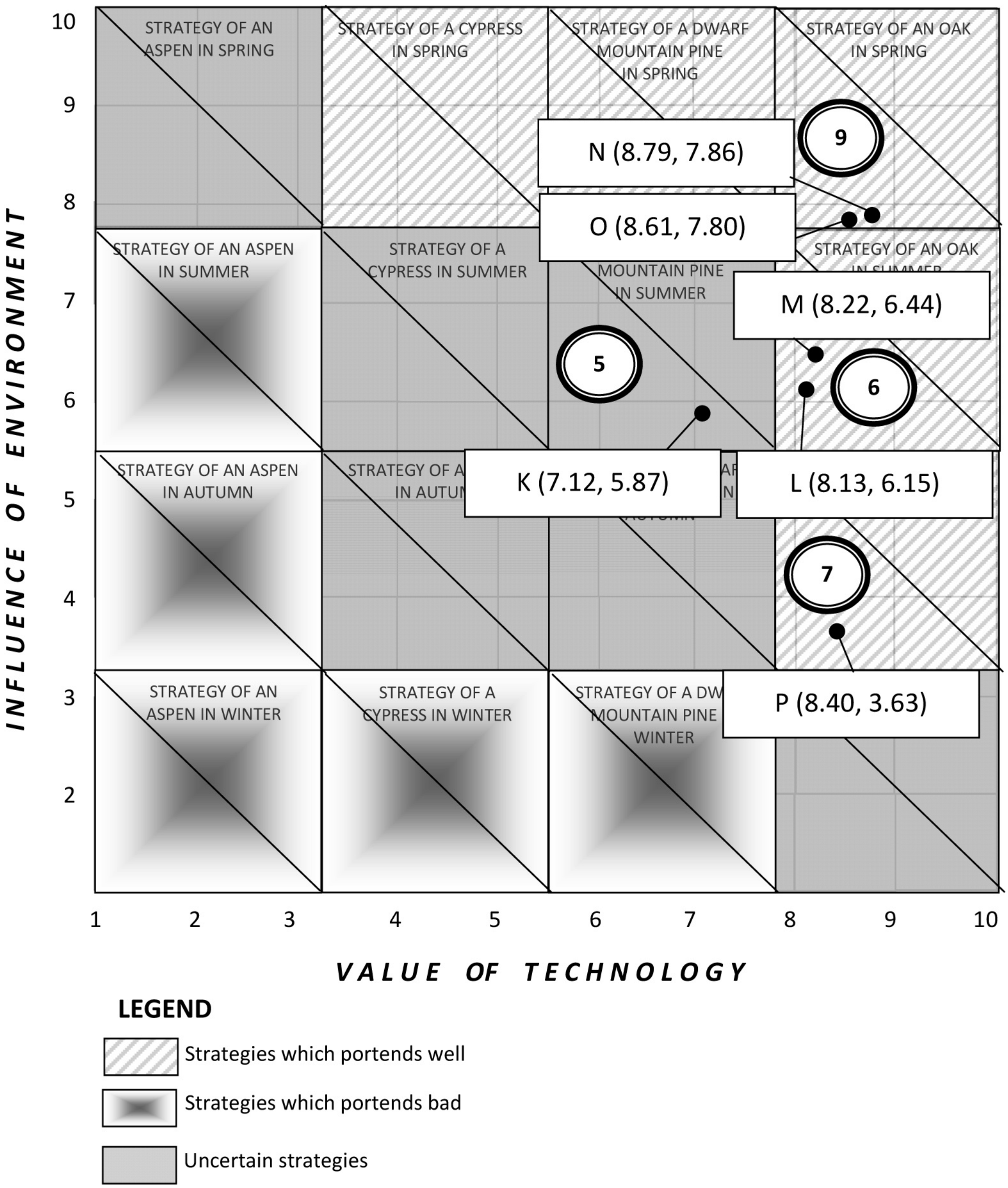
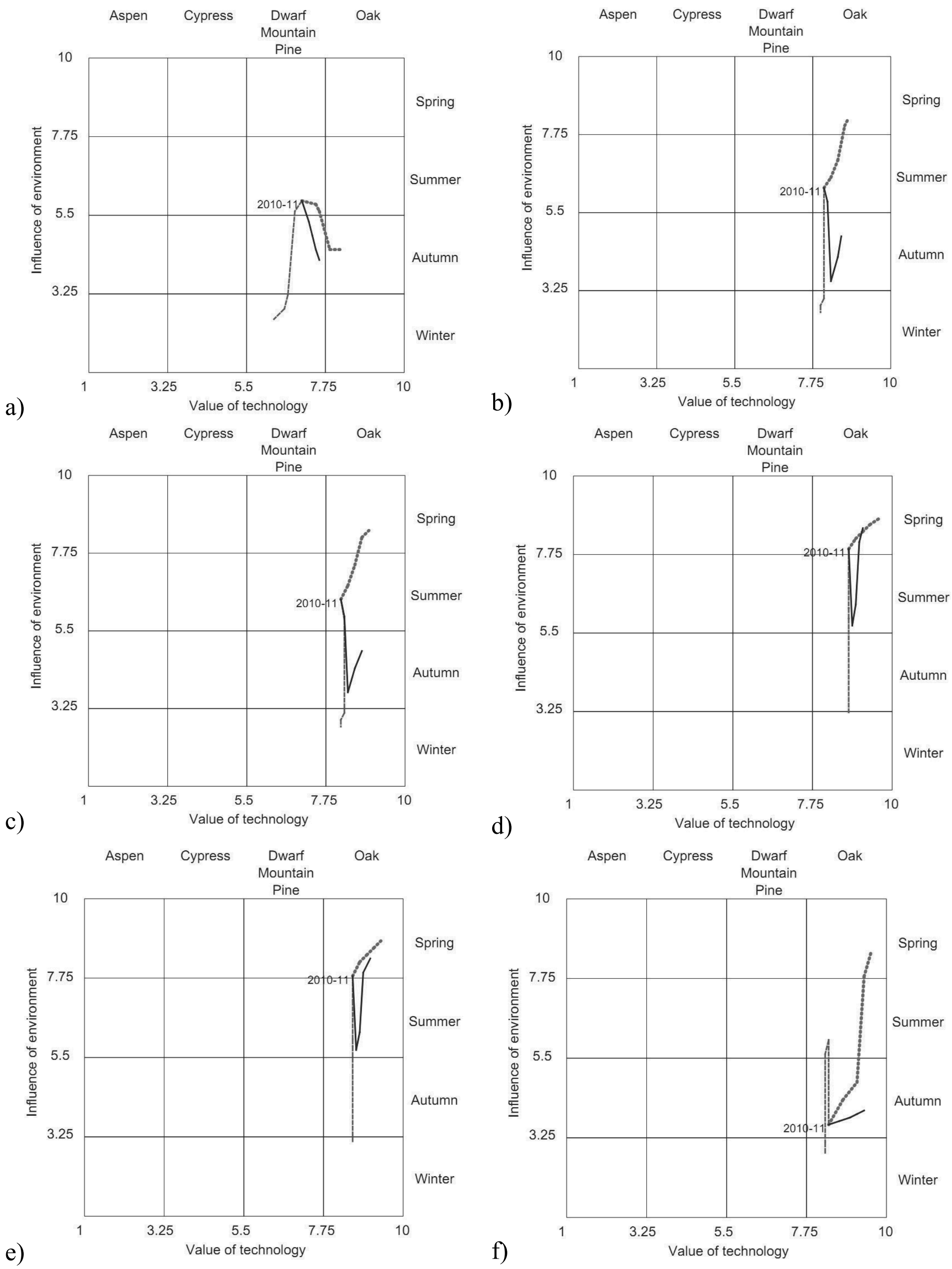


Figure 30. *The matrix of strategies for technology prepared for (K) laser remelting and alloying of hot-work alloy tool steels using NbC (L), TaC (M), TiC (N), VC (O) and WC (P) powders*



Types of strategic development tracks:

Optimistic   Neutral   Pessimistic

Figure 31. *Strategic development tracks prepared for (a) laser remelting and alloying of hot-work alloy tool steels using as follows: (b) NbC, (c) TaC, (d) TiC, (e) VC and (f) WC powders*

Strategic development tracks for the individual specific technologies representing a forecast of their development for the years of: 2015, 2020, 2025 and 2030 according to the three variants: optimistic, pessimistic and the most probable one, were next entered into the matrix of strategies for technologies. Simplified charts presenting the results of all the investigations carried out for the six analysed groups of technologies corresponding to the different types of powders deposited or to remelting without using powders are given in Figs. 31a-f. technology roadmaps and technology information sheets being a useful tool of comparative analysis according to the materials science, technological or economic criterion adopted were prepared at the last stage of the works using reference data in form of the results of materials science and foresight research and this is discussed in detail in the chapter 6.

