



TEM microstructure investigations of aluminium alloys used as coating substrate

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ABSTRACT

Purpose: The aim of this paper was investigated structure and properties of gradient coatings produced in PVD process on AISi9Cu aluminium alloys.

Design/methodology/approach: The following results concern the structures of the substrates and coatings with the application of electron transmission and scanning microscopy; phase composition of the coatings using X-ray diffraction and grazing incident X-ray diffraction technique (GIXRD); microhardness and wear resistance.

Findings: The deposited coatings are characterized by a single, double, or multi-layer structure according to the applied layers system, and the individual layers are coated even and tightly adhere to the substrate as well to each other. The analysis of coatings obtained on the surface of cast aluminium alloys by the PVD processes show a clear - over 100% - increase of the microhardness, compared to the base material microhardness.

Practical implications: Achieving of new operational and functional characteristics and properties of commonly used materials, including the Al-Si-Cu alloys is often obtained by heat treatment, ie, precipitation hardening and/or surface treatment due to application or manufacturing of machined surface layer coatings of materials in a given group of materials used for different surface engineering processes.

Originality/value: The paper presents the research involving the PVD coatings obtained on an unconventional substrate such as aluminium alloys. Contemporary materials should possess high mechanical properties, physical and chemical, as well as technological ones, to ensure long and reliable use. The above mentioned requirements and expectations regarding the contemporary materials are met by the non-ferrous metals alloys used nowadays, including the aluminium alloys.

Keywords: Thin & thick coatings; PVD and CVD coatings; Structure; Properties

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

In recent years, together with the development of the automobile industry, and the demand to reduce energy consumption in the production process there is emerged a tendency to come back to aluminium alloys produced as sand casts made for high performance automatic lines. Examples of such options can be widely used technologies like: Cosworth, CPS, BAXI and HWS. These technologies provide a good filling of the cast dies with increased pressure and minimizing of oxidation of the used alloy. Among the most commonly used cast aluminium alloys the most frequently used are cast alloys containing silicon, traditionally called silumin [1-3]. Silumin due to its mechanical properties, good castability and corrosion resistance are primarily used in engineering and automotive industry. Characteristics properties of certain silumin types make them particularly useful for specific applications, related to the working conditions of the produced elements such as pistons for combustion engines and heavy-duty engines. In addition, aluminium alloys, with a share of the total weight of the motor vehicle reaches today approximately 200 kg, are also used for powertrain components (pistons, drive shafts, cylinder heads, cylinder blocks, gear boxes), body parts (frame and construction vehicles, truck cabins, engine bonnet, doors, seat structures, bumpers, roof cargo rails), chassis elements (braking systems, wheels, rear- and front bridges) as well as trailers, fuel tanks and heat exchangers [1-8].

Deposition of hard layers of nitrides, carbides or oxides on surface of the engineering materials in the PVD processes features the most intensely developing direction of extending the functional elements' life [7-15]. Selection of the substrate material onto which the investigated coatings were deposited by PVD technique in the presented project was not incidental either. Traditionally automotive components, products used in the building and power industries should have - apart from their special aesthetic features - a high corrosion-, erosion- and wear resistance. Thin, hard PVD coatings on a soft substrate, turn out to be an advantageous material combination from the material point of view [16-25].

The aim of this innovative work was to obtain best possible hybrid coatings, consisting of - a gradient transition layer, with a continuous change of one or more components reaching from the substrate to the surface top - as well as an outer coating using the cathodic arc evaporation process on the surface of the cast aluminum alloy, for the reason to increase of low stiffness of the substrate material [26-35].

2. Experimental procedure

Investigations were performed on samples made of AlSi9Cu cast aluminium alloy after heat treatment (Table 1). In order to determine the relationship between structure and properties of the achieved hybrid coatings, - a system composed of a soft substrate - transition gradient layer, with a continuous change of one or more components from the substrate to the outer surface - and the outer layer, independent coatings were produced by means of the cathodic arc evaporation PVD CAE (called: Cathodic Arc

Evaporation) on a substrate of aluminium cast alloys, Al-Si-Cu. PVD CAE process was implemented using the device DREVA ARC400 supplied by Vakuumentchnik by the cathodic arc evaporation method. The device is equipped with three pairs of independent sources of metals vapour. Prior to the coating process the substrate was cleaned chemically by washing and rinsing in an ultrasonic cleaner and dried in a stream of hot air. Moreover, the samples were ion-cleaned using Ar ions at a polarisation voltage of 800/200 V for 20 min. For the PVD coating process it was used water cooled discs with a diameter of 65 mm, containing pure metals (Cr, Ti) as well as alloy TiAl. The samples were coated in an inert Ar atmosphere as well in reactive gases atmosphere N_2 to obtain nitrides, and mixtures of N_2 and C_2H_2 in order to obtain carbonitride layers. The particles knocked out of the target surface react with the reactive gas molecules to form phases with specific of elements concentration ratio. Achieving of a right balance of elements concentration in the phase significantly improves the control of the coating properties. A gradient change of the chemical composition on the coating cross-section was achieved by changing the proportion of reactive gas dose or target evaporation current change on the arc sources. The determined coating process conditions are presented in Table 2. During the PVD coating process the substrates -made of cast aluminium alloys - move relative to the vapour sources, by performing of rotational movements in order to obtain uniform thickness of the layer, and preventing at the same time the phenomenon of so-called "shadow on the coated surfaces".

