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The effect of TiAl and Ti_3Al reinforcement on microstructure changes and properties of aluminium matrix composites

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ABSTRACT

Purpose: The main purpose of this monograph research is to present the results of the author's own investigations concerning development and characterisation of the aluminium matrix composite's reinforced with intermetallic particles being the outcome of the concept of merging the mechanical alloying with the powder metallurgy. As the result the nanostructured composites were fabricated with the exclusive mechanical properties as the result of fine microstructure.

Design/methodology/approach: Powder metallurgy (PM) combined with (MA) offers innumerable advantages over casting metallurgy, making possible to improve the existing properties but also conferring new properties. Results presented in this paper were obtained in such processes. Experiment have been developed to improve the characteristics of aluminum matrix composites, because of produced fine and uniform dispersions of reinforcements particles. Final consolidation, shaping and forming have been done by hot extrusion. Testing of mechanical properties encompassed hardness testing, tensile as well as compression testing, and determining the sliding wear resistance. Detailed structural examinations have been carried out to determine the effect of mechanical alloying and reinforcing particles on microstructure changes and properties of investigated composites.

Findings: Applying mechanical alloying route of composite powders production, makes it possible to obtain diminution of reinforcing particles size as well as homogenous reinforcement particles distribution. Extruded composites are characterized by a very homogeneous distribution of intermetallic particles and the absence of any reaction and with good cohesion at the matrix/particle interfaces. Observed changes in the microstructure, influence on the mechanical properties of obtained composite particles.

Practical implications: Aluminium matrix composites (AMCs) reinforced with ceramic particles have already found several applications. However, they suffer from some drawbacks due to high abrasiveness and brittleness of ceramics. Lately, intermetallic particles have emerged as possible substitutes for ceramic reinforcements in aluminium alloys.

Originality/value: Employment of the modern composite production techniques, and especially of the nanostructured one with emerging potetial of intermetallics as the reinforcing phases, makes it possible to obtain specific properties thus application. The paper presents extensive knowledge related to microstructure and effect of reinforcement on it.

Keywords: Composites; Powder metallurgy; Mechanical alloying; Structure; Properties

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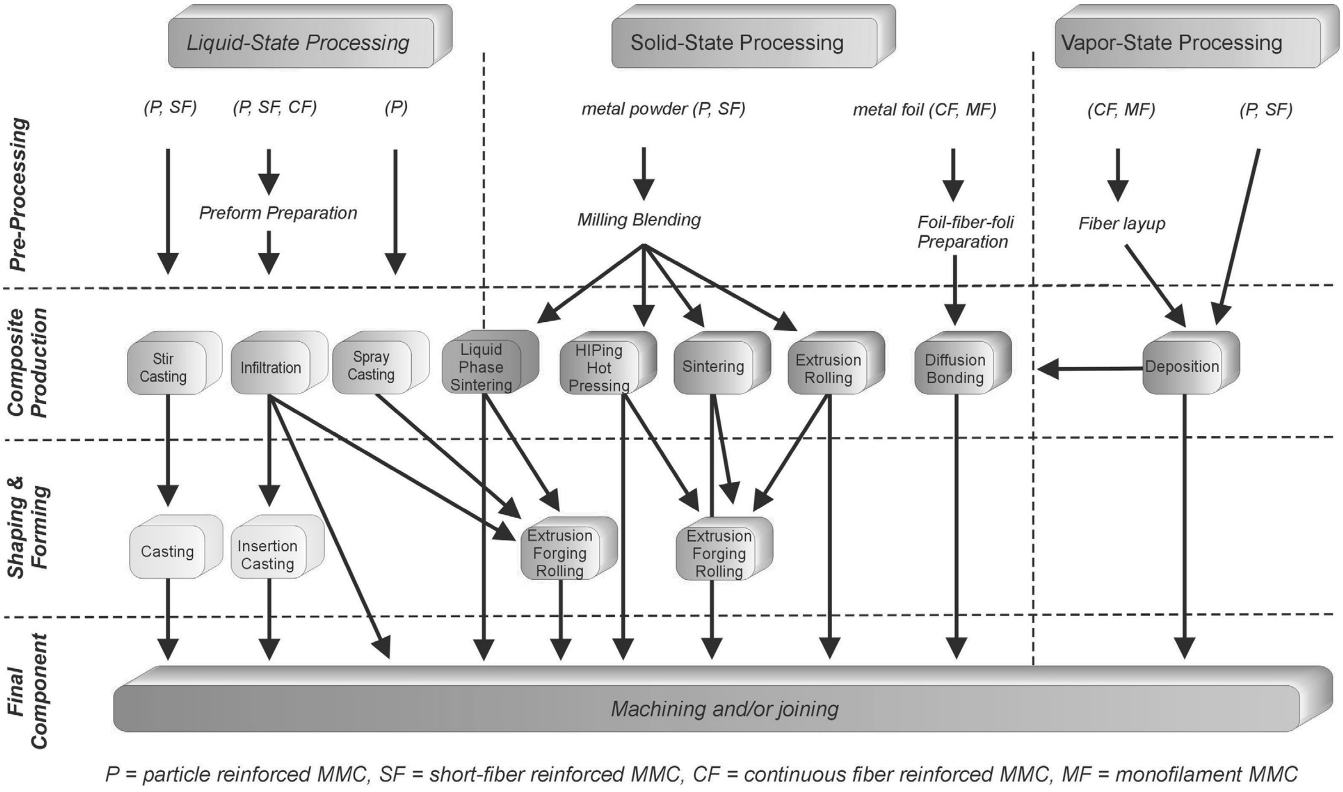
RESEARCH MONOGRAPH

1. Introduction

Investigation into development and fabrication of new materials with special properties, which are not attained by the traditional constructional materials, have added to the significant interest in materials with the preplanned properties, in their broad ranges. As the result of the done research, the continuously growing variety appears in the global market of composite materials, the polymer-, metal-, and ceramic ones. They find many engineering applications, especially in areas like aviation industry, as well as the automotive- and machine building ones, in transportation, in sport equipment manufacturing, and also in the design of spacecrafts [1-10]. Composite materials with Al matrix feature a special group in this area, and among them, composites reinforced with the discrete particles, attaining the higher level of properties in comparison with alloys without the reinforcing particles, and moreover, composite materials reinforced with the dispersive particles demonstrate the higher temperature stability and better abrasion wear resistance. Albeit these materials do not attain the same level of mechanical properties as composite materials reinforced with high-strength continuous fibres, their cost and fabrication methods may be easily accepted, and the obtained properties reach the satisfactory level in many applications. Many different technologies may be used for fabrication of composite materials with aluminium matrix, in which matrix materials many be in the gaseous state (vapour), liquid, solid, or semi-liquid (Fig. 1). For many of these technologies, occurrence of defects like the poor interface of the reinforcing phases with the matrix, excessively wide reaction zone or its absence, segregation and non-homogeneous properties, etc., is the deficiency, making difficult the wide use of these materials on the commercial scale [11-19].

In case of composite materials with aluminium matrix, most research to date was carried out and described in the literature for the ceramic reinforcing phases, mostly the Al_2O_3, Si_3N_4, and SiC. In case of using ceramic reinforcing phases, employment of the new reinforcing phases mostly has the objective to avoid the deficiencies occurring in these materials, and also improvement of service properties of the newly developed composite materials. Employment of particles of the intermetallic phases as the relevant reinforcing material in aluminium alloys is dated from the 1980s. Nowadays, two main drawbacks are encountered in using this kind of the reinforcing materials. These are their still high fabrication cost and high chemical reactivity of many intermetallic phases in the interfacial boundary area: reinforcing particle - matrix material, both during the manufacturing process ("in situ" manufacturing processes) and also in service at elevated temperature. Reactive diffusion at the interfacial boundary: reinforcement - matrix influences the zone in which diffusion takes place of the composite material components, entailing structure modification, which - in turn - may affect the macroscopic changes of the mechanical- or thermal properties of the composite [20-43].



P = particle reinforced MMC, SF = short-fiber reinforced MMC, CF = continuous fiber reinforced MMC, MF = monofilament MMC

Fig. 1. Steps of metal matrix parts fabrication in different processes [10]

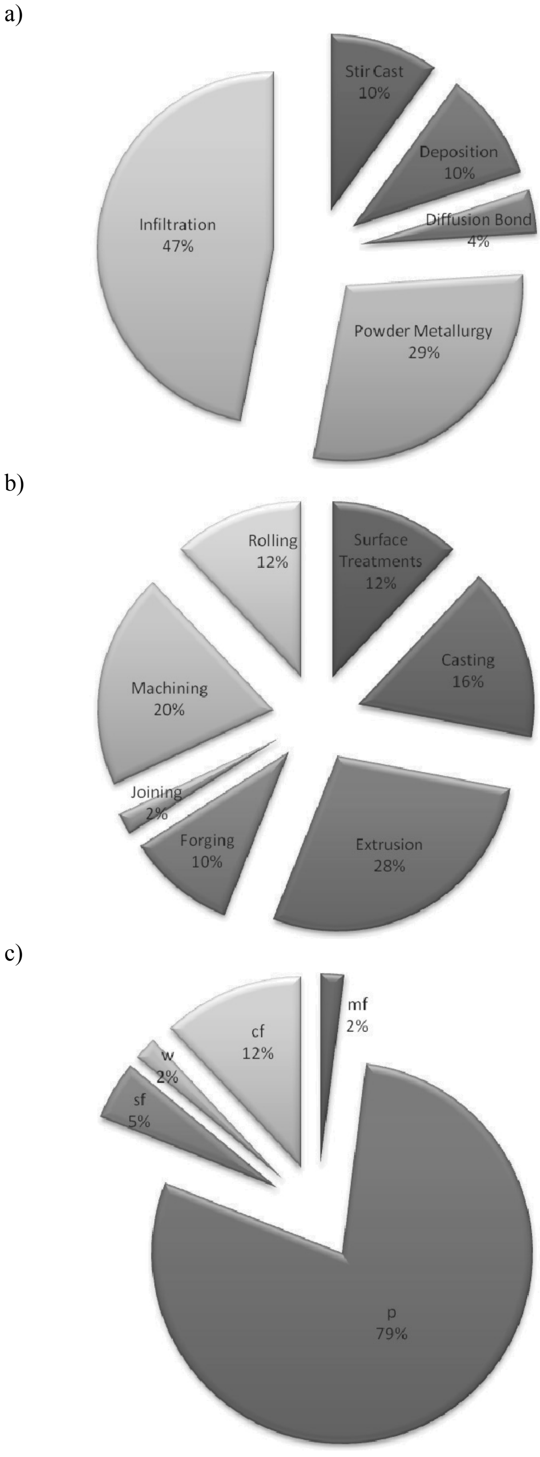


Fig. 2. Percentage of companies a) manufacturing MMC materials by the categorised primary processing methods, b) performing the categorised secondary processing methods, c) applying a particular reinforcement type [11], sf- short fibres, w - whiskers, cf - continuous fibres, mf - monofilament, p - particles

Processes of mechanical alloying and mechanical milling have found application in the last years fabrication processes of composite materials, albeit these processes were taken into account earlier, because of the possibility of developing in them the intermetallic phases, supersaturated solid solutions, amorphous- or nanocrystalline materials. These processes, used in manufacturing of composite materials reinforced with particles, eliminate to the highest degree all problems mentioned above, and - in addition - ensure the best distribution of the reinforcing particles in the matrix and good connection with it. Properties of the newly developed alloys and composite materials will get clearly improved thanks to the possibility to obtain the amorphous structure during the mechanical alloying process or to obtain the nanocrystalline or sub-nanocrystalline materials. The synergistic action of the structure modification processes in the mechanical synthesis process, along with the dispersive hardening of materials by introducing the reinforcing particles with the additional action of the highly dispersive particles of oxides originating during the mechanical synthesis as thin layers on the outer surface of powders, which next, in the extruding process are homogeneously distributed in the structure, which allows to expect a clear improvement of the mechanical properties and thermal stability, and higher abrasion wear resistance respectively. Literature studies show that the prevailing majority of reports pertain to the composite materials reinforced with particles of ceramic materials, mostly Al_2O_3 and SiC, out of which many were fabricated using the powder metallurgy and mechanical milling technologies. Employment of intermetallic phases as reinforcement is described relatively rarely; however, research projects like that are ongoing, and lastly their number grows significantly. Moreover, albeit so many centres deal with the intermetallic phases' problems, only a few of them carry out research using this type of phases as particles reinforcing the composite materials. Because of the rather big group of the available intermetallic phases, only a few publications are connected with the intermetallic phases of the Ti-Al equilibrium system, referring mostly to cases using the intermetallic phase growth method "in situ" from the metal bath, and in publications, in which powder metallurgy methods are used for fabrication of the composite materials described in them, mechanical alloying - was not used for improvement of the reinforcing particles distribution and also for refinement and resultant structure reinforcement [45-78].

One should also note that introducing the new reinforcing phases is in the canon of the currently ongoing research projects and its goal is mostly improvement of the service properties of composite materials with Al matrix, and also avoiding the deficiencies resulting from employment of the ceramic reinforcing materials, connected mostly with their brittleness, maladjustment of the thermal expansion coefficients resulting with the low thermal fatigue resistance, limited wettability, abrasion interactions, and problems with recycling. Introduction of Ti_3Al and TiAl particles of the intermetallic phases as reinforcement eliminates most of all these deficiencies and makes it possible to use advantages of these materials, mostly the higher ductility, lower abrasivity, and obtain the thermal expansion coefficients close to those of the Al alloys. Features characteristics for these materials make minimising the construction mass possible, simultaneously maintaining the required mechanical properties, high stiffness, and abrasion wear resistance.

2. Materials for research and experiments

2.1. The selection of the constituents

For aluminium matrix composites, the matrix selection plays a significant role taking into account final properties and processing rout. Ductility, toughness and high temperature properties are strongly dependent on the matrix. In this study Al alloys belonged to 6xxx series were used because of their excellent combination of properties and their easy processing. As a matrix material air atomised powders of aluminium alloy - grade EN AW-Al Mg1SiCu from The Aluminium Powder Company Ltd. (UK) were used. The chemical composition of the alloy is given in Table 1. The particle size of the powder was < 75µm with the volume size distribution D50 = 29.6 µm. SEM morphology and microstructure of the initial (as received) powder particles used are given in Figure 3.