7. Summary

The scenario method was employed in order to identify the forecast development directions of materials surface engineering. As there is no one correct and generally accepted method of creating the scenarios of future events or a management algorithm recommended for implementation in the scenario creation process, it became necessary to elaborate a custom concept that would aptly merge a presentation and description of the phenomena featuring a different extent of generality and seize the cause and effect relationships existing between them. In order to solve the so formulated research task, all the phenomena analysed were split into the three groups: a macrogroup with the single critical phenomena of the general nature characterised by strong interaction with the other phenomena; a mesogroup with a limited number of phenomena interacting moderately with the other phenomena and a microgroup comprising numerous specific phenomena highly sensitive to the interaction of other phenomena. Deductive reasoning by way of a synthesis was undertaken under the research consisting in seeking the combinations of micro- and mesofactors that would contribute, with a specific probability, to the occurrence of each of the three possible macroscenarios in the future. A simple method of presenting the research results is used in the chapter. The investigated phenomena are, therefore, presented starting with the highest level of generality (macro), through an indirect level (meso) ending with the most detailed aspects (micro). A cross analysis of how the key mesofactors of materials surface engineering development and the individual thematic areas are impacting the emergence of each of the three alternative macroscenarios, i.e. optimistic, neutral and pessimistic scenario, was undertaken using artificial neural networks. Due to the scale of the phenomena described, a representative group of laser technologies in surface engineering was chosen to demonstrate the method of presenting the development directions of the phenomena for the mesolevel, and laser remelting and alloying with special stress focus on hot-work alloy tool steels was chosen for the microlevel. A development forecast of laser technologies in materials surface engineering was established based on the results of the e-Delphix method differing from the classical Delphi method in that experts are surveyed electronically and in that the level of generality for the questions asked to the experts is growing along with the subsequent iterations of the research. The results of the foresight research are provided graphically in a bar chart. In order to identify the strategic position of the relevant groups of the critical laser surface treatment technologies against the thematic area of "Laser technologies in surface engineering", each of the groups was placed into a matrix of strategies for technologies. In addition, a chart was prepared presenting, in percents, the values of the predicted growth, stabilisation and decrease in the importance of the individual critical technologies as projected by the experts. The results of the materials science-foresight research concerning laser remelting and cladding of hot-work alloy tool steels with special emphasis laid on the results of materials science investigations and the strategic position of the specific analysed technologies was presented in the matrix of strategies for technologies. The forecast strategic development tracks of the relevant, specific technologies were next entered into the matrix. Technology roadmaps and information sheets were also prepared for the groups of the critical technologies and specific technologies.

The approach discussed allows to present the development directions of materials surface engineering according to the level of generality and according to the influence intensity of the phenomena analysed on other phenomena. A hybrid was created to fulfil this task. The hybrid embraces a collection of analytical methods and tools including: the scenario method, artificial neural networks, e-Dephix method, statistical lists as bar charts, foresight matrices together with technology development tracks, technology roadmaps and technology information sheets. Moreover, the results of the classical materials science methods are taken into account at the microlevel.

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14 E-transfer of materials surface engineering e-foresight results

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Abstract

Purpose: *The purpose of the chapter is to present an innovative concept of e-transfer of technology e-foresight results concerning materials surface technology, allowing for the practical industrial implementation of the results of materials science-heuristic research using a state-of-art IT technology.*

Design/methodology/approach: *Technology e-transfer is an innovating concept popularised in industry, especially in SMEs, developed in the course of the previously pursued e-foresight research of modern knowledge concerning the priority innovative materials surface technologies and the forecast directions of their development.*

Findings: *Technology e-transfer embracing e-information, e-advisory and e-learning, is an efficient method of disseminating the results of technology e-foresight of materials surface engineering within industry.*

Research limitations/implications: *If technology e-transfer is used in the Open Access mode, anyone, for free and at the same terms, has access to a database on the priority innovative technologies of materials surface engineering and the anticipated directions of their development, which has a positive effect on the development of a knowledge- and innovation-based economy.*

Practical implications: *The idea of disseminating the results of e-foresight of materials surface engineering by way of technology e-transfer is especially attractive for small- and medium-sized companies lacking the funds required to perform their own research in this scope.*

Originality/value: *The concept of technology e-transfer represents the Authors' original contribution into the development of Computer Aided Knowledge Management.*

Keywords: *Technology e-transfer; Technology e-foresight; Surface engineering; E-Delphix method; The Critical Technologies Book*

1. Introduction

Foresight research, conducted intensively abroad [1-3] and in Poland [4-7] in the recent decade and still continued, is aimed at seeking the innovative areas deserving financial support. The research is pursued with the participation of distinguished, internationally recognised experts. The research is an important source of diagnosing the key scientific, technological, economic and environmental issues and is an instrument of prediction and decision-making for the domestic authorities responsible for science, for business environments and for public administration. Foresight research, when referred to material engineering, enables, in particular, to identify the priority, innovative technologies of materials surface engineering and to define their strategic development directions. The dissemination of such research and the related public debate and the broadening awareness of entrepreneurs within the scope considered is tangibly translating into statistic growth in the quality of the technologies implemented industrially, into sustainable development and into the strengthening of a knowledge- and innovation-based economy. It is a common expectation these days that materials be manufactured possessing the properties ordered by product users [1, 2]. This expectation is significantly influencing the development of the products material design methodology as it is expected that materials are delivered having the required structure and physiochemical properties, meeting functional requirements, matching customer demands and usable functions of a product, i.e. so-called *materials on demand*. The functional properties of products can often be enhanced through the formation of the structure and properties of engineering materials surface layers [8, 9]. A full overview of the contemporary treatment technologies decisive for the formation of the structure and properties of engineering materials surface layers, including those exhibiting a nanometric structure, together with a general view on the current state of the art on the basis of analyses into the basic literature data and prior own research [10-20] is presented in an own book publication [9]. The publication [21] presents, on the other hand, the expert evaluation of the relevant material surface treatment technologies. The selected critical technologies of materials surface engineering, understood as priority technologies with the best development prospects and/or of key significance in industry over the assumed time horizon, were subjected to own research [10, 21, 22] in order to evaluate their value, according to objectivised criteria, against the micro- and macroenvironment and to identify their development prospects over the nearest 20 years. The development directions of the most advantageous technological solutions of surface layers structure and properties formation of products and their elements produced using engineering materials considered as critical were indicated as part of own materials science-heuristic and foresight research [10, 21, 22, 23], pursued together with top-notch, internationally recognised experts. The culmination of all such efforts is an own methodological monograph [22] pertaining to the computer integrated development prediction methodology in the area of materials surface engineering. Diagnosing the underlying scientific, technological, economic and environmental aspects of materials surface engineering and defining the directions of their strategic development and decision-making boils down to creating few alternative, feasible scenarios of their development. The aim of the scenarios is to improve the functional properties, durability and reliability of products and to select the most effective technologies that must be popularised in industry which – in terms of their modernity and a price to quality ratio – are most suitable for effective implementation in industry, grouped by the most avant-garde thematic areas. One of the final outcomes of the technology foresight of materials surface engineering is the Critical Technologies Book with technology roadmaps and technology information sheets. The Book is characterising, in a harmonised fashion, the critical materials surface engineering technologies, offering a convenient tool of comparative analysis, especially for SMEs lacking the funds sufficient to pursue their own research in this field. An important aspect here it to popularise this state-of-the-art knowledge in the broadest possible group of managers and engineers working in industry, especially in small- and medium-sized companies. The issue of technology transfer and knowledge transfer becomes pivotal, therefore, as the gathered results of detailed research may bring the desired economic effects only if put into life in industry and economy.

The purpose of this chapter is to describe the concept of e-foresight as a proven method constituting the key element of the computer integrated development prediction methodology in materials surface engineering area and to describe the method, deriving from this concept, of technology e-transfer, enabling the practical industrial implementation of the materials science-heuristic research performed.

2. Technology e-foresight of materials surface engineering

The development directions of the most advantageous technological solutions of surface layers structure and properties formation of products and their elements produced using engineering materials and biomaterials, considered as the critical technologies of materials surface engineering, were formulated as part of own efforts together with indicating their current strategic position and with identifying their development prospects [10, 22]. The critical materials surface engineering technologies are the priority technologies in this field having the best development prospects and/or being of key significance in industry over the assumed time horizon. They are grouped into two thematic areas, i.e. Manufacturing (M) and Product (P), with each of them grouped into seven specific thematic groups. The area of Manufacturing (M) reflects a manufacturer's point of view and encompasses technological processes determined by the state of the art and by the manufacturing capacity of the machine park. The following thematic groups are distinguished between for the M area: laser technologies in surface engineering, PVD technologies, CVD technologies, thermochemical technologies, polymer surface layers technologies, nanostructural surface layers technologies, other surface engineering technologies. The area of Product (P) is conditioned by the expected functional and usable properties stemming from customer needs and is focussed on the product and on the material it is made of. The following specific thematic groups are distinguished between for the (P) area, i.e. surface engineering of, respectively: biomaterials, structural metallic materials, structural non-metallic materials, tool materials, steels used in automotive industry, glass, micro- and optoelectronic and photovoltaic elements, polymers. The results of own pursuits [10, 22] are useful for a wide group of beneficiaries – the representatives of science, economy, public administration and students.