Structure investigations of the studied cast materials were performed using scanning electron microscope Zeiss Supra 35 with secondary electron detection. The examinations of thin foils microstructure and phase identification were made on the JEOL 3010CX transmission electron microscope (TEM), at the accelerating voltage of 300 kV using selected area diffraction method (SAD) for phase investigations. Qualitative and quantitative point-wise EDX microanalysis as well as the surface mapping of elements on the surface of the cast aluminium samples after heat treatment and surface treatment was performed on the scanning electron microscope Zeiss Supra 35 with X-ray spectrometer Trident XM4 supplied by EDAX. X-ray qualitative phase analysis of the investigated materials was performed using the Philips XPert diffractometer, with 0.05° steps and by a count rate of 5-10/s, using filtered $K\alpha_1$ X-rays, with the copper anode wavelength of $\lambda=1.54056$ nm. The measurements were performed in the 2θ angle range between 20° to 140° .

Microhardness investigations of the obtained PVD coatings were performed using the Vickers ultramicrohardness tester DUH 202 supplied by Shimadzu. The applied load for the measurements was equal 10 mN.

Wear test investigations of the PVD coatings were performed using the ball-on-disk method according to the requirements of the ASTM G 99 and DIN 50324 standards. The tests were carried out in dry friction conditions in horizontal configuration of the disk rotation axis. As a counterpart a tungsten carbide ball was applied with a diameter of 3 mm. The investigations were performed at room temperature, by a defined friction path distance with following testing parameters: $F_n=5$ N load, rotation speed of the disk 200 rot/min, friction path radius 2.5 mm, movement rate $v=0.05$ m/s.

Table 1.
Chemical composition of the investigated aluminium alloys

Chemical composition of the investigated aluminum alloys							
Alloy type	The mass concentration of main elements, %						
	Si	Fe	Cu	Mn	Mg	Zn	Ti
AlSi9Cu	9.094	0.1792	1.049	0.3608	0.2682	0.1409	0.0733

Table 2.
Deposition parameters of the investigated coatings

Coating parameters	Type of the achieved coating	
	Ti/Ti(C,N)-gradient/(Ti,Al)N	Cr/CrN-gradient/CrN
Base pressure [Pa]	5×10^{-3}	5×10^{-3}
Working pressure [Pa]	0.9/1.1-1.9/2.8	1.0/1.4-2.3/2.2
Argon flow rate measurement, cm^3/min	80*	80*
	10**	80**
	10***	20***
Nitrogen flow rate measurement, cm^3/min	0→225**	0→250**
	350***	250***
Acetylene flow rate measurement, cm^3/min	140→0**	-
	70*	60*
Substrate bias voltage, V	70**	60**
	70***	60***
Target current, A	60	60
Process temperature, °C	<150	<150

*during metallic layers deposition, **during gradient layers deposition, *** during ceramic layers deposition

3. Discussion of experimental results

Results of diffraction measurements achieved by the high resolution transmission electron microscope (Figs. 1-4) allowed to identify the Si, Al_2Cu , Al phases occurred in the aluminium substrate as well as CrN and TiN phases which occurred in the Ti/Ti(C,N)/(Ti,Al)N surface layer and Cr/CrN/CrN surface layer respectively. For both cases of the investigated bilayer coatings produced on aluminium light alloys had the crystalline character (Figs. 3, 4). The CrN phase was determined as a cubic phase of the 225-Fm3m space group with the lattice constant of $a=b=c=0.414$ nm. The (Ti,Al)N phase as a cubic phase of the Fm3m (225) space group with the lattice constant of $a=b=c=0.424173$ nm (Figs. 3, 4). Investigations performed on transmission electron microscope made it possible to investigate the character of the transition zone between the analysed Cr/CrN/CrN layer and substrate. It can be observed that the transition zone, separating the revealed phases having different crystal structure, shows a continuous transition character with a distinct heterogeneity. Electron diffraction investigations allowed to identify the CrN phase as the major phase in the surface layer, with the zone axis [-12-1], as well the Al- α phase present in the substrate with the zone axis [2-11] (Fig. 3). Especially investigations performed using the dark field technique on the transmission

electron microscope confirmed in case of the both produced layers that the size of the crystallites does not exceed the limit of ~ 20 nm in the majority of cases (Figs. 3, 4). Also a globular bulk shaped morphology and homogeneity of these crystallites was found, as well a low statistical dispersion in the range between 15 to 20 nm.

In order to investigate the structure and the existing relationship between the type of the substrate made from cast aluminium alloy, the type and technological conditions of the hybrid coatings production process, that means - a soft ground system - transition gradient layer, with a continuous change of one or more of its components reached from the ground to the outer surface - and the outer layer, - in the cathodic arc evaporation process metallographic tests were carried out. Coatings produced using the CAE-PVD technique are characterized by a clear heterogeneity connected to a number of microparticles in form of droplets occurred the structure (Figs. 5, 6). The highest heterogeneity of the surface area compared to second investigated coating is characteristic for the coating of the Ti/Ti(C,N)/(Ti,Al)N type, where a number of solidified droplets of the vaporized metal were identified (Fig. 5). The occurrence of these morphological defects is related to the cathodic arc evaporation process itself. Depending on the process conditions, including the kinetic energy of the drops sputtered into the metal substrate and the nature of the metal vapour source, the observed particles are clearly different in terms of shape and size.