Over the past few decades ceramic reinforced Al alloys have been considered as the candidate to replace heavy iron based parts in many application. However because of mismatch between coefficient of thermal expansions of matrix and reinforcement, high abrasiveness and recycling difficulties their potential for large scale use has been restrained. In the last years new reinforcement candidates such as intermetallics compounds with outstanding mechanical properties and good thermal stability making them a powerful materials to be used in particulate reinforced composites have appeared. Among them one of the more promising system is Ti-Al one because of low density and excellent mechanical properties especially at high temperature application. In this sense application of these reinforcements came from the possibility to make use of low density, slight difference of CTE but still permit to produce aluminium composites with good mechanical properties and good resistance. Taking into account above as reinforcement particles TiAl and Ti_3Al intermetallics compound produced by SE-JONG Materials Ltd. (Korea) were used. The chemical composition of powders used are given at Table 2. Particles size were less than 75 µm for powders of TiAl and less than 50 µm for Ti_3Al titanium aluminide, Figure 4.

Table 1.
Chemical composition of the atomized aluminium alloy powder

EN AW-AlMg1SiCu	Elements' concentration, weight %						
	Fe	Si	Cu	Mg	Cr	Others	Al
	≤ 0.03	≤ 0.63	≤ 0.24	≤ 0.97	≤ 0.24	<0.3	Bal

Table 2.
Chemical composition of the titanium aluminide powders

Particle type	Elements' concentration, weight %						
	Ti	Al	V	Fe	N_2	O_2	H_2
Ti_3Al	≤ 83.36	≤ 15.35	0.55	0.025	0.06	≤ 0.59	≤ 0.15
TiAl	≤ 36.25	≤ 62.14	0.32	0.015	0.043	≤ 0.63	≤ 0.10

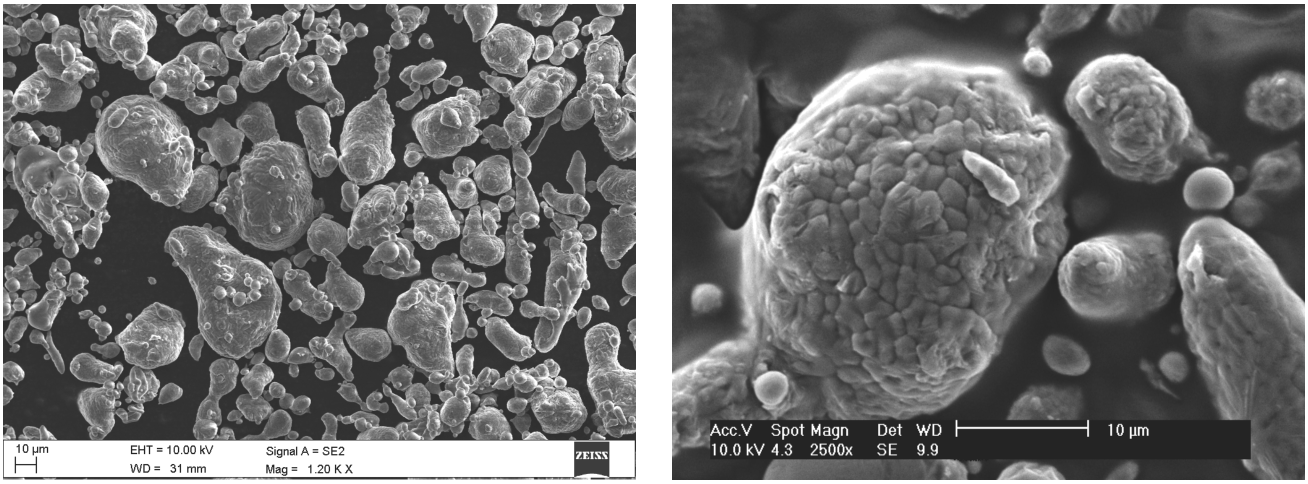


Fig. 3. Morphology of as received EN AW-AlMg1SiCu aluminium alloy powder particles used in the experiments, SEM

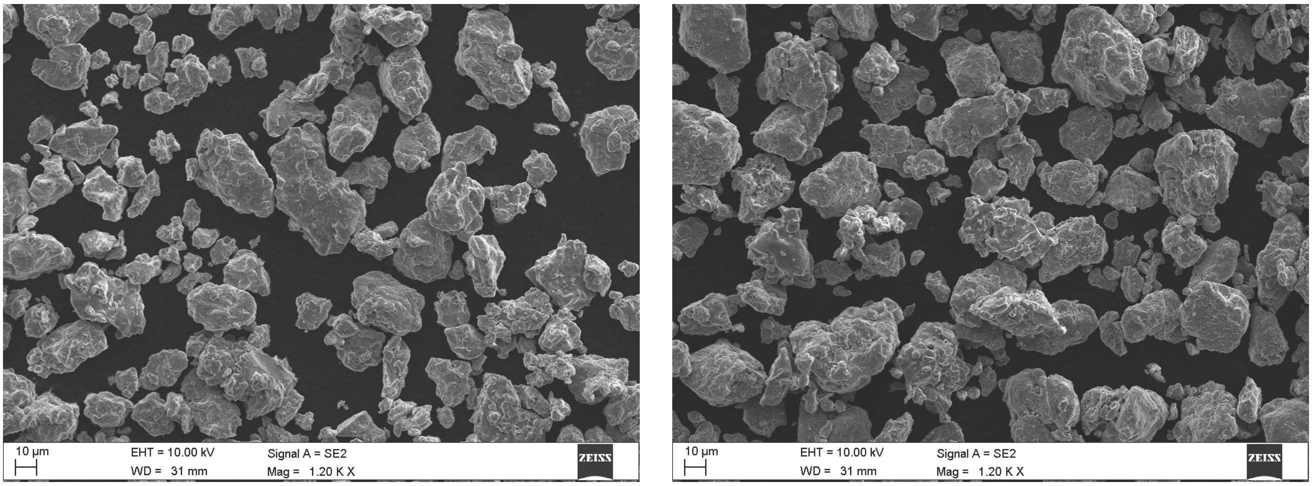


Fig. 4. Morphology of as received titanium aluminides powder particles used in the experiments - left TiAl, right Ti_3Al, SEM

2.2. The processing route

Powder metallurgy techniques combined with mechanical milling offers innumerable advantages over casting metallurgy, making possible not only to improve the existing properties but also conferring new properties. Among all processes that have been developed to improve the characteristics of aluminum matrix composites, the mechanical alloying MA process has received more attention because of produced fine and uniform dispersions of reinforcements particles, which could not be made by conventional powder metallurgy methods, by means of high-energy ball milling of powders. The process of MA begins with the preparation of powder mixture in the right proportion and loading the powder mix into the mill along with grinding balls. Such a mix is than milled for the desired length of time to reach steady state, when the composition of every created powder particles is the same. To produce mechanically alloyed powders different type of high energy milling equipment are used (differences are in their efficiency of milling, capacity, possibility of cooling or milling atmosphere control, etc.). For conducting MA experiments planetary ball mills are used very often. The name of the mill comes from the planet like movements of its vials. In such an arrangement the centrifugal force produced by the rotating around own axes vials and that produced by the rotating support disc both acts on the grounded powders and the grinding balls. Finally running down the inside wall of the vial grinding balls causes friction effect, when another observed effect lifting off and travelling freely through the inner chamber of the vial and colliding against the opposite wall causes impact (Fig. 5).

The process of mechanical alloying consists in repeated welding-fracture-welding of a mixture of powder particles in a high-energy ball mill. The central event is that the powder particles are trapped between the colliding balls during milling undergoing deformation and/or fracture process. In mechanical alloying of at least one ductile component, there is an initial stage when deformation dominates the process, followed by a stage in which welding is the predominant.

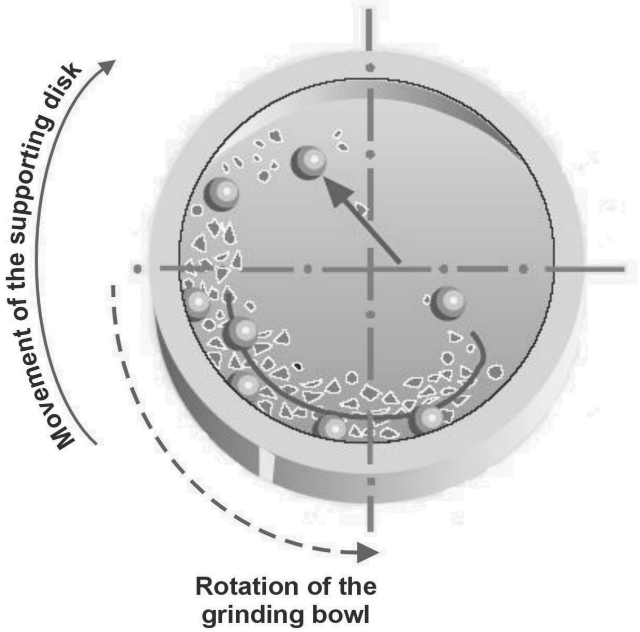


Fig. 5. Diagram depicting movements of the bowl, supporting disk and ball motion inside the ball mill [12]

After a certain period of milling, the powder is hard enough and fracture dominates the process. At the end of the process, the powder reaches a steady state characterised by equilibrium between welding and fracture [4,5]. The temperatures involved during the material production by powder metallurgy are lower which promotes a less interaction between the materials, minimizing interfacial reactions and making possible to reach superior mechanical properties. The use of PM and MA to fabricate metal matrix composites allows also to better control of the volumetric fraction of the reinforcement in a relatively large range and their uniform distribution [6,7].

The effect of mechanical milling was evaluated by comparison of two rout of powder mixture preparation. In this experiment two types of ball mills were used: a low energy powder blending in the horizontal ball mill and mechanical alloying in high energy planetary ball mill (Fig. 6).

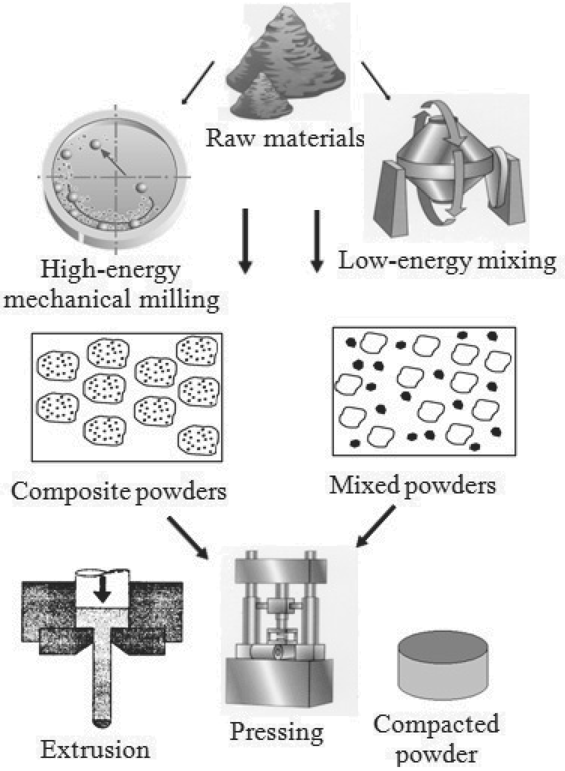


Fig. 6. Diagram of investigated composite fabrication route

The low energy mixing process consists of powders mixing at horizontal ball mill for 2 hours, ball diameter 10 mm, ball material quenched stainless steel AISI 420, ball to powder weight ratio 10:1, rotation speed 150 rpm, process control agent Microwax (1%).

Composite powders of aluminium alloy matrix reinforced with titanium aluminides were produced by mechanical alloying by high-energy ball milling using an high-energy ball mill (Fritsch GmbH, model ‘Pulverisette 6 and 5’) with the following parameters: ball-to-powder weight ratio: 6:1; ball diameter: 20 mm; ball material: AISI 420 quenched stainless steel; speed 700 rpm. A total of 1% (wt.) of microwax was added to control the process (PCA). No atmosphere control gas was used. The high-energy milling time was the time necessary to complete the mechanical alloying process, from the point of view of the phenomenological aspects, determined for each composition. The obtained composite mixture and composite powders were characterized by performed measurements of the apparent density, flow rate for 50 g of powder [10], as well as morphological and metallographic, examination. Sets of mixtures with 5, 10, 15 and 20% (wt.) for each of reinforcement type and preparation process were prepared.

Subsequently prepared powders were compacted in cylindrical die. During the pressing of metal powders in the first step, particles rotate and reposition themselves, witch leads to the collapsing of the bridges and filling of the hollows. With pressure increase, increased contact of the particles is observed because of plastic deformation. Than thin oxide skins break down and powder particles agglomerates by cold welding. With increase of cold working and increase of contact areas adhesion between particles increases. If particles reach plastic deformation limit they disintegrate, if shear deformation of surfaces take place mechanical cold welding is observed. All this processes take place on somehow distinguishable stages but in separate powder particles they may take place simultaneously.

The powders blends and composite powders were uniaxially compacted - cold pressed in the 25 mm in diameter cylindrical die with 300 MPa. To protect them, preforms surface were coated with thin layer of graphite suspension in oil. In case of samples after mechanical alloying, to avoid the excessive grain growth due to the high level of stored energy, annealing process were performed at 400°C for 1 h.

A final stage of MMC production was their hot extrusion in hydraulic press with capacity of 10 t. The experimental procedure for hot extrusion consists of two stages. At the beginning compacted preforms were loaded into the die, than die and punch were heated to 500-510°C. Thermocouple located at the die measured and controlled the preform temperature. Afterwards when the desired temperature was reached and after holding time of 5 min extrusion starts with an extrusion ratio of 25:1 and loading rate 0,5 mm/s, obtaining full dense materials. The die surface were treated with lubricant, mixture of grease oil and graphite powder. Extrusion process was performed without canning and degassing. The composite bars with the diameter of 5-8 mm were obtained as the end product and were studied in T1 - as produced condition.

Morphology examination of the starting powders and observations of the composite powder evolution during mechanical alloying were carried out on Zeiss Supra 35 (SEM) scanning electron microscope. Metallographic examinations of the composite materials with aluminium alloys matrix reinforced with the TiAl and Ti_3Al particles were carried out on LEICA MEF4A optical microscope as well as on the scanning electron microscope, metallographic images were taken of sections transverse and longitudinal in respect to the extrusion direction. The specimens were prepared by standard metallographic techniques and etched in Keller’s solution. Microstructure observations were made also in transmission electron microscope FEI “Titan”, HRTEM. Thin foils of investigated composites were prepared by mechanical pre thinning, followed by low angle ion polishing realized in Gatan PIPS^{TM} high milling rates unit. The microstructure examination and diffraction investigations of phase composition of the thin foils were made on the transmission electron microscope at the accelerating voltage of 300 kV. The diffraction patterns from the transmission electron microscope were solved using a computer aided program.

Particle size and distribution was determined by laser diffraction, Malvern Mastersizer S, with automatic calculation of volume size distribution, D10, D50 (considered to be the average particle diameter by mass) and D90. Phase compositions of the investigated materials were determined using the X’Pert diffractometer with the step data logging, employing the filtered $K\alpha$ X-ray radiation.