The adopted e-foresight research methodology and tools [24-27] (Fig. 1) are innovative, novel and experimental and consist in using the Internet and information technologies, including a virtual organisation, web platform and neural networks used in an innovative manner, for analysing the cross impacts existing between the analysed trends and the events likely to occur in the future within the considered timeframe [20]. Other research methods usually applied in such type of efforts were also applied in the research pursued [28-35].

The following methods of work, organisation and management were used at the different stages of the research [10]: review of literature, reference data analysis, defining the key technologies, environment scanning, technology mapping, beneficiary mapping, trends extrapolation,

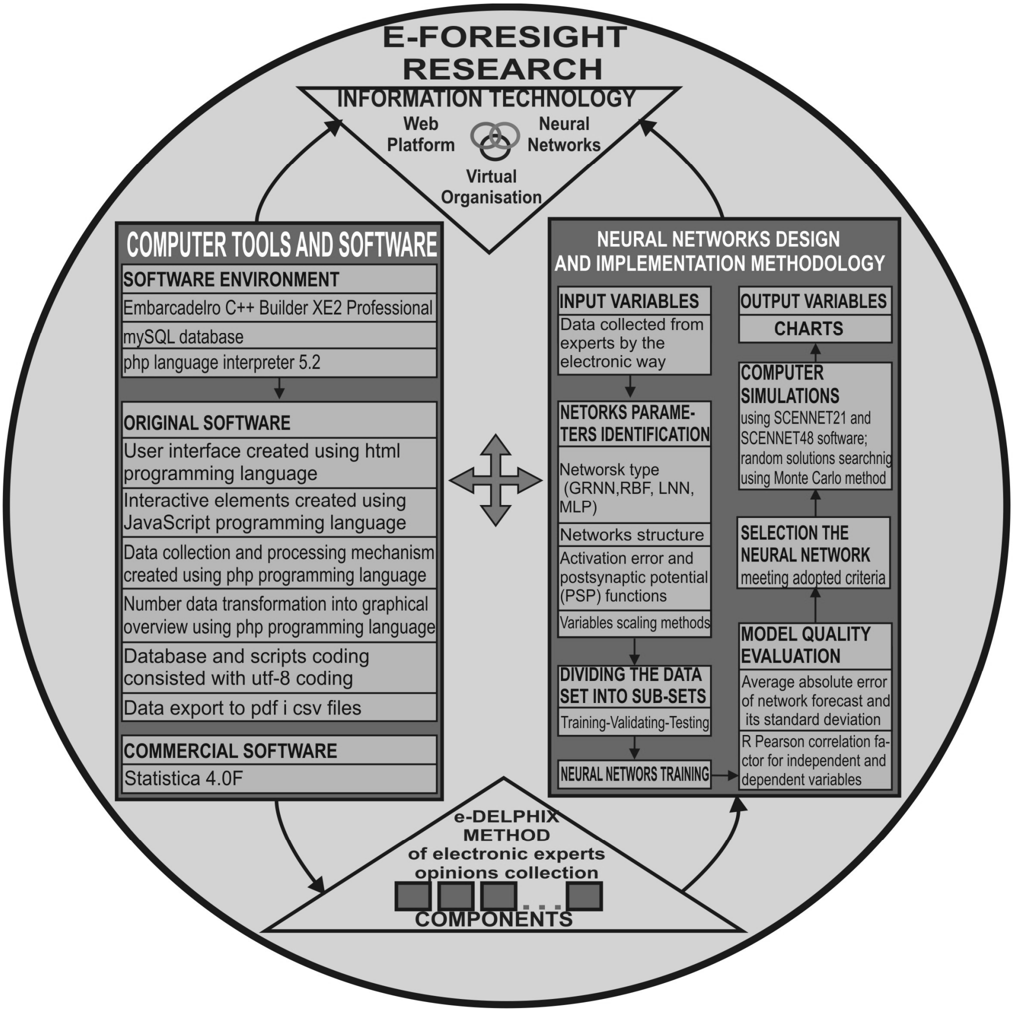


Figure 1. *The e-foresight research scheme [22]*

SWOT analysis, expert panels, brainstorming, workshops, sketches, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric analyses and statistical methods. The scheme of correlations between used research methods and indirect results of the e-foresight process is presented in Fig. 2. An analysis of development prospects was made in the initial phase of research for approx. 500 groups of detailed technologies including an issue state assessment, technological review and a strategic analysis using integrated methods [36-38]. 10 critical technologies were selected in 14 thematic areas as a result of the works performed, representing the priority technologies of materials surface engineering with the best development prospects and/or a crucial role for industry over the nearest 20 years. A set of 140 critical technologies was thoroughly analysed for three iterations of the e-Delphix method [38] according to the e-foresight methodology [24-27] the scope of which is depicted in a chart in Fig. 3.

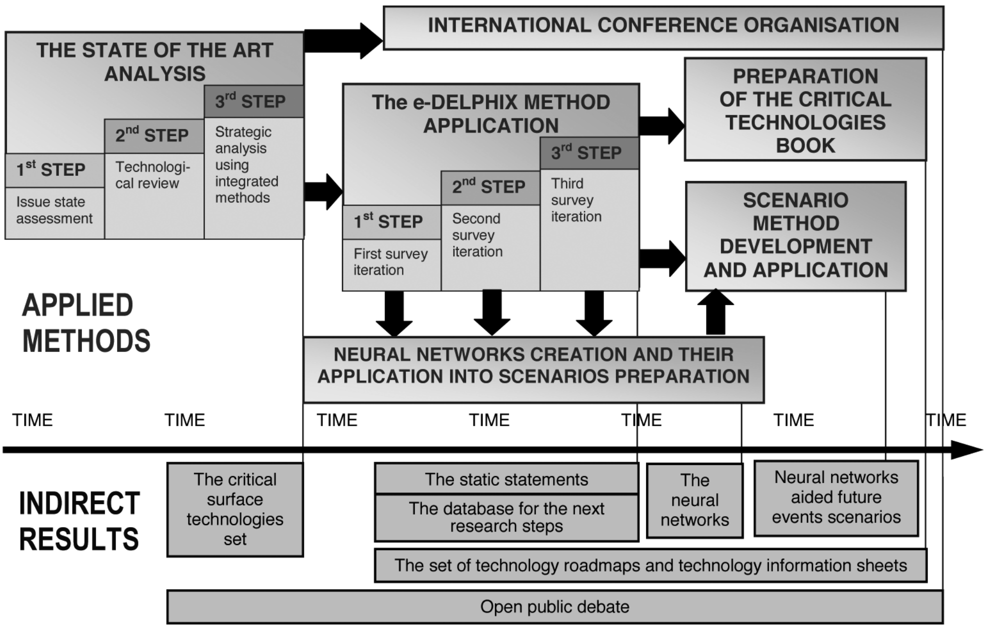


Figure 2. *The scheme of correlations between used research methods and indirect results of e-foresight process[9, 38]*

The e-Delphix method is a modified version of the classical Delphi method [28-34], differing from the original method mainly in that experts are surveyed electronically and in that the level of generality for the questions asked to the experts is growing along with the subsequent iterations of the research. Distinguished, internationally recognised experts participated in the expert studies relating to the future of materials surface engineering. They completed around 650 multi-question, complex surveys at different stages of the works. Alternative macro- and mezo-scenarios of future events for materials surface engineering were established using neural networks and the opinions of the experts surveyed were used as reference data. The scenarios can be and should be used for steering appropriately a process of channelling the development of the areas studied and also to accommodate R&D institutions and enterprises effectively to the swiftly changing demands of the global environment.