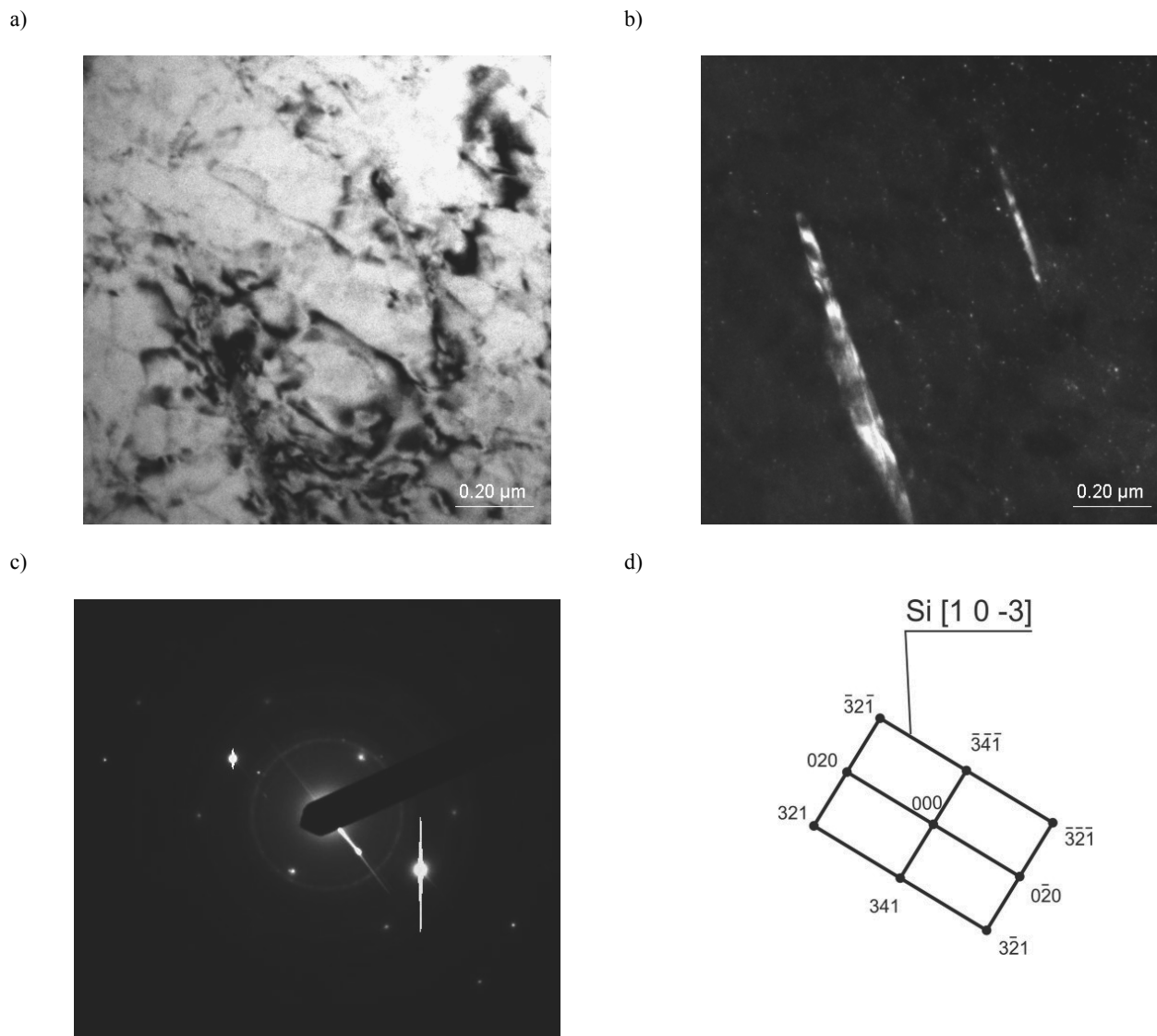


Fig. 1. Thin foil structure from the AlSi9Cu aluminium cast alloy: a) bright field image, b) dark field image, c) diffraction pattern from Figs. a), d) solution of the diffraction pattern from Fig. c)

As a result of metallographic fracture investigations of samples made from aluminium alloys coated with the analysed coatings, performed using scanning electron microscopy, there were identified a clear transition zone between the substrate and the coating. The obtained coatings reveals a compact structure, without visible delamination and defects, they are uniform and tightly adhere to each other as well to the substrate (Figs. 7, 8). Investigations of fractures confirm that the shells of the type Ti/Ti(C,N)/(Ti,Al)N show a layered structure, with a clearly visible transition zone between the coating and the gradient wear resisting coating, achieved by appliance of separate sources of the metal vapour (Fig. 7). The thickness of the Ti/Ti(C,N)/(Ti,Al)N layer is in the range from 3.2 to 3.5 μm . It was also found that the examined layers were not uniform and

consisted of three sub-layers, where the upper one had a thickness of ca. 0.6 μm . In the case of the Cr/CrN/CrN coating, where the same set of chemical elements in the gradient coating as well in the wear resistant coating any differences on the cross section has been found and the thickness of the layer is in the range from 1.7 to 1.9 (Fig. 8).