Microhardness tests of the composites powders were made on nanoindentation tester with Berkovich indenter and hardness tests on extruded composite materials were made on hardness tester with the Vickers indenter. Several indentations were made on the transverse section diameter for specimens taken from bars obtained by extrusion to determine their average hardness.

Static compression and tensile tests of the fabricated composite materials were made on the ZWICK 100 type testing machine at room temperature. Yield stresses (YS), ultimate tensile strength (UTS) and compression strength were determined.

The tribological tests were carried out with the use of equipment for „pin-on-plate" reciprocating tests. The „pin-on-plate" test was made on Tribometer CSM. The wear tracks were examined on a confocal microscope. The replaceable pin in the form of a ball made with Al_2O_3, loaded with 3, 3.5, 4, 4.5 N force is slid on the flat surface of the sample tested. It must be emphasised here that the ball surface wear is negligibly low.

3. Results and discussion

3.1. Characterisation of composite powders

As previously mentioned, the main process which takes place in a mill during the MA method to produce quality powders with controlled microstructure is the repeated welding, fracturing, and rewelding of a mixture of powders. The morphology of the initial powders is modified when they are subjected to ball collisions. It is worth noting that the effects of collisions on the milled powders depend on the type of the constituent particles.

It has been shown that the initial ball-powder-ball collision causes the ductile metal powders to flatten and work hardened when they are cold welded and heavily mechanically deformed. They are brought into intimate contact, forming layered structure of composite particles consisting of various combinations of the starting ingredients.

Further milling results in cold welding and deformation of the layered particles and a refined microstructure is obtained. Due to the initially low hardness of the starting elemental powders, the lamellar spacing of the agglomerated particles are quickly reduced upon further milling. Increasing the MA time increases the hardness and this leads to fracturing of the agglomerated powders into smaller particles. In the following stage, the welding predominates, causing the equiaxed particle formation. Then welding and fracture mechanism reach equilibrium and the formation of particles with randomly oriented interfacial boundaries. The final stage is characterized by the steady state process, in which the microstructural refinement can continue, but the particle size and size distribution remain approximately the same.

When considering the result obtained with the matrix alloys EN AW-AlMg1SiCu there is good agreement between these description and observed morphological powder changes. With presence of intermetallics particles submitted to high energy mechanical alloying, the system have to be described as partially ductile - brittle one. At the beginning in the first stage of milling, the ductile particles and intermetallics undergo deformation but with different rate, which brings brittle particles to fragmentation. Then, when welding of ductile particles starts, the brittle particles come between two or more ductile particles at the instant of the ball collision. Fragmented reinforcement particles will be placed in the interfacial boundaries of the welded metal particles, as the result formation of a real composite particle begins. Schematic presentation of mechanical alloying for ductile - brittle system was proposed by (Fig. 7). These repeated phenomena, deformation, welding and solid dispersion, harden the material and increase the fracture process. The predominantly spherical or equiaxial morphology of the initial (as received) powders, allows good powder packing, which results in the high initial apparent density values. The laminar morphology of the shorter-time mechanically milled powders brings poorer powder packing, and consequently decrease in the apparent density values. In a later stage of the process, the morphology of the particles will change again to an equiaxed one, with better powder packing and an increase in the apparent density. Confirmation regarding morphology and microstructure evolution described above gives morphology and microstructure analysis of the powder particles carried out in LM and SEM.

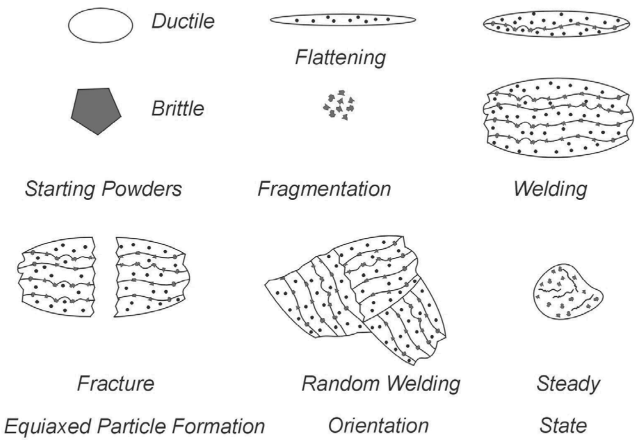


Fig. 7. Evolution of different stages of mechanical alloying of a ductile - brittle system according to [12]

Exemplary microstructure of initial powders can be observed in Fig. 8. Atomized aluminium powder are characterized by typical for cast alloy dendrite structure, when intermetallics powders are dens and without cracking. After short milling times, 2, 4 and 6 h, there is a predominance of deformed - flat particles (Fig. 9). Important is to notice that intermetallics particles undergo with more frequency fragmentation as well as in some extent plastic deformation. Crushed intermetallics reinforcement particles placed in the interfacial boundaries of the welded aluminium particles, result in the formation of a real composite particle but still with lamellar microstructure (Fig. 10).

After 8, 10 and 12 h of milling time, welded particles are observed with more frequency (Fig. 11), which indicates that welding is the predominant phenomenon at this stage of milling. Repetition of this basic mechanisms (deformation, welding and solid dispersion), contributes to the final equiaxed morphology at the steady state after 18 h of mechanical alloying (Figs. 12 and 13).

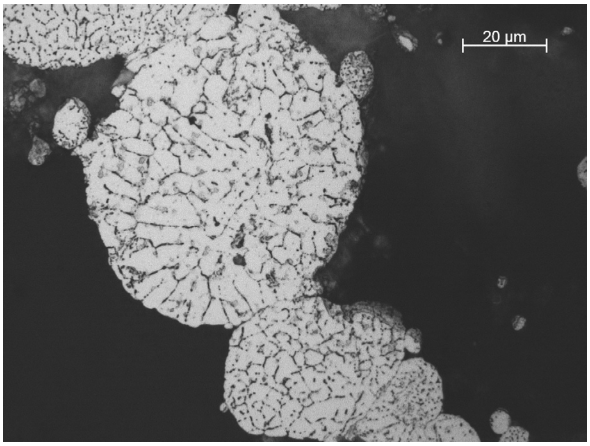


Fig. 8. Microstructure of EN AW-AlMg1SiCu as received powders, etched with Keller's, LM

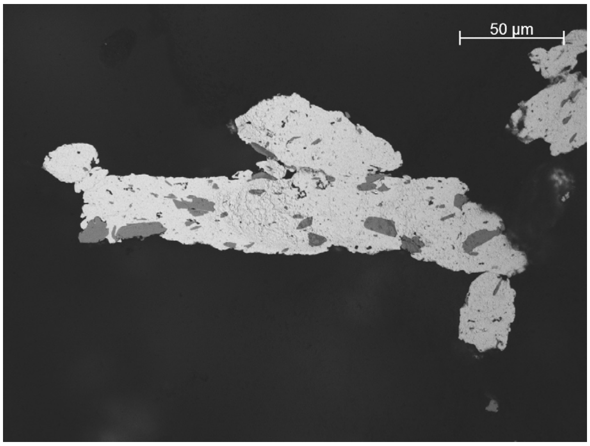


Fig. 9. Microstructure of EN AW-AlMg1SiCu powders with 5% of TiAl reinforcement after 6 h of mechanical alloying, LM

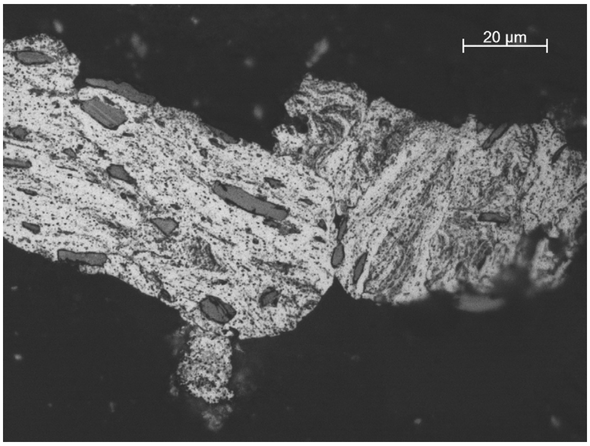


Fig. 10. Microstructure of EN AW-AlMg1SiCu powders with 10% of TiAl reinforcement after 10 h of mechanical alloying, etched with Keller's, LM

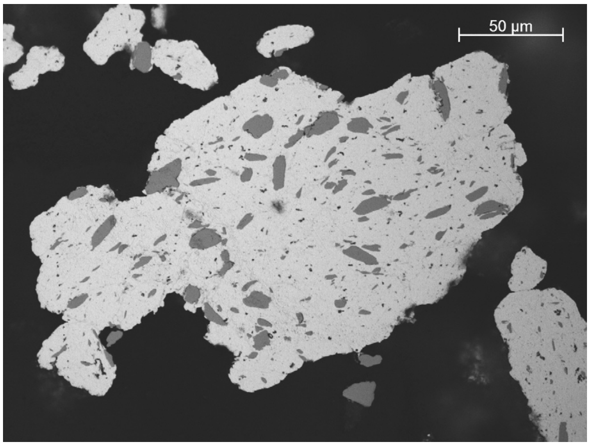


Fig. 11. Microstructure of EN AW-AlMg1SiCu powders with 15% of TiAl reinforcement after 14 h of mechanical alloying, LM

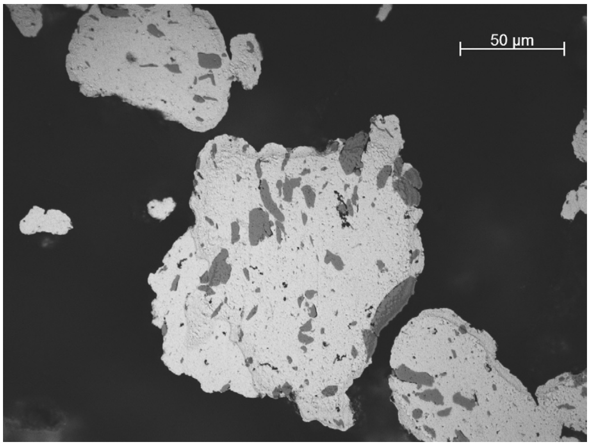


Fig. 12. Microstructure of EN AW-AlMg1SiCu powders with 15% of TiAl reinforcement after 18 h of mechanical alloying, LM

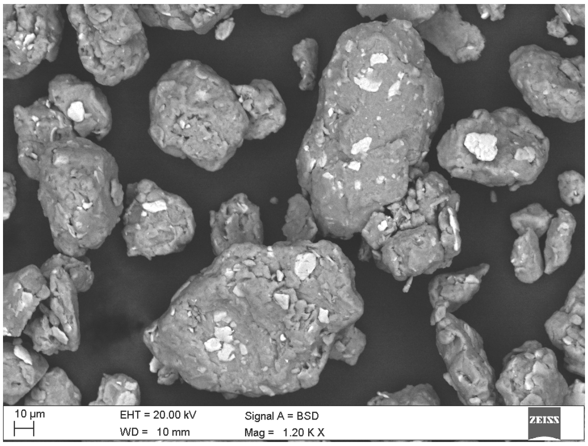
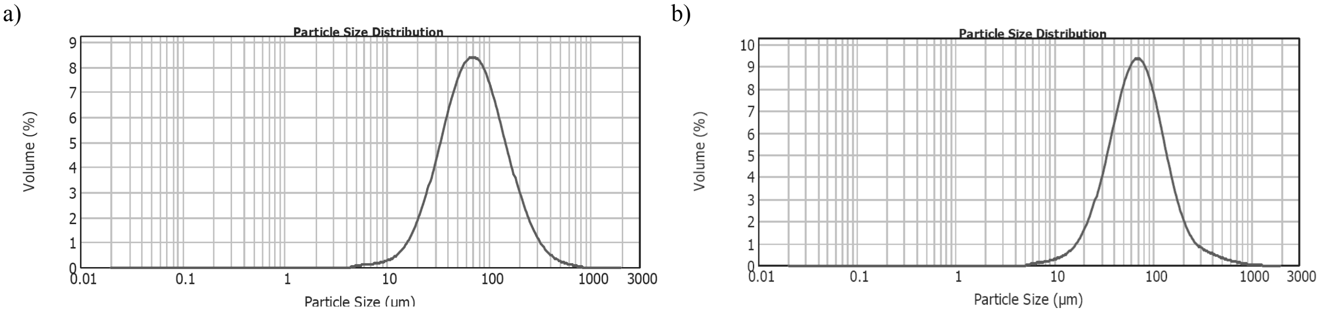


Fig. 13. Microstructure of EN AW-AlMg1SiCu powders with 20% of TiAl reinforcement after 18 h of mechanical alloying, SEM

Table 3.
Apparent density, volume size distribution and microhardness measurements results for investigated powders

Powder type/reinforcement type	Apparent density [g/cm^3]		Volume size distribution D50 [µm]		Microhardness HV	
	Ti$_3$Al	TiAl	Ti$_3$Al	TiAl	Ti$_3$Al	TiAl
EN AW-AlMg1SiCu	1.16		29.26		75.9	
EN AW-AlMg1SiCu +5%	1.18	1.17	80.12	70.45	181.4	178.5
EN AW-AlMg1SiCu +10%	1.20	1.20	72.35	71.89	188.7	182.7
EN AW-AlMg1SiCu +15%	1.22	1.22	72.45	70.15	198.8	196.3
EN AW-AlMg1SiCu +20%	1.24	1.25	70.40	69.10	205.5	203.4



D10=27.74 µm; D50=70.45 µm; D90=180.39 µm

D10=28.15 µm; D50=69.10 µm; D90=163.24 µm

Fig. 14. Exemplary result of volume particle size distributions for EN AW-AlMg1SiCu powders with a) 5% of TiAl and b) 20% of reinforcement after 18 h of mechanical alloying

In the processes applied widely in the PM manufacturing there are requirements for different particle size and flow characteristics. Difference in morphology is seen in the different flow and apparent density results. The apparent density is the mass per unit volume of a material including voids inherent in the as tested material. In the case of powder materials, the apparent density expresses the density without the application of pressure. The first point observed in the high-energy process is the dependence of milling time on apparent density. Fig. 15 shows exemplary dependence for the EN AW-AlMg1SiCu matrix composite reinforced with 15% of titanium aluminide intermetallic particles.