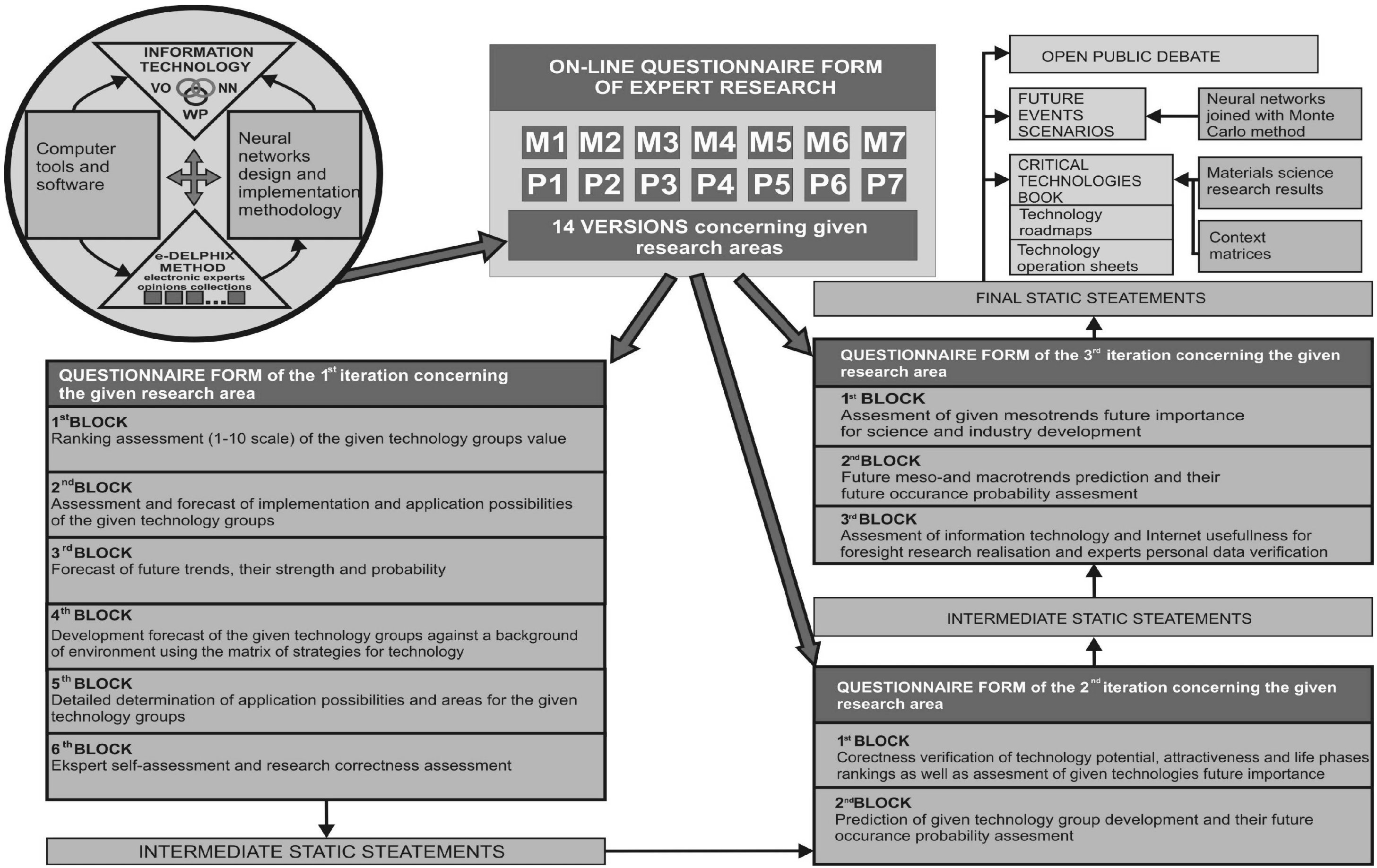


Figure 3. *The e-Delphix method scheme presentation; where: (VO) Virtual Organisation, (NN) Neural Networks, (WP) Web Platform [22]*

The relevant specific technologies were assessed according to the opinions of the key experts using the e-foresight research methodology provided in the references [24-27]. A universal scale of relative states being a single-pole scale without zero was used in the research undertaken, where 1 is a minimum rate and 10 an extraordinarily high rate. Homogenous groups were differentiated between for the technologies analysed in the first place. The groups were evaluated for their potential representing a realistic objective value of the specific group of technologies and for their attractiveness reflecting the subjective perception of a specific technology by its potential users. The objectivised values of the potential and attractiveness for the relevant, selected technology groups are visualised by means of the dendrological matrix of technology value [11, 25] consisting of quarters into which the results of the evaluation made are entered. According to the assumptions adopted, the most promising quarter guaranteeing a future success is a wide-stretching oak, with a soaring cypress and a rooted dwarf mountain pine also likely to ensure a success, provided an adequate procedure is applied, which is unlikely or impossible for a quaking aspen. The metrological matrix of environment influence [11, 25] presents graphically the results of evaluation for the influence of external positive factors (opportunities) and negative factors (difficulties) on the technologies analysed. Each of the technologies groups assessed by the experts was entered into one of the matrix quarters. Sunny spring illustrates the most favourable external situation ensuring the future success. A technology's success is also possible in rainy autumn providing a chance for steady progress in a neutral environment and it is risky but feasible in the quarters corresponding to hot summer in a stormy environment. Frosty winter informs that technology development is difficult or impossible.

A matrix of strategies for technologies [11, 25], presenting graphically a position of each technology according to its value and environment influence intensity and identifying a recommended action strategy, is prepared according to the results of expert studies provided in prior in the dendrological and meteorological matrix. The strategic development prospects of a given group of technologies are expressed in encircled numbers. An example of a matrix of strategies for technologies made for the most promising groups of PVD technology is shown in Fig. 4. Strategic development tracks, reflecting the predicted situation of a given technology if positive, neutral or negative external factors occur, are entered into the matrix of strategies for technologies investigated or their groups based on the outcomes of materials science-heuristic research. The examples of strategic development tracks prepared for the texturisation of polycrystalline silicon and for classical, selected methods of thermochemical treatment are shown in

Fig. 5. The forecast established describes a vision of future events consisting of several variants and concerns the time intervals of 2015, 2020, 2025 and 2030.

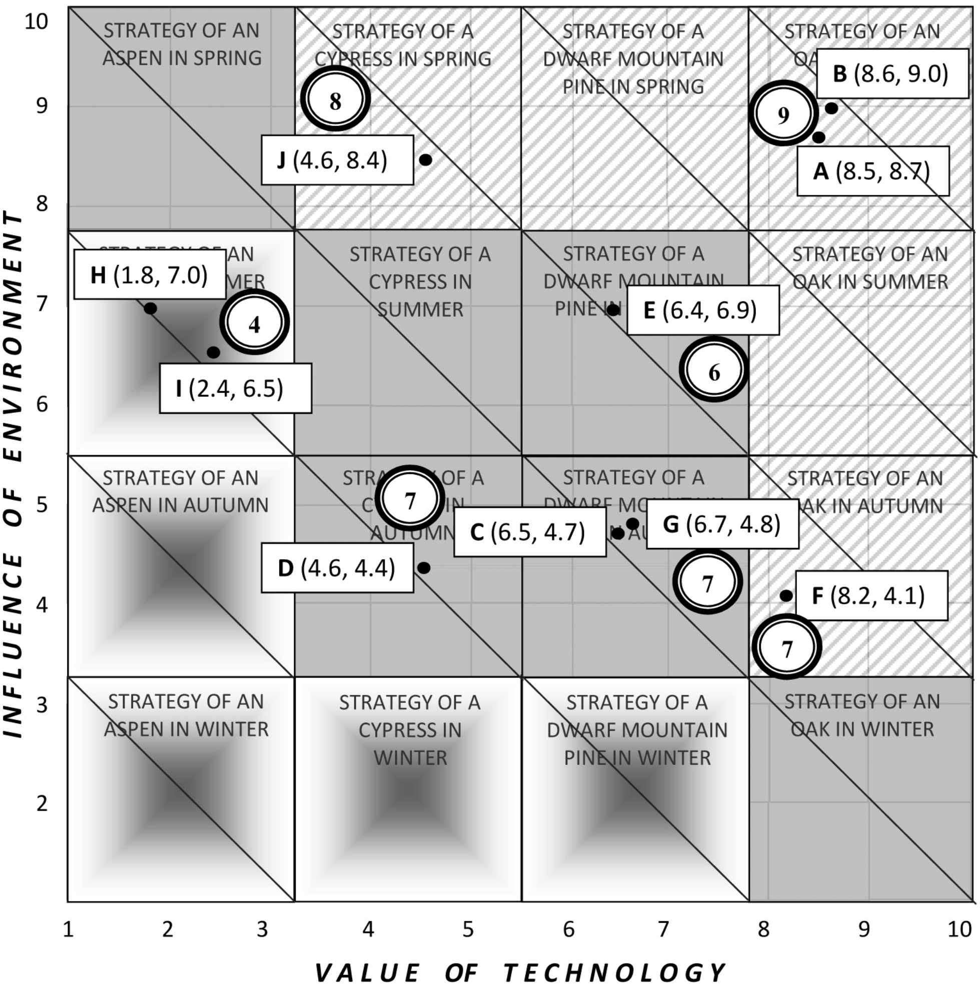
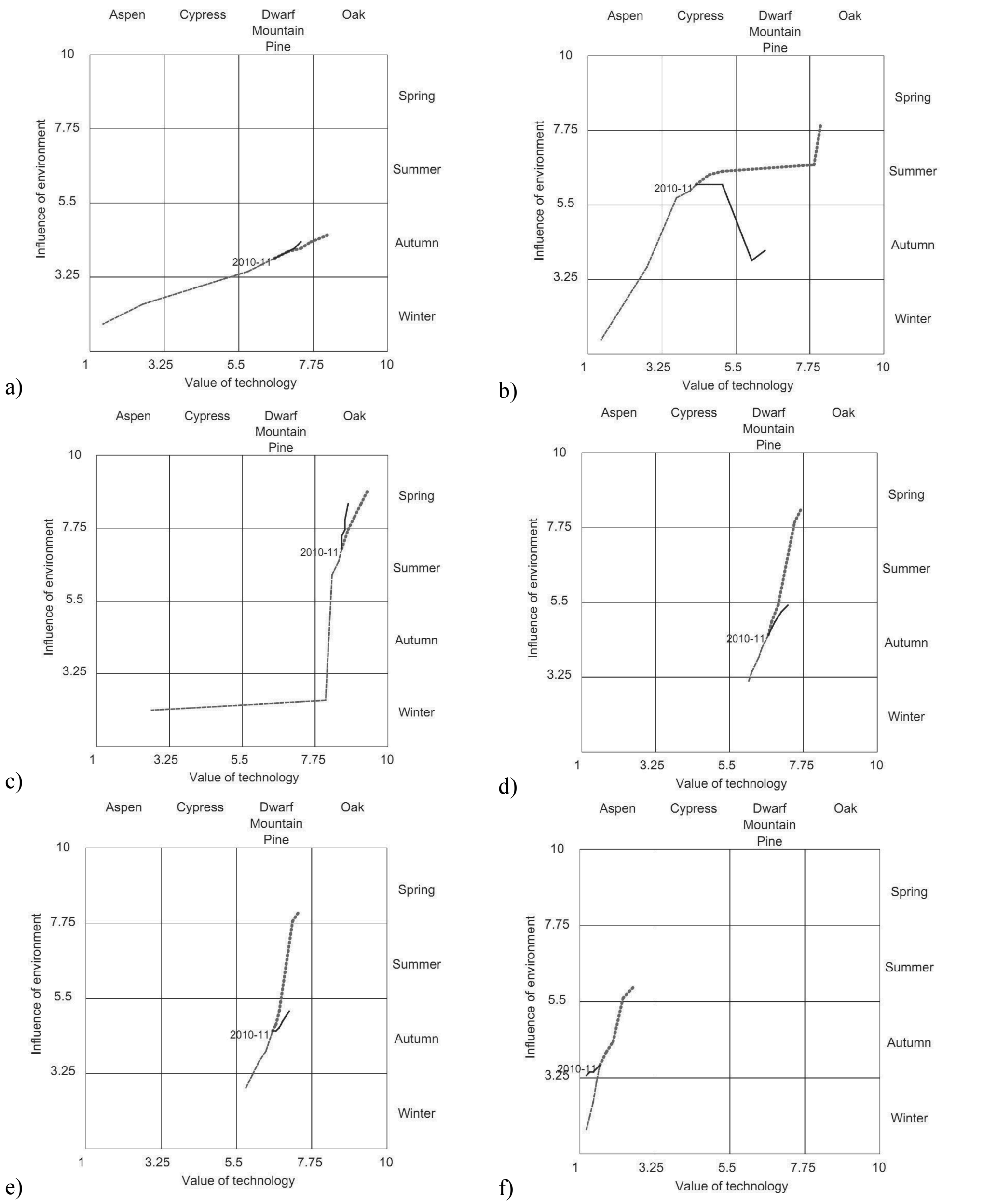