As a result of quantitative and qualitative X-ray microanalysis performed using the EDS spectrometer the presence of major alloying elements Mg, Al, Zn, Ti, Cr, C, N, was confirmed, both the investigated cast aluminium alloys as well the tested layers (Fig. 9, Table 3), also information were obtained about the mass and atomic concentration of elements in point-wise tested microareas of the substrate and of the obtained coating.

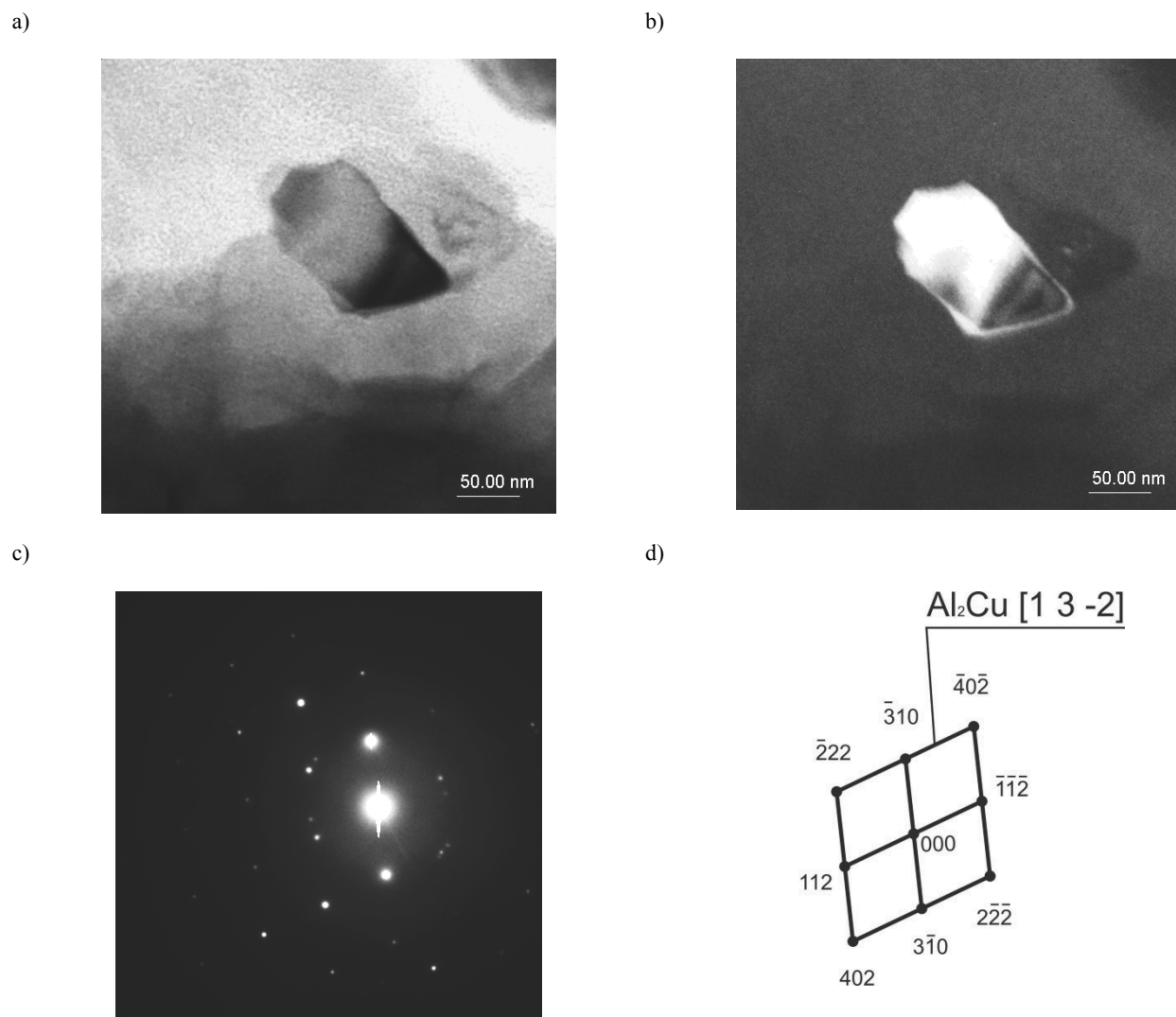


Fig. 2. Thin foil structure from the AlSi9Cu aluminium cast alloy: a) bright field image, b) dark field image, c) diffraction pattern from Figs. a), d) solution of the diffraction pattern from Fig. c)

The applied qualitative X-ray analysis method for phase composition performed in the Bragg-Brentano geometry confirmed the presence of the following phases: (Ti,Al)N, CrN, α -Al, β -Si in the investigated coatings as well in the substrate (Fig. 10). Some of the identified reflections are shifted relative to the reflection angle given in the JCPDS tables and their intensity is also different from the values given in these tables, which may indicate the presence of internal stresses in the investigated coatings, what is characteristic for the PVD process. Because of reflections overlapping coming from the substrate and the coating material, their intensity and a relatively small thickness of individual layers up to 3.5 μm , as well a similar 2θ angle values for the coatings of the Ti(C,N) and Ti(Al,N) type, the phase identification was difficult to perform. The presence of reflections from the substrate material was confirmed on all diffraction

patterns made from the coating, because of the thickness of the obtained coatings, smaller than the X-ray penetration depth of the investigated material. To obtain more accurate information concerning the analysed layers of the surface coatings in further investigations, there was applied a diffraction technique performed with a constant angle of the incident X-ray beam, what leads to achieving of diffraction lines coming from thin films by increasing the tested material volume. At different incidence beam angles, chosen experimentally and individually for each type of coating, there were reflections registered only from the thin surface layers (Fig. 10).