Zero hours of milling time means the apparent density of the as-supplied powder in the case of the unreinforced metal alloy. With short milling times, there is a continuous decrease in the apparent density with increasing milling time; it reaches a minimum value at 6 h of milling time, about one third of the initial value, and then starts to increase with increasing milling time. After longer milling, 16 to 18 h, depending on volume fraction of reinforcing particles the apparent density reaches a steady value, similar to that of the as-received powder. Driven by cold deformation welding process taking place in the mechanical is connected with layers consolidation. The presence of reinforcement particles between the particles during welding increases local deformation in the vicinity of the reinforcement particles. Figure 16 shows the EN AW-AlMg1SiCu alloy reinforced with 15% of Ti3Al. Reinforcement particles because of higher hardness when trapped in the interfacial boundaries, creates high deformation in the area surrounding reinforcement particle. An increase in the local deformation improves the particle welding process but also by the deformation hardening, leads to an improvement of the fracture process. The influence of the presence of reinforcement in the matrix during the mechanical milling is not that visible as in the case of ceramic reinforcements. However exemplary results of particle size distribution (Fig. 14) indicates that presence of reinforcing particles leads to constrained of the distribution curve.

Influence of intermetallics particles fraction (in the investigated range) on the steady state appearance is quiet small. This effect is greater - shorter milling time needed, with a higher concentration of Ti$_3$Al or TiAl (20%) reinforcing materials.

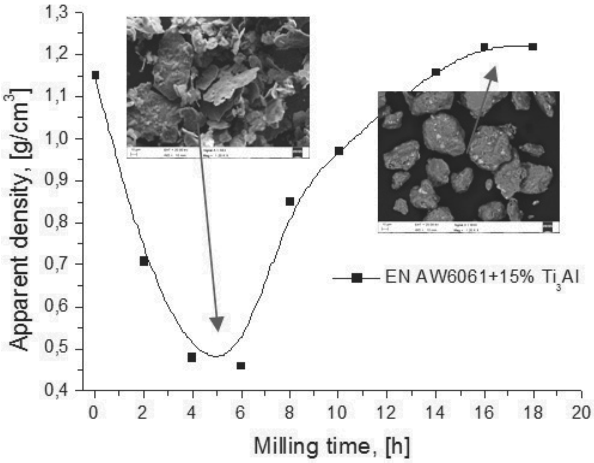


Fig. 15. Apparent density changes versus time of mechanical alloying for EN AW-AlMg1Si powders with 15% of Ti$_3$Al reinforcement

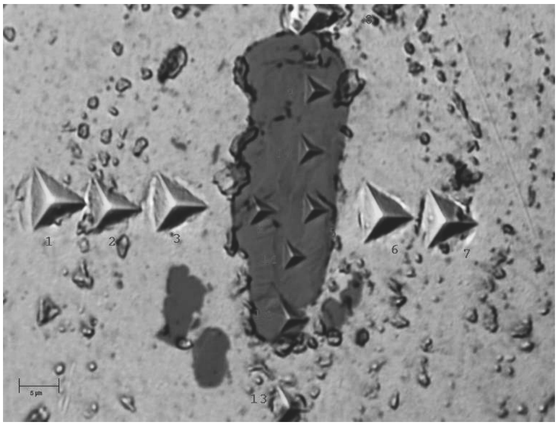


Fig. 16. Changes of the indentations around reinforcing particles generated by deformation hardening.

Table 3 presents results of apparent density measurements and volume size distribution D50 considered to be the average particle diameter by mass as well as microhardness for investigated powders at steady state, after 18 h of mechanical alloying with different volume fraction of reinforcing particles powders and for initial aluminium alloy powder. Results of apparent density measurements indicates better packing efficiency of composite powders in comparison with unreinforced powders, however incorporation of second phase with higher density must not be forgotten. On the other hand recuperation of flowability and their increase for higher percentage of intermetallics reinforcing particles indicates that composite powders have better particles size distribution achieved by removal of fine size fraction and irregular in shape particles typical for air atomized powders. The particle size distribution of investigated composite milled for 18 h shows a symmetric distribution, typical for the final stage of mechanical alloying process. The final particle size is, more than twice of that of as-received powder. The narrowing of distribution is because of welding occurs especially on the smaller particles. It is well known that mechanical alloying promote a high degree of deformation, reduce the grain size to nanometer level and produce an extremely fine distribution of oxides and others reinforcing materials as well constituents of raw materials. This hardening mechanism is mainly responsible for hardness increase in composite powders. Microstructure refinement from dendritic of atomized powder resulting from the fast cooling imposed by atomization process to fine distribution of reinforcing particles and oxide layers, as well as high density of dislocations because of strong plastic deformation leads to hardness increase. Mechanically milled powders are almost three times harder than the as-received powder. Applying mechanical alloying route of composite powders production, makes it possible to obtain diminution of reinforcing particles size as well as homogenous reinforcement particles distribution. As was expected mechanical milling process has improved the reinforcement distributions throughout the whole particle. Observed changes in the microstructure, influence on the mechanical properties of composite particles obtained.

The results of XRD phase analysis are presented on Fig. 17, depending of reinforcement contents presence of peaks coming from Ti_3Al and TiAl phases on the Al phase is more and more visible. Broadening of registered peaks indicates refinement of the microstructure in the mechanical alloying processes.

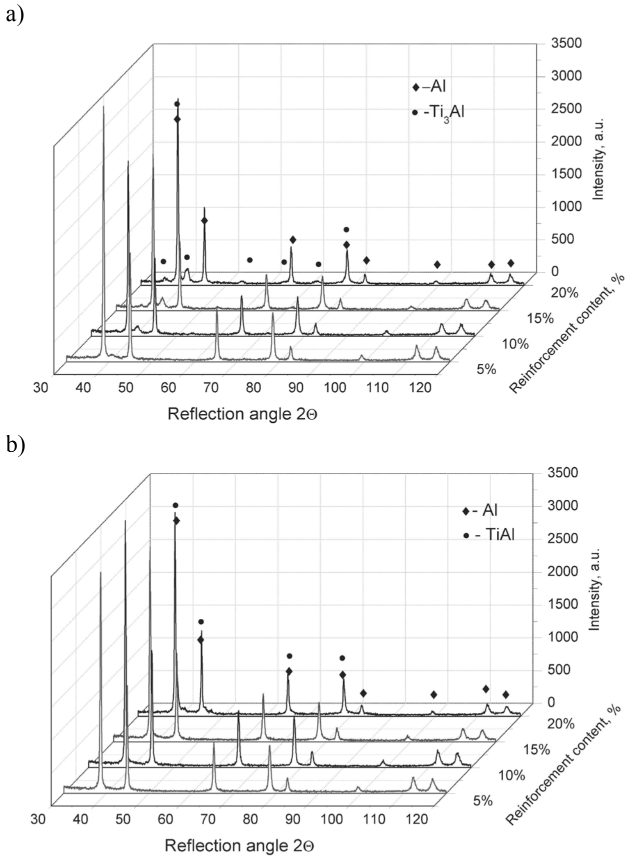


Fig. 17. XRD patterns for composites powders based on EN AW-AlMg1SiCu alloy with different content of a) Ti_3Al and TiAl reinforcing particles

3.2. Microstructure of extruded composites

A critical step in the processing of metal matrix composites reinforced with particles is the insertion of these particles into the metal matrix alloy. This greatly influences the strength of the composite since it is controlled by the metal-particle interfacial bond strength. The most important aspect of the microstructure is the distribution of the reinforcing particles, and this depends on the processing and fabrication routes involved. Extrusion can homogenize the structure to some extent, but minimizing reinforcement inhomogeneity during initial processing is important to achieve optimum properties [1-14].

Applying PM route of composite materials production, makes it possible to obtain homogenous reinforcement particles distribution without typical for conventional casting processes segregation. The microstructures of the as-extruded materials were examined by optical and electron microscopy. The samples were polished with diamond compound on a lapping wheel, than polished surfaces were etched lightly with Keller's reagent to reveal the outer contours of the intermetallics - matrix interface and precipitates in the matrix. As it was expected mechanical milling process has improved the reinforcement distributions throughout the whole composite powder particles. It can be seen that the mechanical milling has improved the reinforcement distributions throughout the whole particle, and has produced equiaxial morphology of composite powder particles. Additionally intermetallic particles has undergone plastic deformation as well as the fragmentation resulting in diminutions of reinforcing particles size. Examinations performed indicates that the composite materials obtained after extrusion were fully dense and exhibit very good cohesion at the interface between the aluminium matrix and the reinforcing particles. Typical and exemplary microstructures observed by light microscope LM and scanning electron microscope SEM in the case of the material containing 10 and 20% volume fraction of intermetallics particles after extrusion shows Figures 17 and 18. In the final products of composites materials, depending on the reinforcement size and shape, the density difference, type of matrix material - agglomeration can occur. Although the extrusion processes tends to minimize this problem reinforcement particles agglomeration is the most appointed cause of low performance of this class of materials. To avoid this problem mechanical milling can be used to improve the distribution of the reinforcement particles through the matrix. In the mechanically milled composites one can see very fine distribution of small reinforcement particles and absence of agglomerates.

Based on metallographic examination performed on light microscopy arrangement of the reinforcing particles in matrix material was determined. These observations indicate that the Ti_3Al and TiAl intermetallic particles are uniformly distributed in the aluminum alloy matrix, however, are characterized by differentiation in size and shape (Figs. 18, 19). Observations of metallographic specimens parallel to the direction of extrusion reveals that many of the reinforcing phase particles are elongated, and are arranged parallel towards to the direction of extrusion. This research also enables determine a high degree of uniformity of distribution of reinforcement particles and their fragmentation as a result of mechanical alloying process and previously described fragmentation. During stereological measurements made on composite materials cross sections examination of visible constituents on the surface plane were performed. Based on these studies, it was found that most of the reinforcing particles in the test material is not an equiaxed, which is particularly clearly visible in longitudinal cross sections. Reinforcing particles are oriented in the direction of the long axis of the extrusion process. These differences are characterized by rounding index which in most cases of analyzed particles is bigger than 1.5.

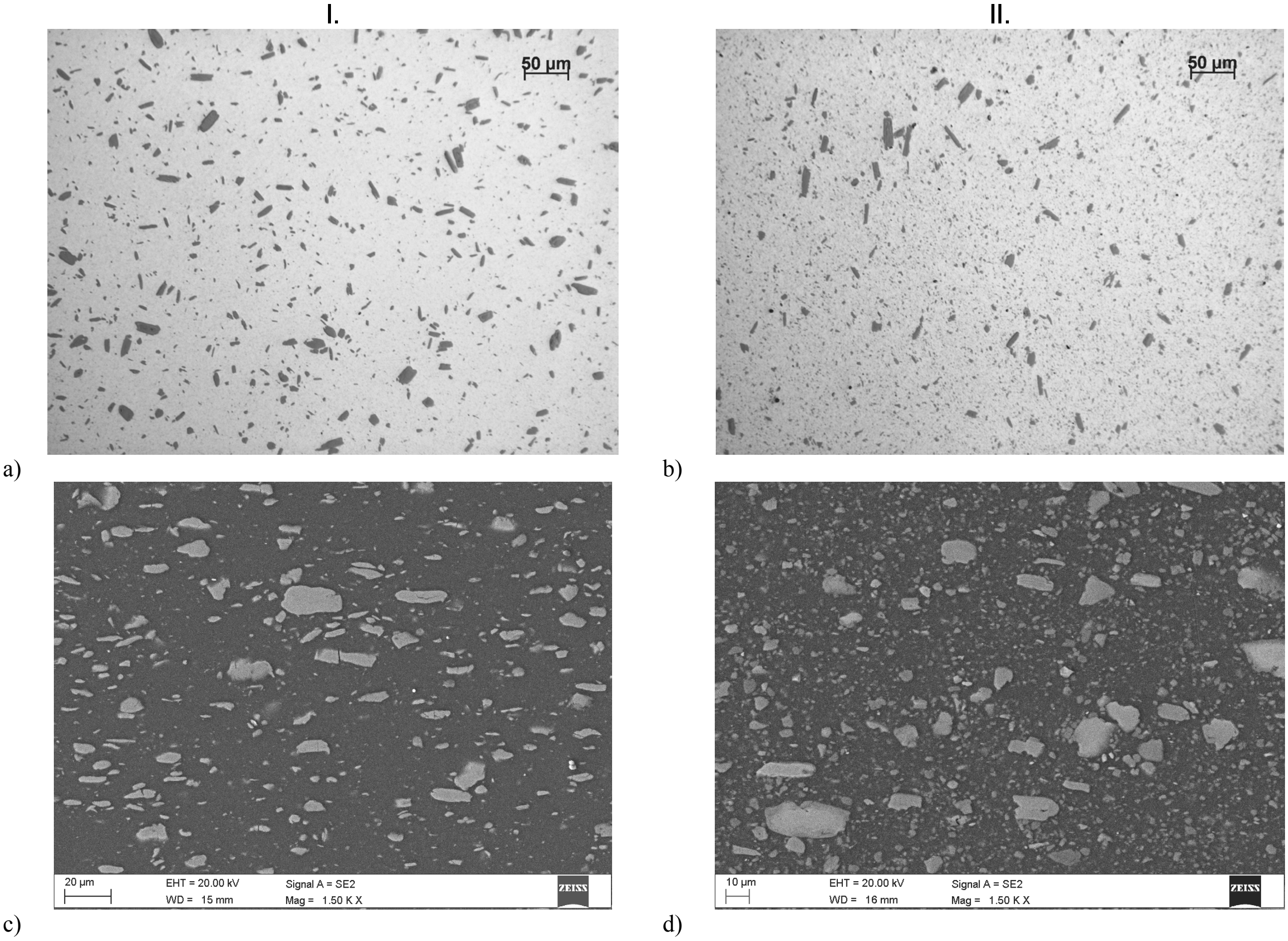


Fig. 18. Microstructure of EN AW-AlMg1SiCu alloy based composite materials reinforced with intermetallics particles: I 10%Ti_3Al, II 10%TiAl; a, b) transverse cross section of extruded bars, LM; c, d) longitudinal cross section of extruded bars images from scanning electron microscopy, SEM