Figure 4. *The matrix of strategies for technologies prepared for "PVD technologies" thematic area, where: (A) CAD, (B) RMS, (C) PPM, (D) IBAD, (E) HHCD, (F) EB-PVD, (G) BARE, (H) ICB, (I) TAE, (J) PLD [9, 39]*

A lot of the evaluations and conclusions under the project [10] were formulated on the basis of materials science-heuristic research, as a customs approach in many works followed in connection with this foresight [12-20, 39-44]. The relevance and adequacy of the assessments performed according to the methodology developed [11, 22, 25] is ensured by the synergic



Types of strategic development tracks:
• • • • • • Optimistic — Neutral – – Pessimistic

Figure 5. *Strategic development tracks prepared for polycrystalline silicon: (a) alkaline texturisation, (b) laser texturisation, (c) laser texturisation with chemical etching as well as for thermochemical treatment, i.e. (d) nitriding and its variants, (e) carburising and carbonitriding, (f) diffusion boriding; Co-authors: A.D. Dobrzańska-Danikiewicz, A. Drygała, E. Hajduczek*

influence of the materials science research and foresight methods of an interdisciplinary character conducted with the research methodology concerning primarily technology foresight [33, 34] being part of the field of knowledge known as organisation and management and of surface engineering forming part of the widely understood material engineering. Methods deriving from artificial intelligence, statistics, information technology, machine construction and operation and strategic and operational management were also used at some stages of the research. The overall methodology of the integrated computer-aided prediction of development trends in materials surface engineering embraces also the methodology of interdisciplinary materials science-foresight-IT research including a group of originally matched, but commonly known analytical methods and tools as well as an original methodological concept, allowing to pursue further research, that encompasses context matrices, the Critical Technologies Book of materials surface engineering and the neural networks-aided creation of alternative scenarios of future events. The underlying methodological assumptions of the research are shown in Fig. 6.

3. The Critical Technologies Book and its industrial usefulness

Of the approx. 500 technologies / groups of technologies of materials surface engineering analysed initially, 140 technologies (10 for each of 14 thematic areas) were classified for detailed expert studies with the e-Delphix method. The results of the studies, supplemented by the results of own materials science research, have represented reference data underlying the preparation of the Critical Technologies Book. The Book is comprised of a pool of roadmaps and information sheets being a compendium of concise knowledge on the priority materials surface engineering technologies with their best development prospects and/or of vital significance in industry over the nearest 20 years (Fig. 7).

The technology roadmaps, developed according to a customs concept, presented in particular in [12-20, 25, 40-44] – considering their coherent, harmonised formula – are a very convenient tool of comparative analysis, and allow to choose the best technology in terms of the materials science, technological or economic criterion selected. The set-up of the roadmaps corresponds to the first quarter of the Cartesian system of coordinates. Three time intervals, respectively, 2010-11, 2020 and 2030, are provided on the axis of abscissa. Seven main layers ordered by their

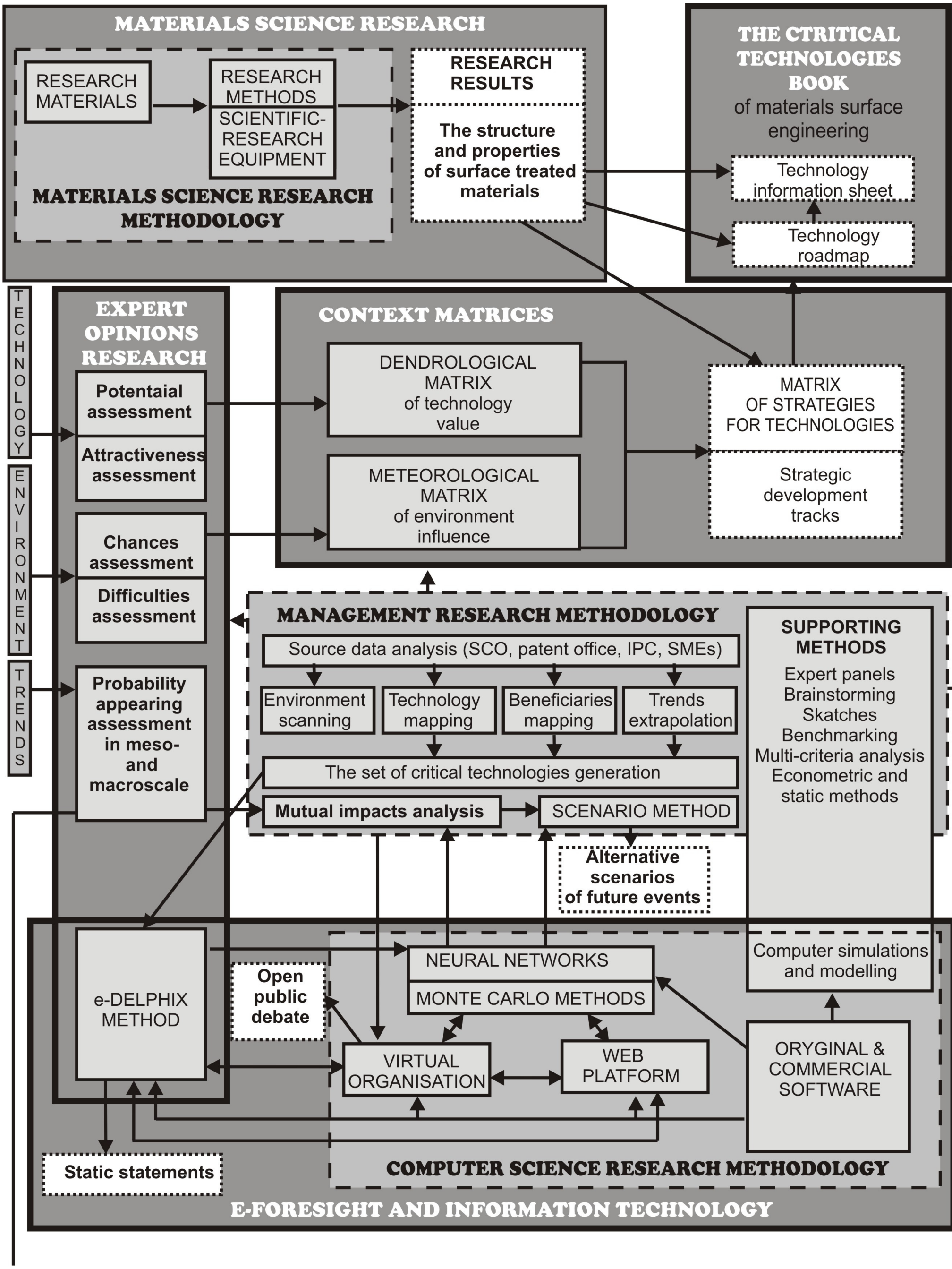


Figure 6. *The computer integrated development prediction methodology in materials surface engineering area [22]*