To determine the tribological properties of the investigated coating deposited on the aluminium alloys substrate, an abrasion test under dry slide friction conditions was carried out with the ball-on-disk method.

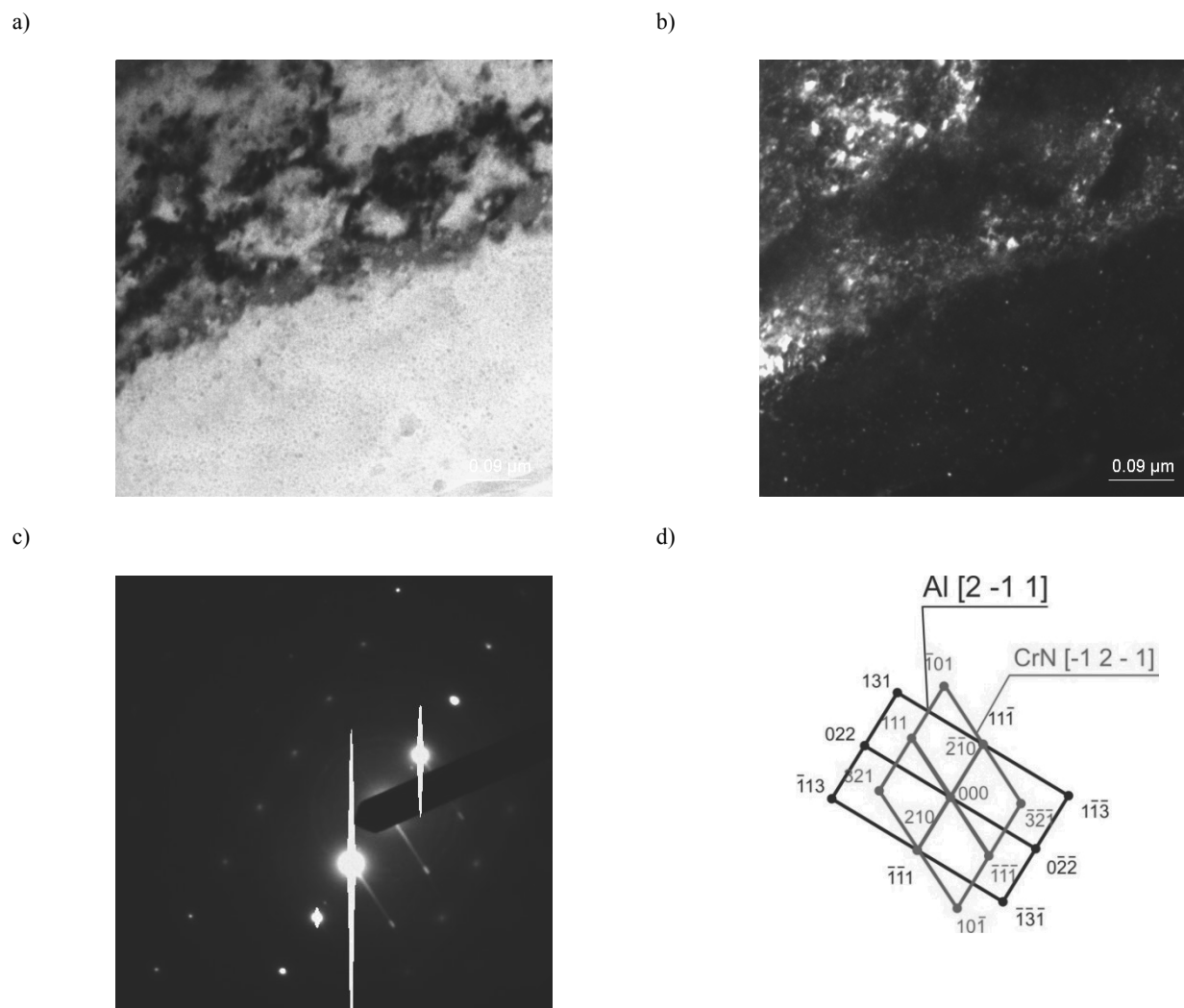


Fig. 3. Boundary between the Cr/CrN/CrN surface layer and the AlSi9Cu substrate: a) bright field, b) dark field, c) diffraction pattern of the monocrystalline surface CrN layer presented in Figs. a, d) solution of the diffraction pattern presented in Fig. c

Table 4 presents the friction coefficient and sliding distance results for each type of the investigated substrate. Under technically dry friction conditions, after the wearing-in period, the friction coefficient recorded for the associations tested is stabilized in the range 0.16-0.33 depending on the used substrate and coatings. Comparing the friction coefficient results with the friction path length, it was found that the best wear resistance is characteristic for materials coated with Ti/Ti(C,N)/(Ti,Al)N. However, the results of the friction path length for the Ti/Ti(C,N)/(Ti,Al)N coatings were at a level exceeding even 5 times the results of the friction path length achieved for the Cr/CrN/CrN coatings.