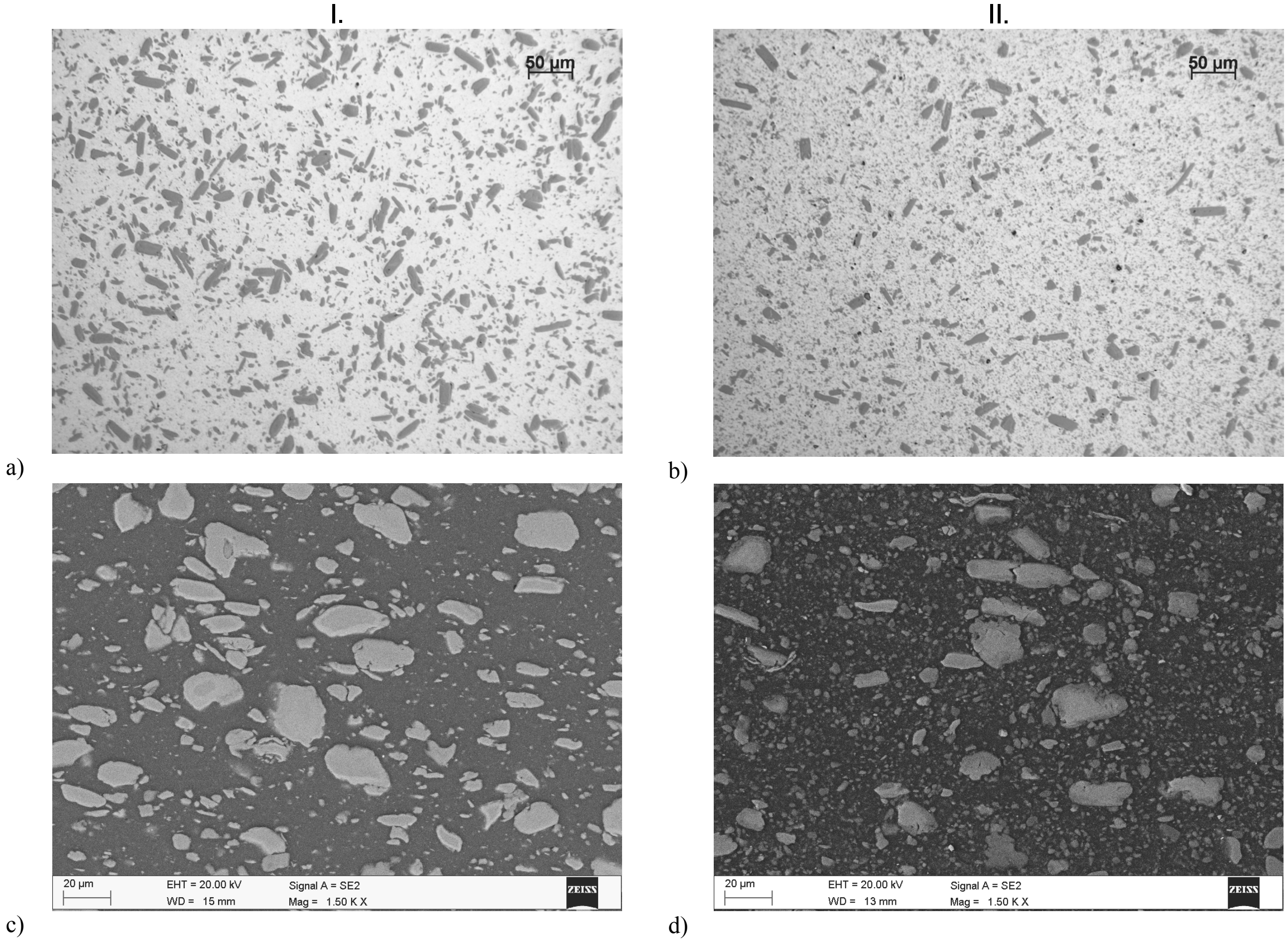


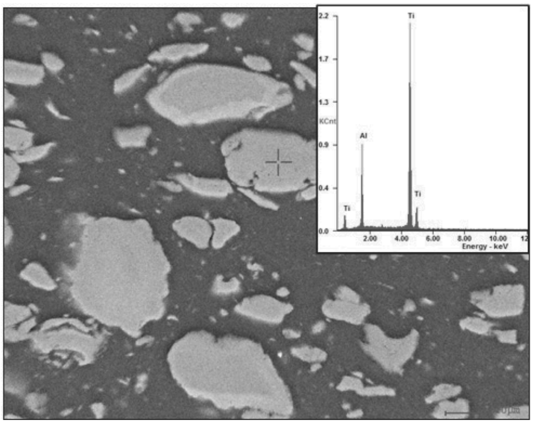
Fig. 19. Microstructure of EN AW-AlMg1SiCu alloy based composite materials reinforced with intermetallics particles: I 20%Ti_3Al, II 20%TiAl; a, b) transverse cross section of extruded bars, LM; c, d) longitudinal cross section of extruded bars images from scanning electron microscopy, SEM

Figures (18 b, c, and 19 b, c) shows the microstructure of composite materials observed in the scanning electron microscope. The composite materials prepared are free of porosity indicating a good consolidation achieved by compacting and extrusion process. Based on the observation made also on the scanning electron microscope one can see good bond between the intermetallic and matrix. No second phases produced by reaction at the matrix/particle interfaces after the extrusion process were detected in these or higher magnification images. Bigger magnification allows to state that some of the intermetallics particles posses already developed internal cracks, most probably as the result of cracking initiated in the milling process.

In such application as composites, interfaces exert an important and sometimes controlling influence on performance. Bonding of two different materials and resulting interfaces must typically sustain mechanical forces without failure. An understanding of the properties of interfaces requires a knowledge of both the structure and chemistry of the interface. Chemical processes can occur that lead to a modification of the properties of the interfaces. The reactions there will lead to reaction product and or gradients in the chemical composition in one or both components adjacent to the interface. X-ray microanalysis (Figs. 20-22) performed on energy dispersive spectrometer EDS in the scanning electron microscope for materials without reinforcement and composite materials, confirms the presence of constituents making up individual phases and presence of precipitates constituents in areas relevant to the investigated alloy mainly precipitates of intermetallic phases containing Al, Cu, Mg, Si, Mn. Analysis performed on the reinforcing particles confirm their relevant chemical composition. These particles after mechanical alloying are characteristic of reduced dimension and homogeneous distribution. Figures 20 and 21 shows exemplary results of EDS x-ray analysis of chemical composition of reinforcing phases. As can be seen on the scanning electron images of the sample surface with the selected analysis area, selected for analysis particles were big enough to eliminate influence of the matrix material on the results represented by energy dispersion plot for the X-ray analysis area. Results of the quantitative analysis of the elements concentration are presented in attached tables. These observations made together with the analysis, did not allow to detect the formation of the transition zone - reactive interface between the matrix material and the reinforcing particles. Confirmation of this results are presented in the EDS line scanning across of selected particles. Figure 22 shows the measurements result of concentration changes of Ti and Al within analysis line.

Sharp changes of concentration in the boundary region exclude any interface reaction in this area for both Ti_3Al and TiAl reinforcing system. One can understand that resolution of EDS analysis in scanning electron microscope is around 0.5 μm than for very thin interface formation this technique will not bring good results.

a)

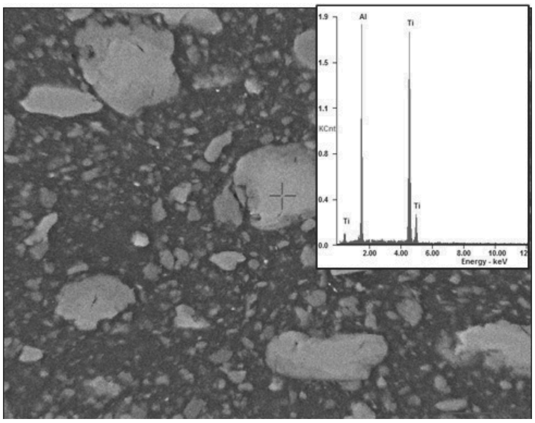


b)

Element	Wt%	At%
AlK	16.96	26.61
TiK	83.04	73.39
Matrix	Correction	ZAF

Fig. 20. EDS X-ray microanalysis of composite material EN AW-AlMg1SiCu + 20% Ti_3Al, a) image of the sample surface with the selected analysis area and energy dispersion plot for the X-ray analysis area, b) results of the quantitative analysis of the elements concentration

a)



b)

Element	Wt%	At%
AlK	31.11	44.49
TiK	68.89	55.51
Matrix	Correction	ZAF

Fig. 21. EDS X-ray microanalysis of composite material EN AW-AlMg1SiCu + 20% TiAl, a) image of the sample surface with the selected analysis area, b) energy dispersion plot for the X-ray analysis area, c) results of the quantitative analysis of the elements concentration

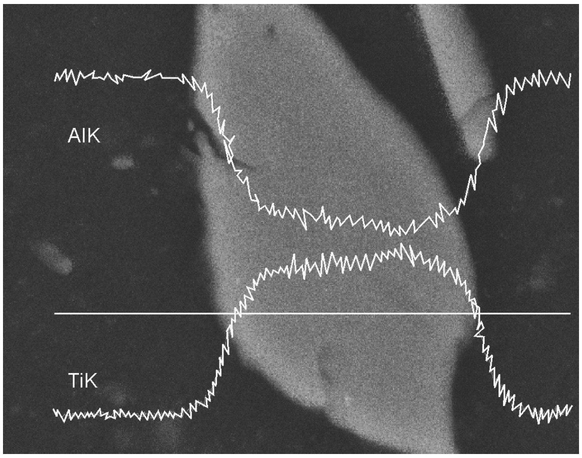


Fig. 22. EDS X-ray line scans for Al and Ti across Ti_3Al reinforcing particle in the EN AW-AlMg1SiCu alloy matrix

Observation of thin foils prepared from extruded bars by means of transmission electron microscopy (TEM) allowed revelling microstructure and constituents of examined aluminium alloy as well as composites. Selected results of this investigations are presented in Figs. 23 and 24 respectively of the EN AW-AlMg1SiCu alloy and composite materials. As expected predominant phase observed during thin foils examination for matrix alloy is the fine grained α-Al based solid solution with visible subgrain and dislocation microstructure. Fig. 23 b) shows dark field micrographs, selected area diffraction patterns and solution of the diffraction patterns. Fig. 23 d) shows EDS X-ray microanalysis energy dispersion plot for the analysed area. Observed on the presented figures subgrain microstructure exhibit subgrain size, which in most cases is bigger than 0.5 μm.

Similarly observation of thin foils for composites materials allowed revelling ultra fine grained α–Al based solid solution with visible subgrain and dislocation microstructure. Among the α-Al solid solution phase presence of Ti_3Al intermetallic phase can be easily detected. Fig. 24 a) and c) shows exemplary micrographs with fine and coarse Ti_3Al particles. Ultra fine microstructure is confirmed also by selected area diffraction (SAD) pattern typical for fine polycrystalline microstructure. Also this images confirm that intermetallic reinforcing phases are well bonded with aluminium matrix without interface zone. Fig. 24 d) shows EDS X-ray microanalysis energy dispersion plot for the analysed area, among the elements constituting matrix and precipitates, oxide presence is detected. That's indicate that beside of those findings presence of fine precipitates of Al_2O_3 oxide phase can be observed. Obviously very fine powders have relatively large surface areas and, thus, are highly reactive with oxygen, during air atomisation process of initial powders but also during composite powders production process with mechanical alloying. Figure 24 e) and f) shows microstructure with intermetallics precipitates and EFTEM Energy-filtered transmission electron microscopy, in which only electrons of particular kinetic energies are used to form the image in this case of Ti distributions.

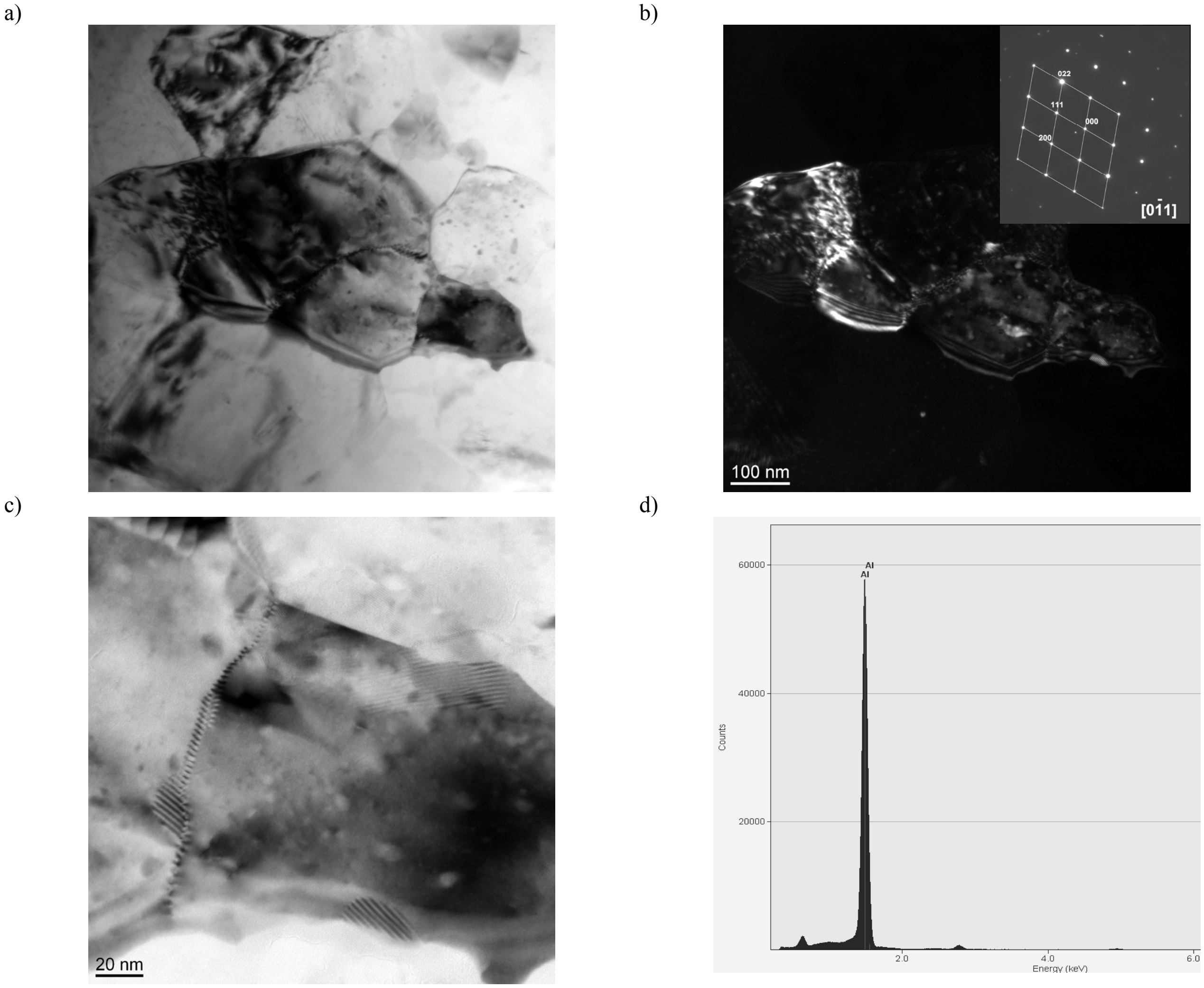


Fig. 23. TEM microstructure of thin foil for investigated EN AW-Al Mg1SiCu alloy, a, c) bright field micrographs showing subgrain structure of thin foil, b) dark field micrographs, selected area diffraction pattern and solution of the diffraction pattern, d) EDS X-ray microanalysis energy dispersion plot for the analysed area

Additionally to presented above results an investigations with Scanning transmission microscopy STEM mode combined with X-ray EDS elements map distribution were performed. Results of this experiment are presented on Fig. 25. One can see that in selected to the observation area precipitates rich in Ti characteristic for reinforcing particles as well as rich in Cu characteristic for matrix alloy can be observed.