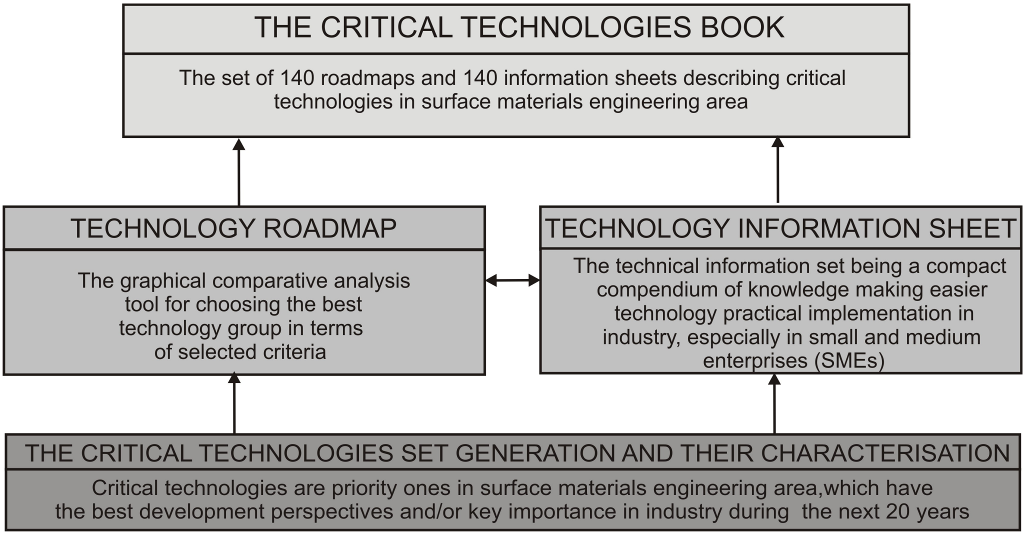


Figure 7. *The Critical Technologies Book as the set of technology roadmaps and technology information sheets*

growing hierarchy are provided on the axis of coordinates with the rising level of specificity characterising the technologies analysed, respectively for: sequence, relevance, subject, method, place, contractor and costs. Different arrows present different types of relationships between the relevant layers and sublayers of technology roadmaps. Flexibility is their undisputed advantage. If needed, they can be continuously accommodated to the particularity of the sector, size of an enterprise or an entrepreneur's expectations by adding the relevant content [11]. An example of a technology roadmap prepared for the PVD deposition of several (in the amount15) layers of coating onto the brass substrate is shown in Fig. 8.

Technology roadmaps are supplemented by technology information sheets, containing technical details helpful in implementing a specific technology in the industrial practice, especially in small- and medium-sized enterprises that must use the commonly available information as they are unable to carry out usually costly own research in this field. The technology information sheets provide, in particular, a description of a technological process, an overview of physiochemical phenomenon related to the technological processes, the advantages and disadvantages of the relevant technology, the most prospective detailed technologies and substitute / alternative technologies. The following details are also given in the other fields of the sheets: the types of a coating / surface layer that may be deposited or the processes occurring at the substrate surface; the specific properties of coatings / surface layers / substrate surfaces achieved as a result of technological processes; general physiochemical conditions of technological process implementation; substrate material preparation methods; types of research instruments; specific accessories. Each of the technology information sheets also contains information, expressed with a universal scale of relative states consisting of ten degrees, on: impact of technology implementation on the properties of the material and mechanisms of their wear, the industry with the broadest application prospects, computer modelling and steering methods and technology development prospects. A graphical chart and an index of the recommended references complete all the aspects presented in technology information sheets the example of which is made for ions implantation in the thematic area of non-metallic construction materials presented in Fig. 9.

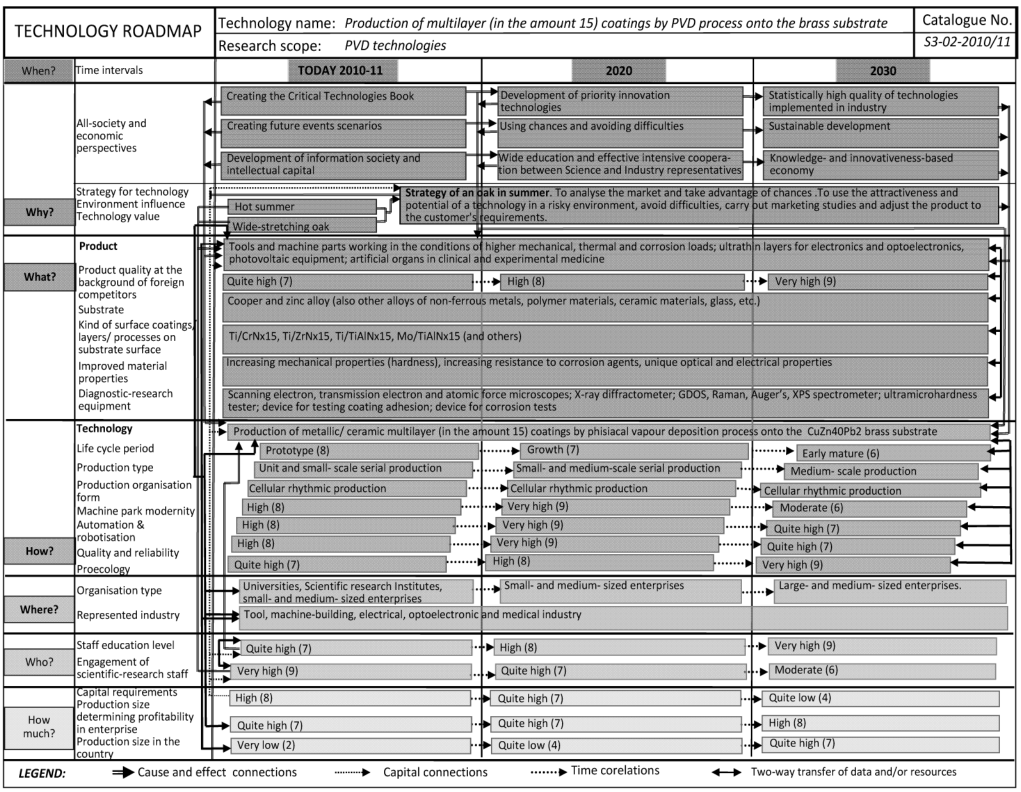


Figure 8. *An exemplary technology roadmap prepared for production of metallic/ceramic multilayer (number of layers 15) coatings by physical vapour deposition process onto the CuZn40Pb2 brass substrate; Co-authors: A.D. Dobrzańska-Danikiewicz, K. Lukaszkowicz*

a)

TECHNOLOGY INFORMATION SHEET	Technology	*Ion implantation*	Catalogue No.
	Thematic area	*Non-metallic structural materials surface engineering*	P3-05-2010/11

The essence of the physiochemical phenomenon
Ion implantation is a process of introducing ionised atoms of any element into a foreign body, with a high energy (of more than ten keVs to several dozens of MeVs) they gain in a vacuum in an electrical field accelerating and forming ions into a beam. Ion implantation is a treatment process aimed at modifying the properties of non-metallic construction materials, most of all ceramics and plastics. A 0.1÷1 µm thick layer is formed as a result of implantation joined integrally with the substrate with its properties being different, better than the substrate. Ions can be implanted into a solid in a continuous and pulse manner.