As a result of microhardness investigation using the Vickers method it was revealed, that the applied PVD coatings on the aluminium alloys substrate makes it possible in an effective way to enhance the resistance to plastic deformation of the analysed surfaces. The hardness of the aluminium alloy substrate without

coating are AlSi9Cu-100 HV, as settled upon hardness tests. It was confirmed that both the chemical as well the phase composition, working conditions of the PVD process as well the substrate material influence the microhardness of investigated coatings. In the case of coatings produced by the cathodic PVD process in a N₂ nitrogen atmosphere of the type: Cr/CrN/CrN there was found an clearly microhardness increase of 100% compared to the microhardness of the substrate material (after precipitation hardening).

The microhardness of the produced coatings, in this case did not exceed the value of 2000 HV. However, for coatings with a gradient carbide-nitride coating obtained in an atmosphere containing CH₄ and N₂ of the type: Ti/Ti(C,N)/(Ti,Al)N there was found the highest increase of surface microhardness over 2000HV (Table 4), which also significantly correlates with the wear resistance of these coatings.

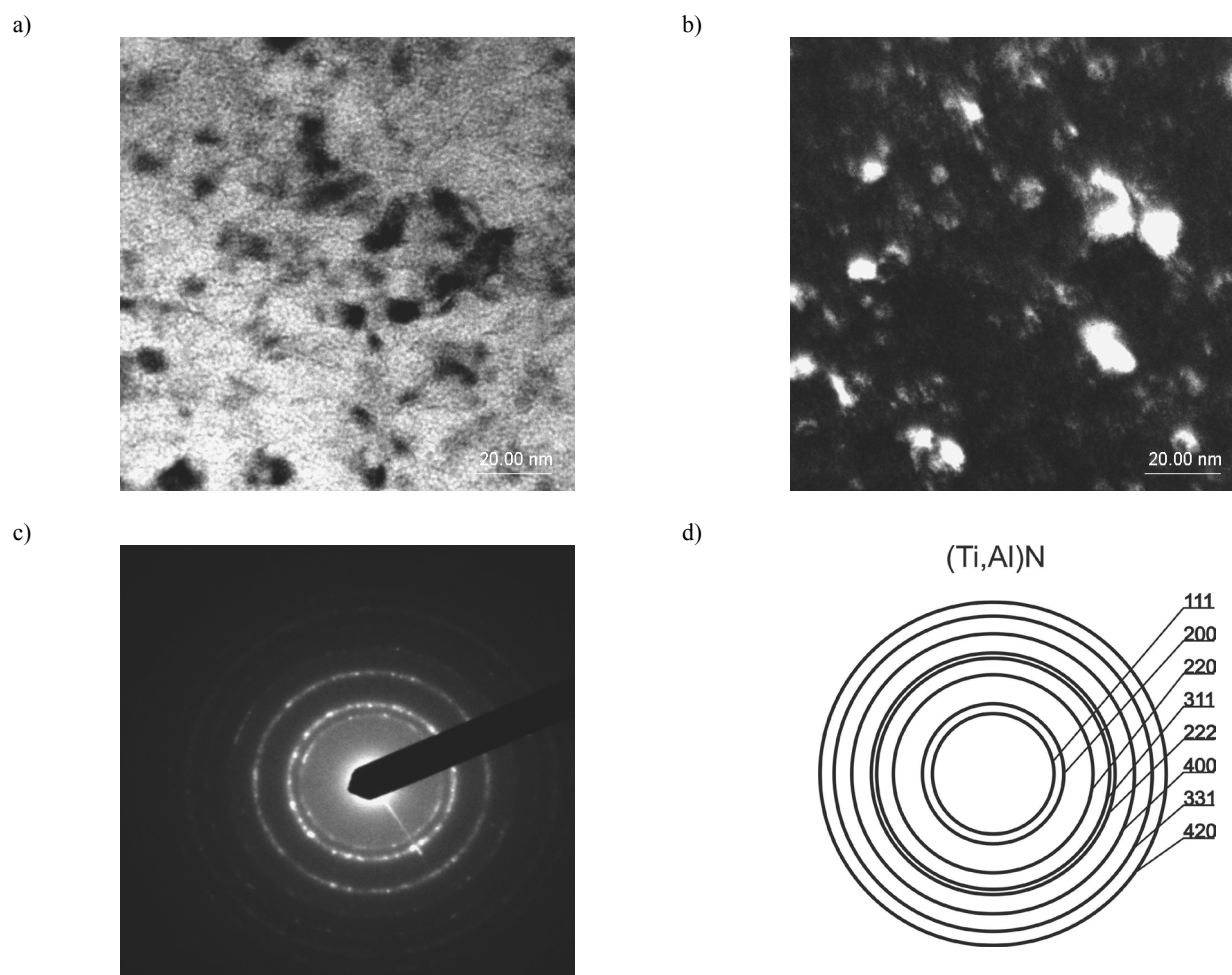


Fig. 4. Fine crystalline structure of the (Ti, Al)N coating fracture: a) bright field, b) dark field, c) diffraction pattern of the polycrystalline surface (Ti, Al)N layer presented in Figs. a, d) solution of the diffraction pattern presented in Fig. c