3.3. The mechanical properties of the mechanically alloyed composites

In case of conventionally blended composites the addition of particulate reinforcements does not always allow to an increase in the material tensile strength mainly because of poor load transfer from the matrix to the reinforcement phase. Additionally problems such as poor bounding between matrix and reinforcements, reinforcement particles clustering, cracks in the reinforcement or their surface, can strongly deteriorate the composite strength. In that class of materials UTS and hardness confirm the poor improvement of these mechanical properties caused by the simple addition of reinforcement particles. There several factors like cracks very often present in particulate reinforcement and reinforcement particles agglomeration which bring about defects that works as stress concentrator deteriorating mechanical properties of the composite. Probability of such defect existence is higher with higher reinforcement concentration, and when UTS is greatly influenced by the presence of such defects the measured values a lower with higher particles amount. In the contrary, process of mechanical alloying promotes a high degree of deformation, reduces the grain size to nanometre level, generate more homogeneous distribution and refinement of the reinforcement particle, the oxide dispersion, all together should result in harder and stronger consolidated materials.

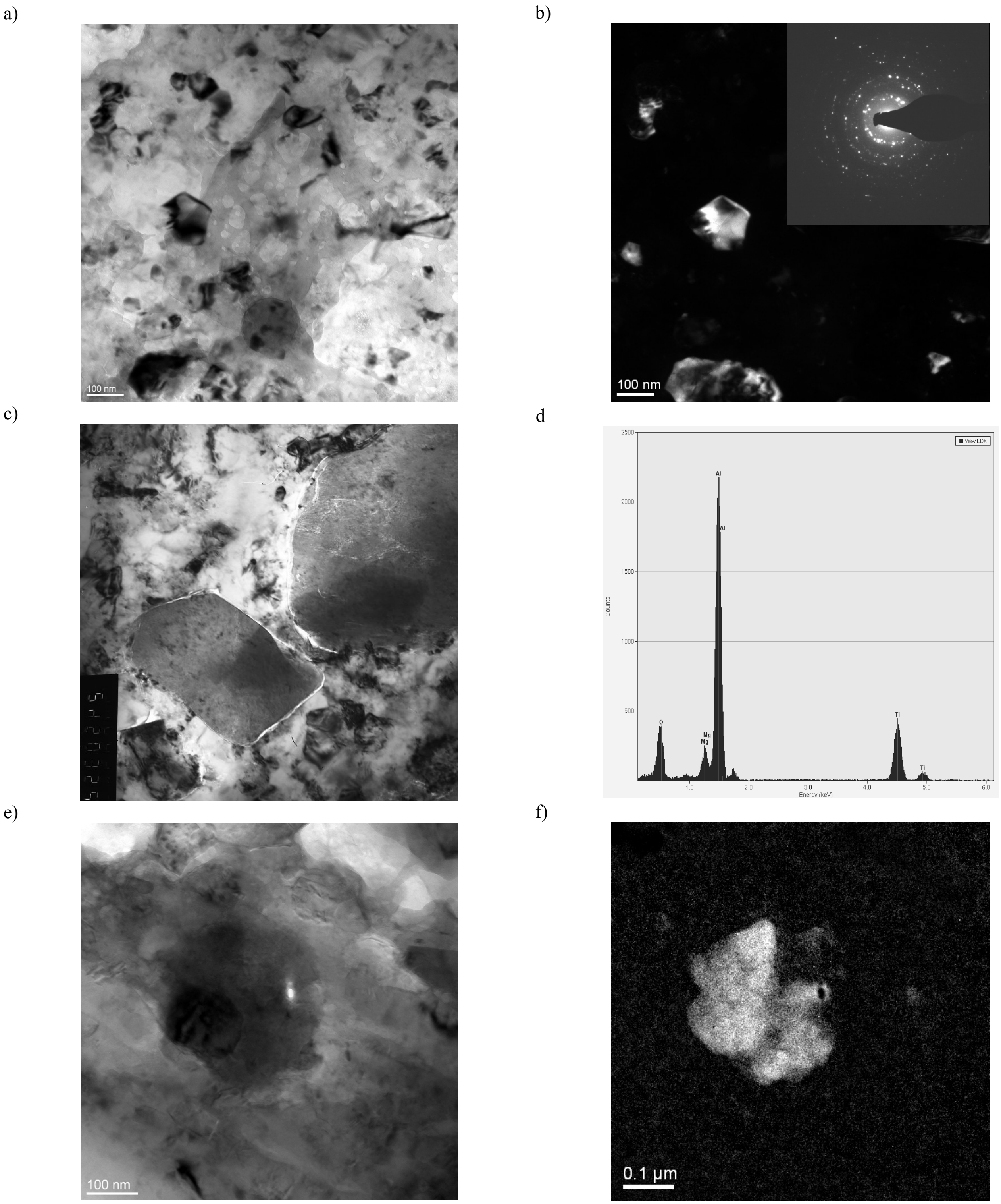
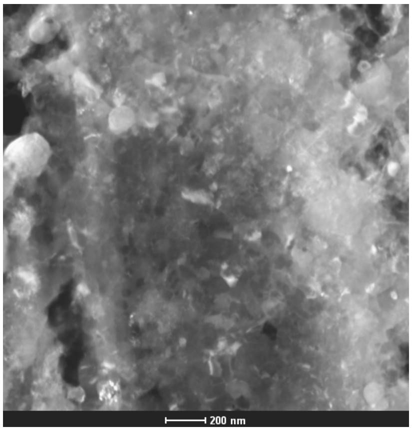


Fig. 24. TEM microstructure of thin foil for investigated EN AW-Al Mg1SiCu +10% Ti_3Al composite, a) bright field micrographs showing ultra fine subgrain structure, b) dark field micrographs with selected area diffraction pattern, c) bright field micrographs showing grains of intermetallics, d) EDS X-ray microanalysis energy dispersion plot for the analysed area, e) f) microstructure with intermetallics precipitates and EFTEM image of Ti distributions



a) Al_K b) Ti_K c) Fe_K

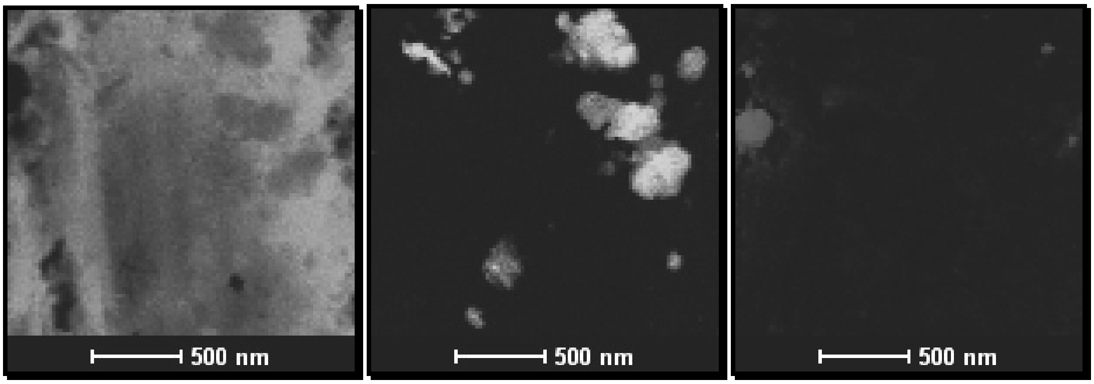


Fig. 25. STEM microstructure of thin foil for investigated EN AW-Al Mg1SiCu +10% Ti_3Al composite , a)-c) EDS X-ray microanalysis of element's distribution for Al, Ti, Fe

The effect of defect typical for blending of powders is less visible for hardness measurements. Hardness increases with milling time and with the reinforcement content. And it can be described as an effect of two main factors connected with deformation in the matrix - which is higher with the higher reinforcement content during mechanical alloying and during extrusion. The mechanical strength and hardness of the mechanically alloyed composites demonstrate the effectiveness of the process to optimize aluminium matrix intermetallics particulate-reinforced materials. Additionally the suitability of the extrusion process in air, which produced practically full density materials with no appreciable oxidation, was demonstrated.

Figure 26 shows the results of hardness measurements of extruded composites with increasing contents of reinforcing particles. One can see increase of the hardness due to increasing content of reinforcement particles and the mechanical milling process. Independent of reinforcing particles type with their increasing share hardness raising achieving for 20% around 150 HV1 when hardness of aluminium alloy matrix extruded without reinforcement particles achieve about 50 HV1. An increase of hardness is evident, it can be described as an effect connected with deformation in the matrix - which is higher with the higher reinforcement content as well as created during mechanical alloying and during extrusion. Decrease of hardness in comparison with microhardness measured for composite powders is connected with recrystallization processes ongoing at extrusion temperature. Hardness of composites prepared by extrusion of blended aluminium powders with intermetallics particles was around 55 HV1. It can be see that hardness of composite materials is only slightly higher after mixing process in comparison to not reinforced alloy and small increase of hardness value is observed when reinforcement contents increase. The same tendency can be observed for tensile properties and again blending process do not influence UTS value. Figures 27 and 28 presents the yield strength measurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles and ultimate tensile strength mesurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles. Again mechanical milling change almost twice ultimate tensile strength of investigated composites. Moreover until 20% of reinforcement particles contents UTS is growing indicating to good interfacial bonding of matrix and reinforcement particles. Figure 29 show ultimate compression strength mesurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles. Aplication of mechanicall alloing and reinforcing with intermetallics particle allows to growth of compression strength up to 770 MPa.

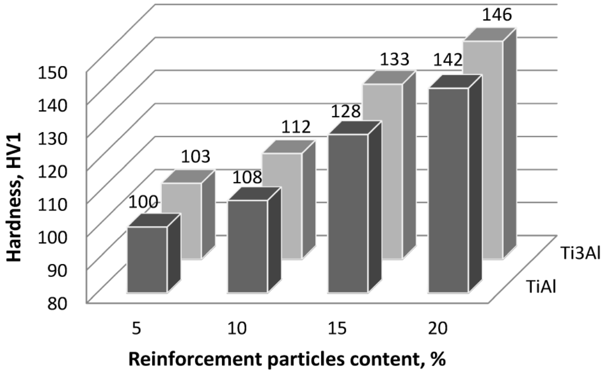


Fig. 26. Hardness of extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles

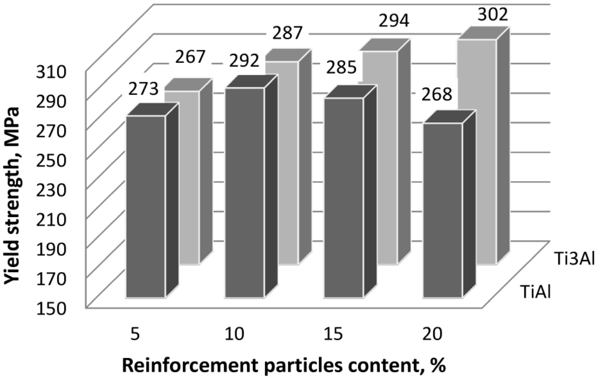


Fig. 27. Yield strength measurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles

The surfaces of tensile test specimens fracture were observed by scanning electron microscopy SEM (Fig. 30). The fracture behaviour in the aluminium matrix becomes ductile which is supported by a series of dimples, microstructural densification in extrusion process and interface bonding contribute to strength enhancement even though particle brittle fracture occurs more severely. The analysis of the fractographs indicate presence of two type of fracture - ductile in the matrix and brittle in the reinforcing particles, but again with good bonding between both. However extruded composites on microscopic scale indicate ductile fracture behaviour on the macroscopic scale composites exhibit limited ductility.

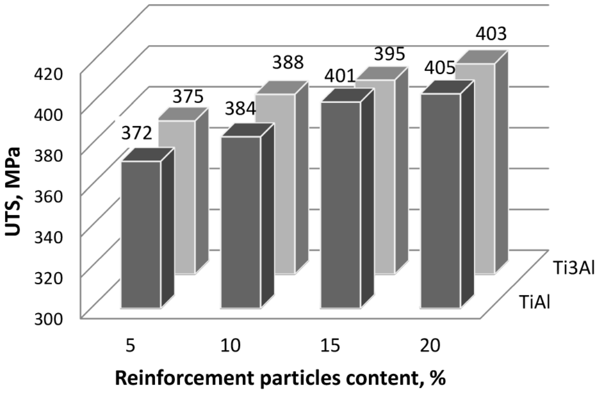


Fig. 28. Ultimate tensile strength measurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles

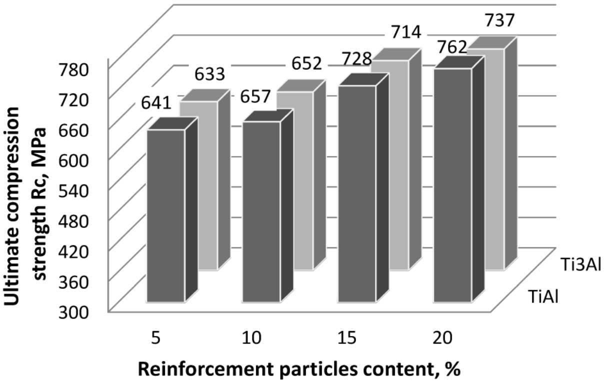


Fig. 29. Ultimate compression strength measurements results for extruded composites based on EN AW-AlMg1SiCu with different content of reinforcing particles

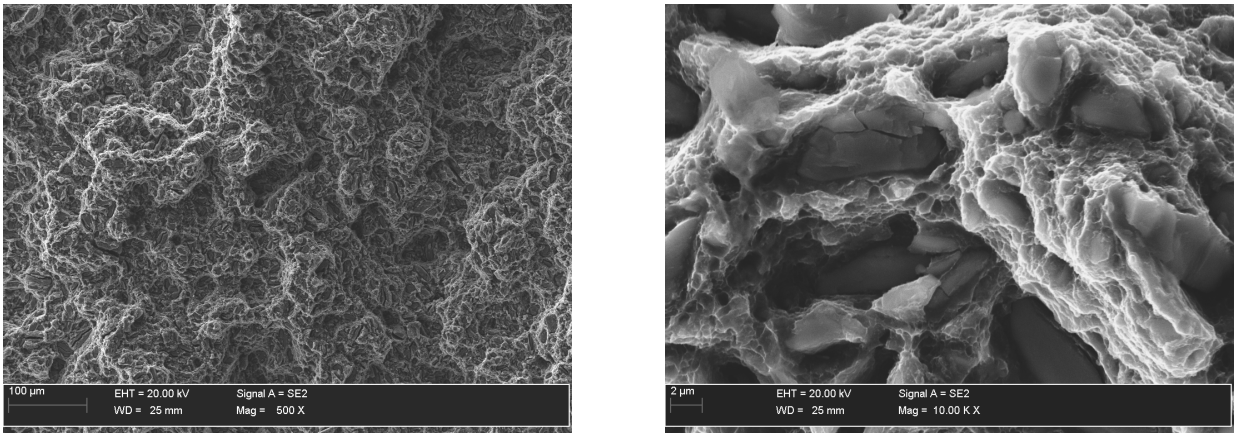


Fig. 30. Scanning electron micrographs of tensile test fracture of composites material with Ti_3Al reinforcing particles

3.4. Statistical analysis of the effect of reinforcing phase on the composite's properties

Preliminary analysis of test results obtained, set out visually in Figures 31-34 indicates that in most cases an increase in property is obtained with increasing share of reinforcing phase. Therefore, the section presents statistical analysis of the influence of reinforcement particles Ti_3Al and TiAl. As a tool was used method for evaluating significance of regression between tested properties and the participation of the reinforcing phase. It was assumed that it is appropriate to analyze the linear relationship. To determine the relations the multiple regression was applied, and to assess their variance significance F-Snedecor test was used. Carried out comprehensive analysis indicates an existence of relationship between tested values, especially in the case of EN AW-AlMg1SiCu alloy, since for most of the tested properties (except YS and the reinforcing phase TiAl) achieved significant linear correlation relationships (Table 4.).