Type of possible coating / surface layer or of the process occurring on the substrate surface					
	monolayer		multi-phase		amorphous
	multilayer		graded		nanocrystalline
	multilayer (>100 layers)		composite		hybrid
	phase transformations of substrate surface	×	change of chemical composition on substrate surface	×	physical processes on substrate surface

Special properties of coatings / surface layers / substrate surface due to processes							
×	mechanical		magnetic		optic	×	tribological
×	chemical		diffusion		thermal	×	anticorrosive
	electrical		hydromechanical		acoustic		Others

Advantages	Disadvantages
Any element can be implanted and the concentrations of the implanted elements are achievable exceeding their solubility in the treated material.	A high apparatus cost; process directivity; items with complicated shapes cannot be treated.

The most prospective specific technologies and/or areas of applications
Ion implantation based on ceramic elements of mechanical devices and ceramic elements of electronic circuits (including microelectronic circuits) and based on polymers to improve tribological, electrical and magnetic properties.

Substitute / alternative technologies
Electron techniques.

Recommended references	
1	E. Knystautas (ed.), Engineering Thin Films and Nanostructures with Ion Beams, Taylors & Francis Group, Boca Rato – London - New York – Singapore, 2005.
2	Llc Books (ed.), Thin Film Deposition: Evaporation, Chemical Vapor Deposition, Sol-Gel, Focused Ion Beam, Thermal Spraying, Diamond-Like Carbon, Books LLC, Tennessee, 2010.
3	R. Hellborg, H.J. Whitlow, Y. Hang (eds.), Ion beams in nanoscience and technology, Springer Verlag, Berlin-Heidelberg, 2009.

Influence of technology application on the predicted and expected material properties	Level
Biocompatibility	Quite high (7)
Resistance to high temperature	Quite high (7)
Wear resistance	Moderate (6)
Hardness	Moderate (6)
Erosion resistance	Moderate (6)
Fatigue strength	Moderate (6)
Corrosion resistance	Moderate (6)
No brittleness	Moderate (6)
Efficiency of technology's counteracting the results of wear	**Level**
Abrasive wear	High (8)
Pitting	High (8)
Scuffing	Quite high (7)
Fretting	Quite high (7)
Erosion	Quite high (7)
Diffusive wear	Quite high (7)
Plastic wear	Quite high (7)
Industry sector with the highest applicability of technology acc. to the PKD classification	**Level**
Scientific research and development works	Average (5)
Manufacture of products with other mineral non-metallic raw materials	Low (3)
Production of computers, electronic and optic products	Very low (2)
Architecture and engineering activity; technical testing and analysis	Very low (2)
Manufacture of rubber and plastic products	Very low (2)
Manufacture of automotive vehicles, trailers and semi-trailers	Very low (2)
Manufacture of other transport equipment	Very low (2)
Other manufacture of products	Minimum (1)
Applicability of computer modelling and steering methods for the technology	**Level**
Large-scale modelling	High (8)
Artificial neural networks	Quite high (7)
Fuzzy logic	Quite high (7)
Genetic algorithms	Moderate (6)
Monte Carlo methods	Moderate (6)
Current life cycle of technology	Mature (5)
Development prospects	Quite high (7)

b)

TECHNOLOGY INFORMATION SHEET	Technology	*Ion implantation*	Catalogue No.
	Thematic area	*Non-metallic structural materials surface engineering*	P3-05-2010/11

Description of the manufacturing process progress

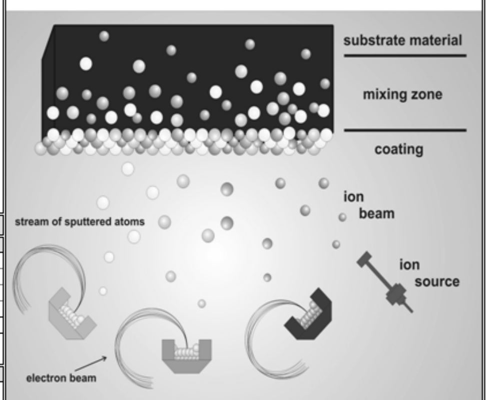
- The charge surface is initially prepared before implantation;
- A thin layer is deposited onto the surface of the implanted material (optionally);
- The charge is placed into the implantator working chamber;
- The implantator working chamber is pumped off;
- Ion implantation;
- Charge cooling;
- The charge is taken out from the implantator working chamber.

Ion implantation is a process of doping materials based on the use of a high kinetic energy. The dope atoms are ionised in the ion source and then accelerated in an electrical field. Any material can be doped with almost any element due to the kinematic character of the process. The concentrations of alloying additives exceeding their solubility in the material used can be achieved by means of the ion implantation technology (usually 20%, maximum to over 50%) and independence from the adhesion effect can be attained.

General physiochemical conditions of technological process performance

Standard range of process temperature	Unit		from		to	
Temperature	°C		20		600	
Pressure	Pa		10^{-5}		10^{-6}	
Current and voltage conditions	kV	mA	20	1	200	25
Time	min.		30		90	
Environment / atmosphere	vacuum					
Specific conditions of process performance	high vacuum					

Method(-s) of initial substrate material preparation
Initial cleaning: mechanical, chemical, physical, physiochemical cleaning; ultrasound cleaning.
Device type / kind
Ion implantators (ion accelerators)
Specific accessories
Ion source; focusing and accelerating system.



Example scheme of ion implantation process progress with the IBAD (Ion Beam Assisted Deposition) method

Figure 9. *Example of a technology information sheet prepared for ion implantation in the thematic area of non-metallic construction materials: a) Page 1, b) Page 2; Co-authors: A.D. Dobrzańska-Danikiewicz, K. Lukaszkowicz*

4. Technology e-transfer as a tool of e-foresight research results dissemination

There is a substantive justification for the outcomes of own works [10, 22] to be implemented and used practically in the economic reality. One of the general purposes of the research undertaken is to kindle a public debate with the participation of domestic and foreign representatives of the world of science, economy and public administration, in order to disseminate the project results in the environments interested in the area discussed. A public debate allows to further intensify liaison between the sphere of research and development and economy and to intensify the flow of human resources between such groups, which is also a utilitarian consequence of the actions taken under the own research conducted [10] resulting in the improved competitive position of the Polish economy and science against other European and global countries. The aspect of cooperation between Science and Industry was found by the experts attending the foresight research to be the most important factor determining the future development of materials surface engineering. 80% out of the 227 experts surveyed expressed such an opinion [10].

A web platform established under the project allows the general public, especially industry, scientific institutions and social organisations to acquire, anytime, detailed information on the project's objectives, assumptions and contractors, to track the results of the actions taken and to express their own opinion though social on-line consultation, ensuring the relevant feedback. By promoting the project results and by using widely electronic tools such as a website, databases on the technologies of biomedical and engineering materials surface properties formation and on the products they can be applied for, as well as by conferences, workshops and seminars, access can be ensured to the project results to a very wide group of users.

A concept of a **technology e-transfer** centre and the related tasks has been formulated in order to extend the objectives of e-foresight to include the domain of application and implementation of knowledge on selected engineering materials surface properties and structure formation technologies and, in general, material processes technologies and engineering materials processing, mainly in the machine and electrotechnical industry. The relationships between the e-foresight process, and technology e-transfer are shown in a chart in Fig. 10. The measures are aimed at the practical deployment of state-of-the-art technologies in this area