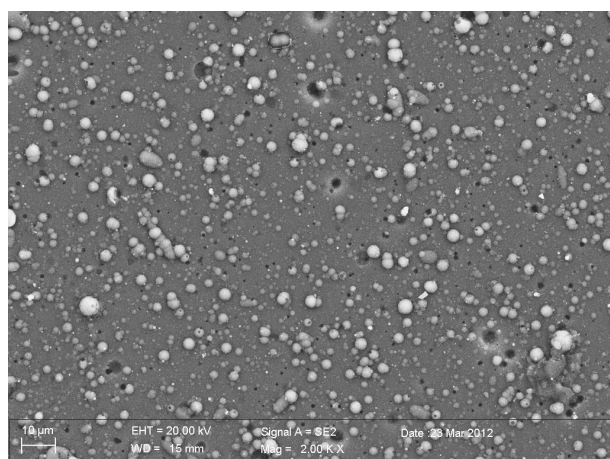


Fig. 5. Surface morphology of the Ti/Ti(C,N)/(Ti,Al)N layer coated on the AlSi9Cu aluminium substrate

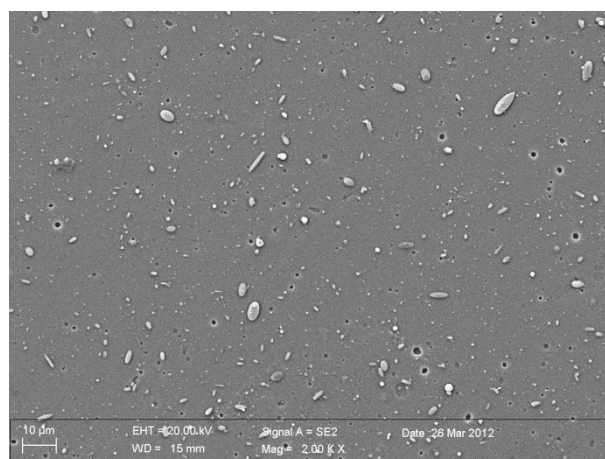


Fig. 6. Surface morphology of the Cr/CrN/CrN layer coated on the AlSi9Cu aluminium substrate

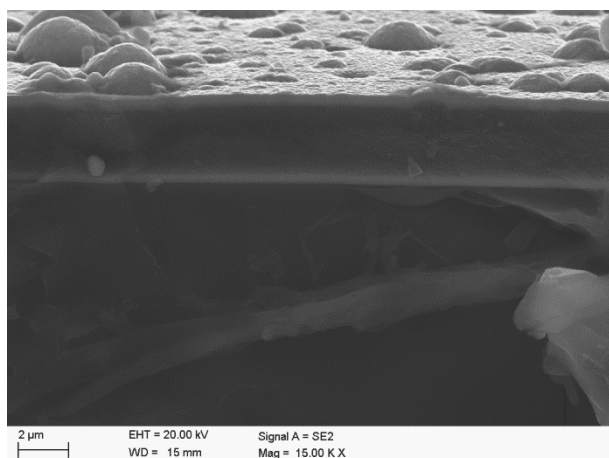


Fig. 7. Fracture of the Ti/Ti(C,N)/(Ti,Al)N coating on the AlSi9Cu aluminium alloy

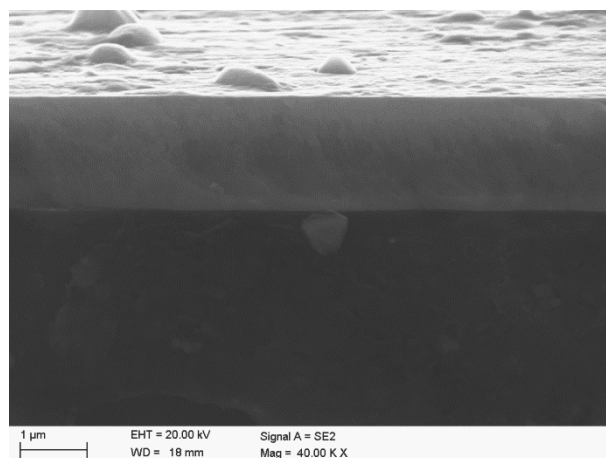


Fig. 8. Fracture of the Cr/CrN/CrN coating on the AlSi9Cu aluminium alloy

Table 3.

The results of quantitative chemical analysis from third 1, 2, 3 areas of coating Cr/CrN/CrN deposited onto substrate from AlSi9Cu alloy marked in Fig. 9

Chemical element	The mass and atomic concentration of main elements, %	
	mass	atomic
Analysis 1 (point 1)		
N	7.92	22.11
Cr	86.78	69.77
Al	03.74	05.79
Si	01.56	02.33
Analysis 2 (point 2)		
N	9.66	27.97
Cr	88.14	68.77
Al	1.56	2.34
Si	0.64	0.93
Analysis 3 (point 3)		
Al	75.02	75.78
Si	22.97	22.29
Cu	0.47	0.20
Mg	1.54	1.73

Table 4.