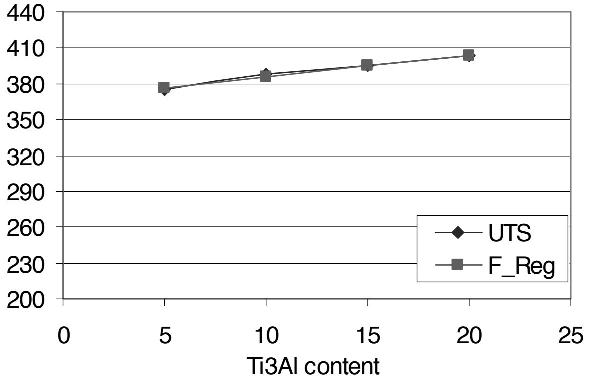


Fig. 31. Ultimate tensile strength changes depending on the reinforcing Ti_3Al particle contents for extruded composites based on EN AW-AlMg1SiCu and plot of calculated regresion function

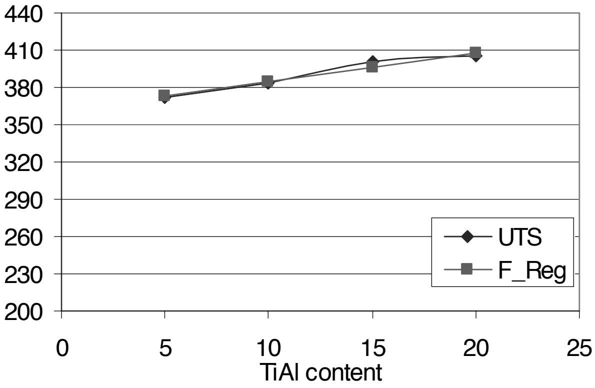


Fig. 32. Ultimate tensile strength changes depending on the reinforcing TiAl particle contents for extruded composites based on EN AW-AlMg1SiCu and plot of calculated regresion function

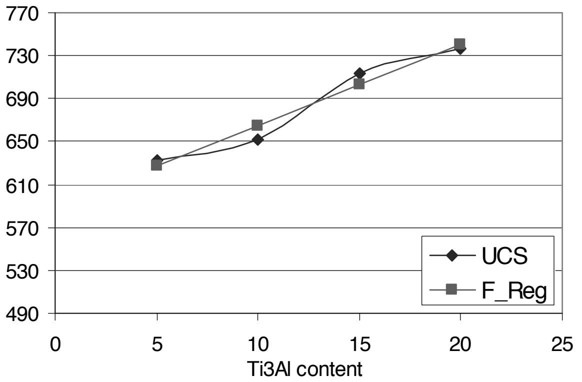


Fig. 33. Ultimate compression strength changes depending on the reinforcing Ti_3Al particle contents for extruded composites based on EN AW-AlMg1SiCu and plot of calculated regresion function

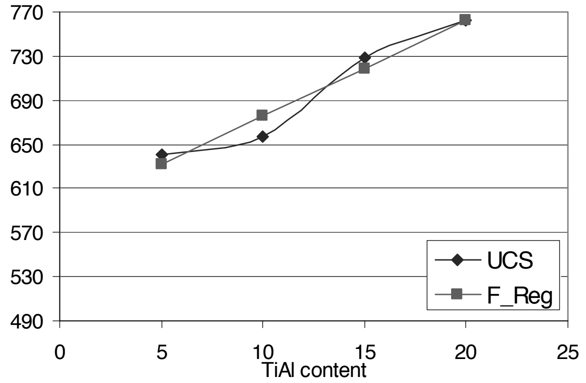


Fig. 34. Ultimate compression strength changes depending on the reinforcing T_3Al particle contents for extruded composites based on EN AW-AlMg1SiCu and plot of calculated regresion function

3.5. Reciprocating sliding wear resistance of composite materials

Reciprocal sliding wear behaviour of composites was chosen taking into account the fact that a number of industrial applications, such as engine cylinders, pistons etc., involve reciprocal sliding wear. Main objective of the investigations was to analyse the wear behaviour of a Ti_3Al and TiAl particle reinforced Al matrix composite sliding against a alumina ball by using a reciprocating sliding wear setup. All specimens were polished down with the grinding paper mesh 1200 and cleaned in ultrasonic cleaner. The weight of the specimens was measured before and after the wear test using an electronic balance. Light microscopy and scanning electron microscopy with an EDS X-ray spectroscopy were used to observe wear scars and material built up on the counterpart ball. Figures 35 and 36 shows the volume loss measurements as a function of the applied load. It can be seen that, with increase of applied load the volume loss of the composite increase. At a given sliding cycles number, the weight loss of the composite specimen is found to be two to six times smaller than that of the unreinforced aluminium alloy independent of the reinforcing particles type.

Table 4.
Results of statistical analysis of the influence of reinforcement particles Ti_3Al and TiAl on tested properties

Base material	Property examined	Reinforcement particle	Correlation coefficient R	Coefficient of determination R^2	Standard error	P-value	Type of dependence
EN AW-AlMg1SiCu	UTS	Ti_3Al	0.989657	0.97942	2.085665	0.010343	linear
		TiAl	0.976896	0.954326	4.012481	0.023104	linear
	YS0,2	Ti_3Al	0.965374	0.931947	4.785394	0.034626	linear
	UCS	Ti_3Al	0.976531	0.953613	13.04224	0.023469	linear
		TiAl	0.974261	0.949184	15.87766	0.025739	linear

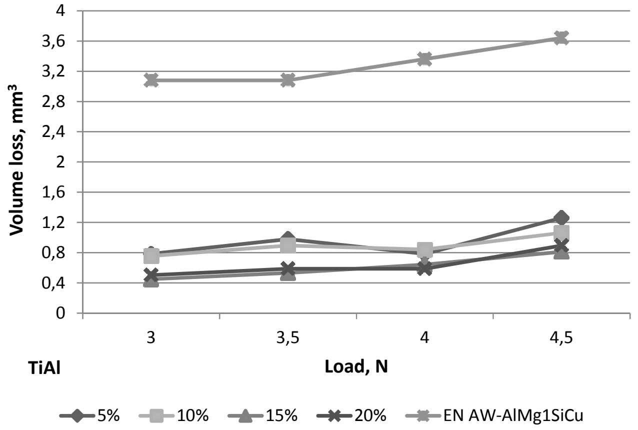


Fig. 35. Volume loss versus applied load for aluminium alloy and composites with different TiAl reinforcing particles content

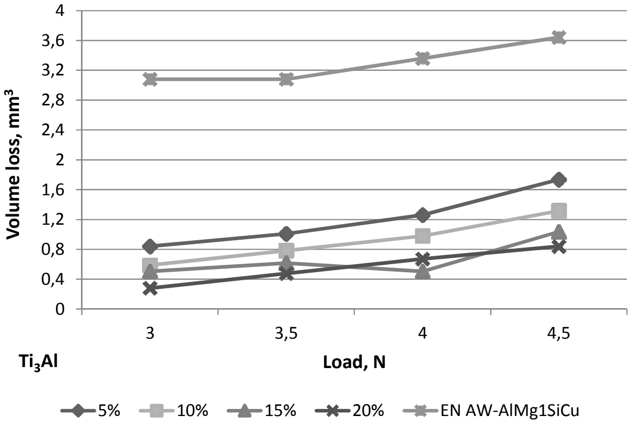


Fig. 36. Volume loss versus applied load for aluminium alloy and composites with different Ti_3Al reinforcing particles content

At the beginning of the test the initial contact between the ball and the composite is a point contact and the contact stress is maximum. The contact area gradually increases with an increase in the sliding cycles, because of the wear on the surface, than the contact stress drops. Figure 37 shows the friction coefficient changes during the reciprocating wear test. One can see evident change of measured coefficient values depending of investigated materials. Figure 37 a) illustrate EN AW-AlMg1SiCu alloy without reinforcing particles, b) composite materials based on the same aluminium alloy with 15% of Ti_3Al reinforcing particles content, prepared by blending of initial powders, and c) illustrate composite materials based on the EN AW-AlMg1SiCu with 15% of Ti_3Al reinforcing particles content, prepared by mechanical alloying.

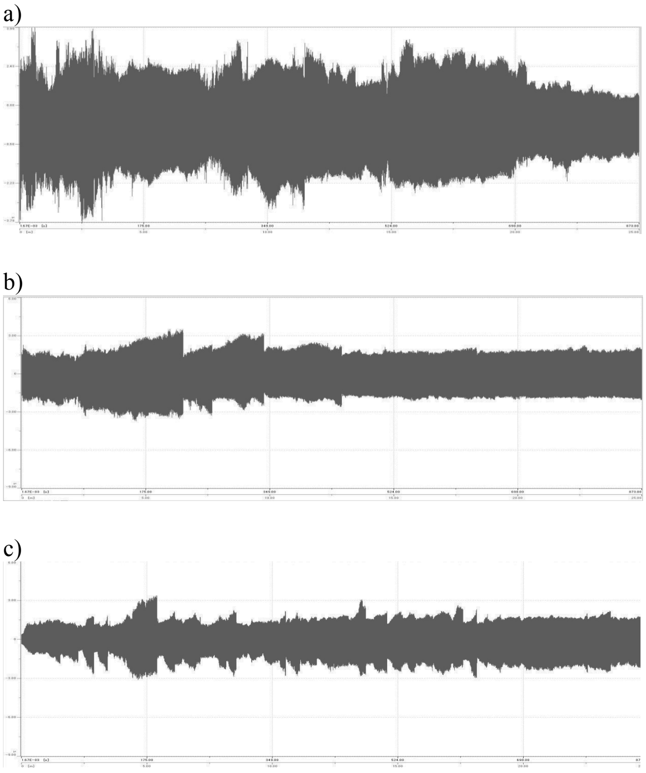
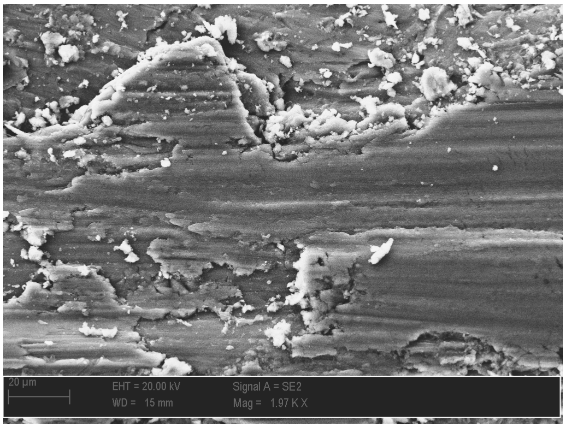


Fig. 37. Friction coefficient changes during the reciprocating wear test: a) EN AW-AlMg1SiCu alloy without reinforcing particles, b) composite materials based on the EN AW-AlMg1SiCu with 15% of Ti_3Al reinforcing particles content, prepared by initial powder blending, c) composite materials based on the EN AW-AlMg1SiCu with 15% of Ti_3Al reinforcing particles contents, prepared by mechanical alloying

Figure 38 shows typical morphology of wear tracks at a sliding distance of 25 m for unreinforced aluminium alloy. It can be seen that two different regions are present on the wear surface: cavities and smooth regions. In the smooth regions (Fig. 38 a), microgrooves and ploughing can be observed, which suggest that abrasive wear was predominant in these regions. The cavities (Fig. 38 b) resulted from local delamination in the wear surface, and the delamination phenomenon was one of the dominant mechanisms responsible for the wear failure of the aluminium alloy.

a)



b)

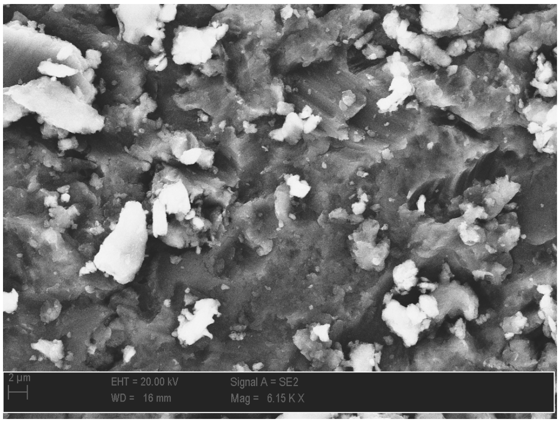


Fig. 38. Morphology of wear tracks for unreinforced aluminium alloy, SEM

Figure 39 shows the morphology of wear tracks for composite materials with Ti_3Al particles obtained by powder blending, the observed area consist of smooth regions and cavities however with very low extent. As shown in the high magnification micrograph rounded particles were present in the cavities. The rounded particles indicate fragmentation of the matrix and delamination from the subsurface, some of them have resulted from the fragmentation of Ti_3Al. Figure 40 shows the morphology of wear tracks for composite materials with Ti_3Al particles after mechanical alloying, the observed area consist mainly of smooth regions with visible on the wear track reinforcing particles slightly deformed. From the above results, it is observed that during dry sliding wear, the main mechanisms responsible for the wear loss of the composite are abrasive wear and adhesive delamination wear initiated in the mechanical mixing layer. With an increase in the sliding load, more matrix material is brought into contact with the sliding ball, and adhesion takes place between the matrix and the sliding ball. Exemplary build up of aluminium alloy on the sliding ball is shown on the Figure 41.