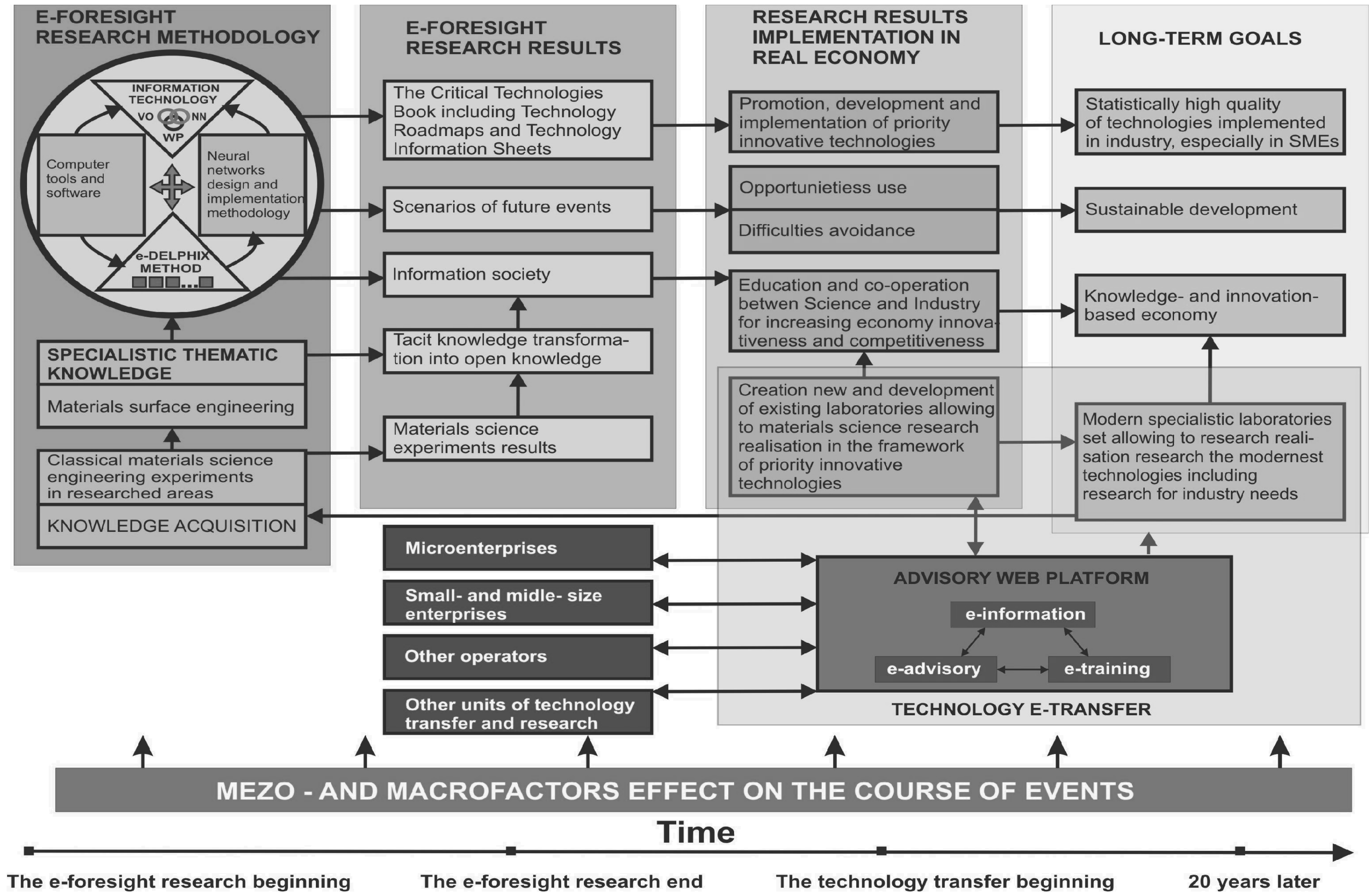


Figure 10. *E-foresight process versus technology e-transfer [22]*

by SMEs to strengthen their innovation and competitiveness. The new technology transfer centre is formed by a group of Thematic Workshops and Teams. The relevant Teams fulfil tasks of technology transfer, without providing services. By means of an the appropriately prepared web platform, they provide – in the Open access mode – information and knowledge on engineering materials and surface treatment and material processes technologies resulting from the e-foresight research performed and on the on-going monitoring of materials science issues without dedicating the offer to concrete users. Such tasks are considered to be technology e-transfer (electronic transfer of technology) to be conducted on a continuous basis without any limitations and for free using an openly available (Open Access) web platform. The role of the first Team is e-advisory, i.e. the stakeholders use freely and without limits, on the relevant pages of the platform, the roadmaps and information sheets concerning the priority innovative materials surface engineering technologies. The activity of another Team performing an e-training function according to the Open Access formula consists of publishing self-control cards at the web platform pages along with knowledge on quality control and on the technical selection of engineering materials and on the technology of material processes and surface treatment, and then to again verify the level of own knowledge after studying the recommended sources available from the web platform. The information team is focussed on conveying e-information, on an Open Access basis, on the development of the web platform's resources and the technology e-transfer initiatives taken and on the possibilities of adapting modern technologies by SMEs. A basis of the advisory, training and information functions, as the essence of technology e-transfer, is represented by specialised materials science research performed at the Thematic workshops that are conducting scientific research according to the development trends and directions formulated through e-foresight research and also problem monitoring. Specialist apparatuses are required for that. The tree of issues presented in Fig. 11 indicates the reasons determining the key problems and details out the predicted consequences of the events occurring at the domestic scale. Most probably one can presume, however, that the similar aspects are considered at the scale of Europe or even World. An innovative technology e-transfer concept discussed in this chapter, connected with e-advisory, e-training and e-information, supported with own scientific research in the areas resulting from the previously conducted e-foresight research and from the monitoring of current issues being a basis of technology e-transfer, will be continued to be developed and represents the Authors' vital contribution into the development of computer aided knowledge management science.

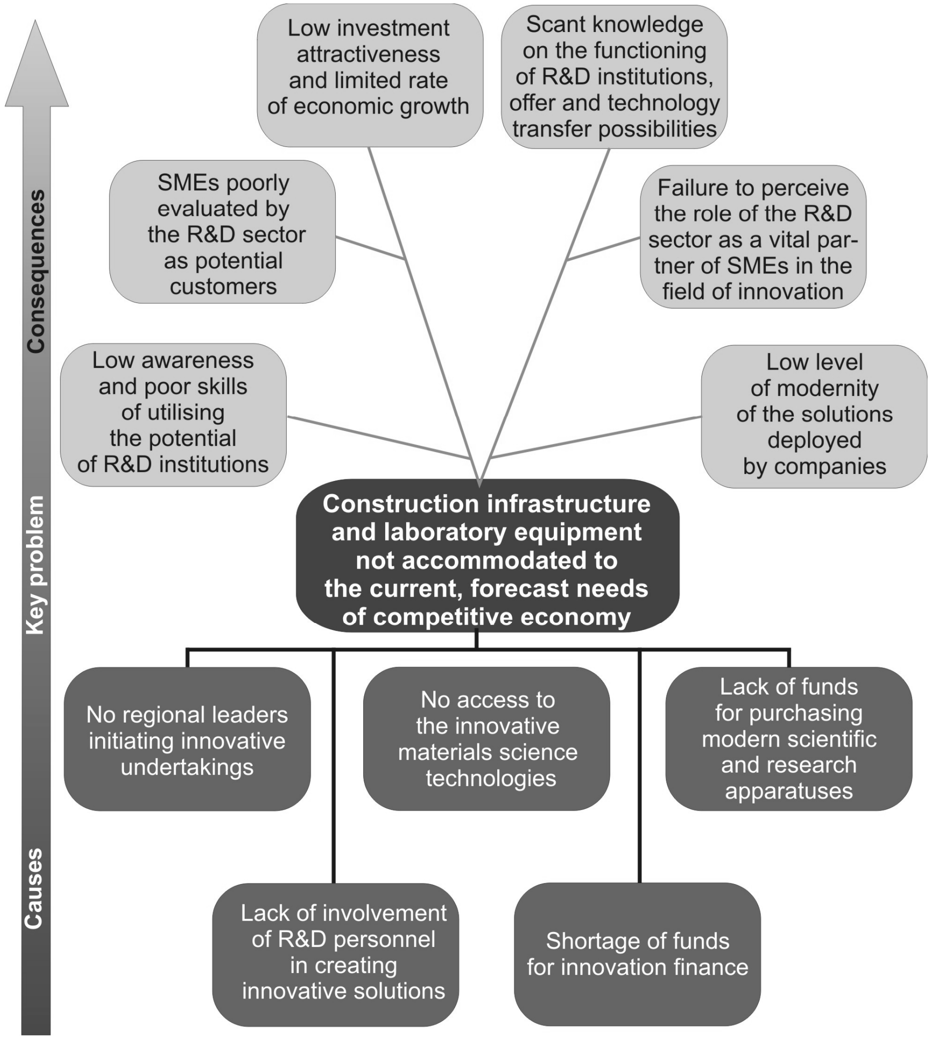


Figure 11. *Problem tree*

5. Conclusions

The purpose of this chapter is to describe the concept of e-foresight as a proven method constituting the key element of the computer integrated development prediction methodology in materials surface engineering area and to describe the method, deriving from this concept, of technology e-transfer, enabling the practical industrial implementation of the materials science-heuristic research performed. The synergic influence of the concepts of e-foresight and technology e-transfer creates a full and integrated system of predicting the development of surface properties and structure formation technologies and of implementing the results of such research in a wide environment of managers and engineers working at industrial entities. In consistency with the primary objective, the technology e-transfer method is related to e-advisory, e-training and e-information and is buttressed with own scientific research in the areas resulting from the previously conducted e-foresight research and from the monitoring of current issues. Specialist research using specialist equipment is essential for the advisory, training and information functions as the essence of technology e-transfer and, regardless the source of finance, work must not be performed at individual order of specific enterprises. Expectedly, the interested enterprises will be using the results of the research available on-line via an interactive web platform established. The Open Access mode enables anyone to use such a platform for free and at equal terms, while preventing the selective solving of any scientific and technical problems. All entrepreneurs can propose the topic of research in the Open Access mode and everyone can then use the results of such research for free. The approach proposed provides that anyone anytime and without any restrictions can be provided with all the information. Besides, the monitoring of issues, being merely an indirect way of interaction with enterprises, should enable to focus research works on satisfying the real needs of a knowledge- and innovation-based economy.

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ISSN 2083-5191
ISBN 83-89728-95-8
EAN 9788389728951