The characteristics of the tested coatings

AlSi9Cu			
Coatings	Hardness, HV	Friction coefficient	Sliding distance, [m]
Substrate	100	-	-
Cr/CrN/CrN	1950.1	0.2-0.29	10
Ti/Ti(C,N)/(Ti,Al)N	2139.1	0.16-0.33	52.3

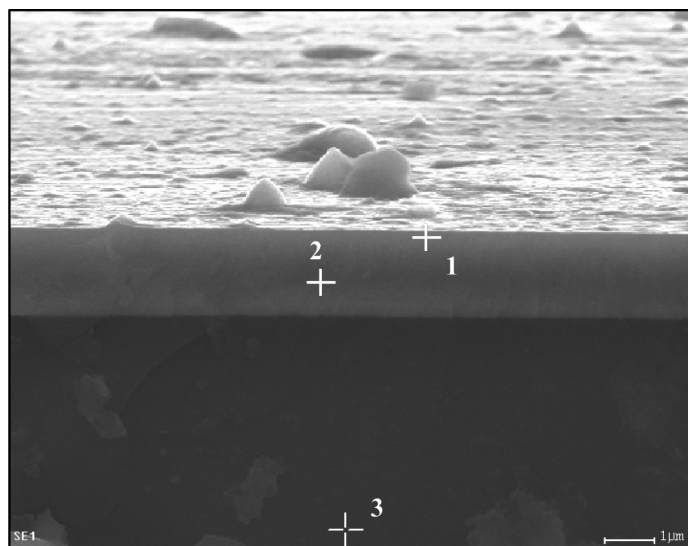


Fig. 9. Cross-section SEM images of the Cr/CrN/CrN coating deposited onto the AlSi9Cu substrate

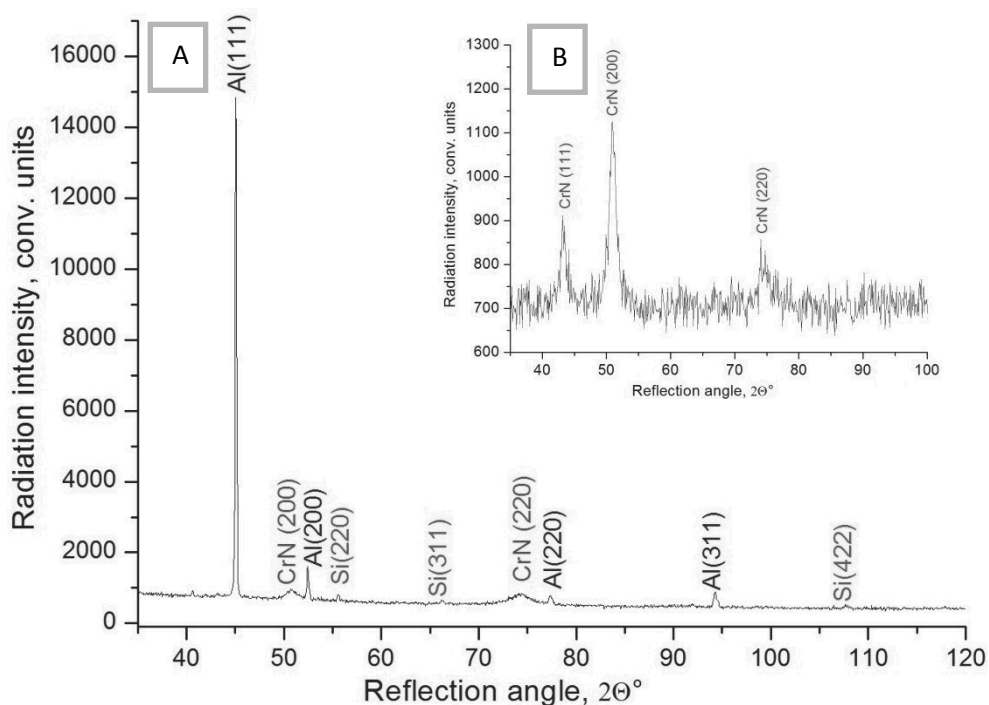


Fig. 10. (A) X-Ray diagrams of the Cr/CrN/CrN layers coated on the AlSi9Cu aluminium alloy, obtained using the Bragg-Brentano method (B) X-Ray diagrams of the coating performer in a stable angle of $\alpha=4^\circ$

4. Summary

Increase of the exploitation life and stability of elements produced from the Al-Si-Cu alloy is also possible using the processing techniques of surface layer refinement in the vapour

deposition process. Due to the character of the investigated material (aluminium alloys) and its relatively low melting point, the technological PVD processes were performed at temperatures of up to 150°C. Results of diffraction measurements achieved by the high resolution transmission electron microscope allowed to identify the Si, Al_2Cu , Al Al phases occurred in the aluminium

substrate as well as CrN CrN and TiN phases which occurred in the Ti/Ti(C,N)/(Ti,Al)N surface layer and Cr/CrN/CrN surface layer respectively. For both cases of the investigated bilayer coatings produced on aluminium light alloys had the crystalline character. In order to evaluate the crystallite size of the obtained phases the dark field technique was used. The size of the measured crystallites is in the range of up to 20 nm. The achieved coatings are characterized by a clear heterogeneity of the surface associated with the occurrence of a number of microparticles in the structure in the shape of droplets sputtered during the deposition process and some cavities occurring due to falling out of some droplets during solidification. As a result of fracture investigation performed using the scanning electron microscope, of the analysed PVD coatings it was found, that the deposited coatings are characterized by a single or multi-layer structure according to the applied layers system, and the individual layers are coated even and tightly adhere to the substrate as well to each other. Moreover, the analysis of coatings obtained on the surface of cast aluminium alloys by the PVD processes, show a clear - over 100% - increase of the microhardness, compared to the base material microhardness. The increase of the surface microhardness of aluminium alloys as a result of the coating deposition from gas phase often corresponds with increased wear resistance of the investigated substrates, like in the case of in of Ti/Ti(C,N)/(Ti,Al)N coatings.

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