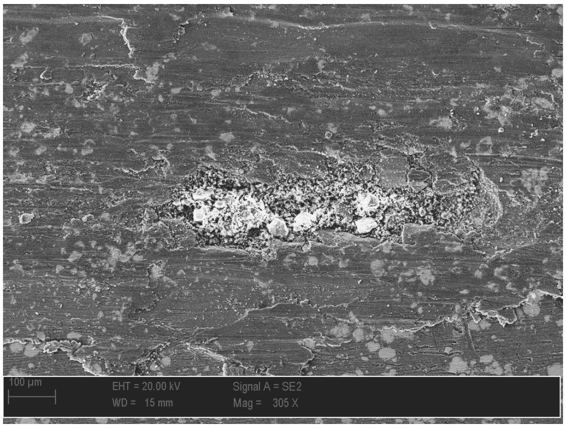


Fig. 39. Morphology of wear tracks composite materials based on the EN AW-AlMg1SiCu with 15% of Ti_3Al reinforcing particles content, prepared by initial powder blending, SEM

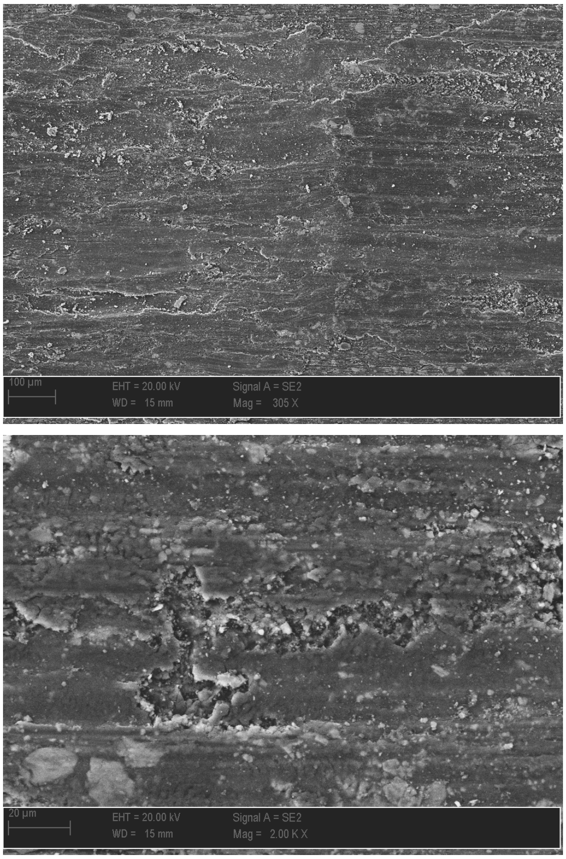


Fig. 40. Morphology of wear tracks composite materials based on the EN AW-AlMg1SiCu with 15% of Ti_3Al reinforcing particles content, prepared by mechanical alloying, SEM

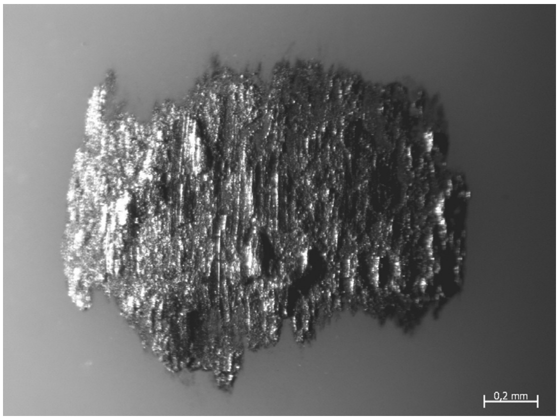


Fig. 41. Morphology of the aluminium alloy build up on the counterpart ball, LM

In the dry reciprocal sliding wear of the composite-ball system, the wear rate of the composite increased with the sliding load. The wear mechanism of the composite was predominantly adhesive. With further sliding, delamination and abrasive wear occurred in the composites as a result of the fracture and debonding of the particles than acting as third body particles. Examination with EDS spectrometer of worn surfaces shows distribution of elements within that surface as well as surface oxidation and oxide layer creation (Fig. 42).

From the presented above results it can be stated that during the sliding wear, the main mechanisms deciding about wear of the composites are adhesive delamination and abrasive wear initiated at the surface layers of the worn surface. In case of aluminium alloy delamination of adhesively bonded to counterpart aluminium tends to their fragmentation into small particles which can acts as abrasive third body of the system. Whereas in case of composites materials especially those received by powders blending in the initial period adhesive wear promote debonding of protruding particles which together with wear debris acts as abrasives between the contacts. Therefore, abrasive wear could be sever with the presence of the third body. In the dry reciprocal sliding wear of the mechanically alloyed composites the wear rate of the composites increase independent of the reinforcing particles type with increase of sliding load. The predominant wear mechanism is adhesion, with successive delamination and abrasive wear. EDS analysis indicates also existence of mild oxidation wear both for reinforced composite as well as aluminium alloy. Oxidational wear resulted in the formation and detachment of thin oxide layer giving the fine wear debris.

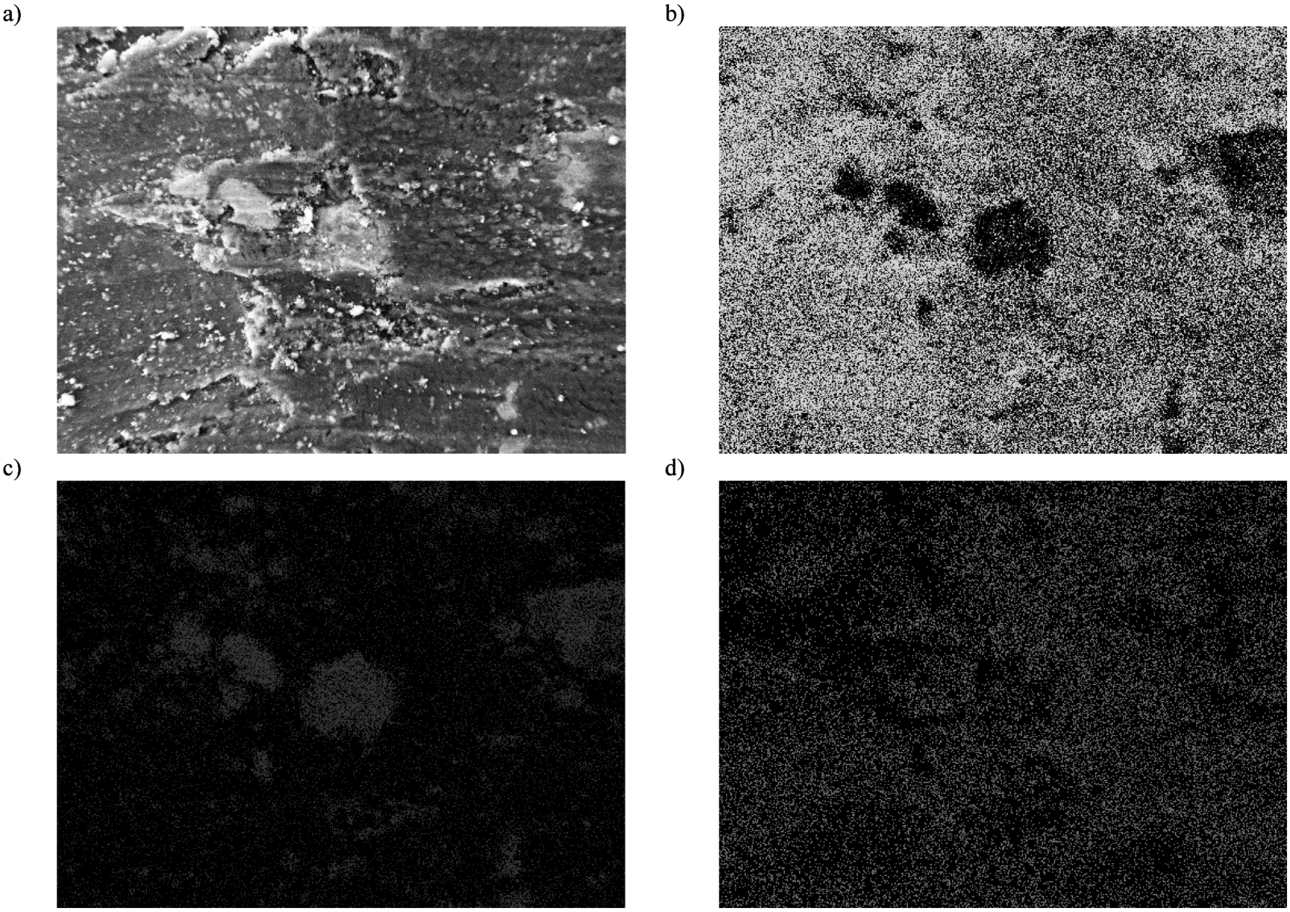


Fig. 42. SEM image of wear track (a) and EDS mapping of worn surface: b) Al distribution, c) Ti distribution, d) O distribution

In the dry reciprocal sliding wear of the composite-ball system, the wear rate of the composite increased with the sliding load. The wear mechanism of the composite was predominantly adhesive. With further sliding, delamination and abrasive wear occurred in the composites as a result of the fracture and debonding of the particles acting as third body particles. Examination with EDS spectrometer of worn surfaces shows distribution of elements within that surface as well as surface oxidation and oxide layer creation (Fig. 40).

From the presented above results it can be stated that during the sliding wear, the main mechanisms deciding about wear of the composites are adhesive delamination and abrasive wear initiated at the surface layers of the worn surface. In case of aluminium alloy delamination of adhesively bonded to counterpart aluminium tends to their fragmentation into small particles which can acts as abrasive third body of the system. Whereas in case of composites materials especially those received by powders blending in the initial period adhesive wear promote debonding of protruding particles which together with wear debris acts as abrasives between the contacts. Therefore, abrasive wear could be sever with the presence of the third body. In the dry reciprocal sliding wear of the mechanically alloyed composites the wear rate of the composites increase independent of the reinforcing particles type with increase of sliding load. The predominant wear mechanism is adhesion, with successive delamination and abrasive wear. EDS analysis indicates also existence of mild oxidation wear both for reinforced composite as well as aluminium alloy. Oxidational wear resulted in the formation and detachment of thin oxide layer giving the fine wear debris.

4. Conclusions

Based on the analysis of the researches carried out, the following final conclusions have been formulated:

1. The powder metallurgy techniques combined with mechanical alloying for composites powder production applied in this research enable the production of composites materials with homogenous distribution of reinforcement particles, which enables to combine the high mechanical properties with the high sliding wear resistance.
2. Analysis of test results enables the explanation of the impact of TiAl and Ti_3Al reinforcing particles on the microstructure changes and properties of the extruded composite materials, characterised with almost theoretical porosity and homogeneous and ultra fine-grained structure, in comparison to the composites produced through classical blending of powders and hot extrusion. Particular role is played by the mechanical alloying process used, thanks to which the refinement of reinforcing particles as well as subgrain formation activates composites strengthening mechanism. Observed microstructure changes influence on the mechanical properties. Microhardness measurements of composite powders indicates their increase in comparison to the initial state of aluminium alloy powders.
3. The strict control of the mechanical alloying process is necessary, because of the evolutionary changes of powders particles morphology leads to changes in packing ability and die filling during compacts production. The apparent density changes versus milling time can be used to control the composite powders production via mechanical alloying.
4. Mechanical alloying promote a high degree of deformation, reduce the grain size to nanometer level and produce an extremely fine distribution of oxides and others reinforcing materials as well constituents of raw materials. This hardening mechanism is mainly responsible for hardness increase in composite powders. Microstructure refinement from dendritic of atomized powder resulting from the fast cooling imposed by atomization process to fine distribution of reinforcing particles and oxide layers, as well as high density of dislocations because of strong plastic deformation leads to hardness increase. Mechanically milled powders are almost three times harder than the as-received powder.
5. The EN AW-AlMg1SiCu aluminium alloy powder with presence of intermetallics powder particles submitted to high energy mechanical alloying, are in good agreement with the model description proposed for ductile - brittle materials. The presence of reinforcements particles accelerates the mechanical alloying process in some extent. Mechanical alloying improves the distribution of the intermetallic reinforcing particles throughout the aluminium matrix, simultaneously reducing their size.
6. Based on metallographic examination, the extruded composites are characterized by a very homogeneous distribution of intermetalic particles and the absence of any reaction and with good cohesion at the matrix /particle interfaces. The mechanically milled and extruded composites show finer and better distribution of reinforcement particles what leads to better mechanical properties of obtained products. TEM examination of thin foils as well as selected areas diffraction pattern solution confirms complex microstructure of investigated materials. Preliminary analysis of this images confirm presence of α-Al solid solution matrix phase, TiAl, Ti_3Al intermetallic reinforcing phase and fine precipitates and Al_2O_3 oxide phases.
7. During stereological measurements made on composite materials cross sections examination of visible constituents on the surface plane were performed. Based on these studies, it was found that most of the reinforcing particles in the test material is not an equiaxed, which is particularly clearly visible in longitudinal cross sections. Reinforcing particles are oriented in the direction of the long axis of the extrusion process. These differences are characterized by rounding index which in most cases of analyzed particles is bigger than 1.5.
8. The composite materials prepared are free of porosity indicating a good consolidation achieved by compacting and extrusion process. Based on the observation made also on the scanning electron microscope one can see good bond between the intermetallic and matrix. No second phases produced by reaction at the matrix/particle interfaces after the extrusion process were detected in these or higher magnification images.
9. The finer microstructure increase mechanical properties of extruded composites materials. The higher reinforcement content results in higher particles dispersion hardening. Composites reinforced with 20% of reinforcement TiAl or Ti_3Al reach about 470 MPa UTS. On the contrary the addition of intermetallic reinforcement particles to the low energy mixed and extruded composites do not influence their tensile properties, slightly increasing their hardness.

10. The fracture behaviour in the aluminium matrix becomes ductile which is supported by a series of dimples, microstructural densification in extrusion process and interface bonding contribute to strength enhancement even though particle brittle fracture occurs more severely. The analysis of the fractographs indicate presence of two type of fracture - ductile in the matrix and brittle in the reinforcing particles, but again with good bonding between both. However extruded composites on microscopic scale indicate ductile fracture behaviour on the macroscopic scale composites exhibit limited ductility.
11. Statistical analysis of the effect of reinforcing phase on the composite's properties indicates an existence of relationship between tested values, since for most of the tested properties (except YS and the reinforcing phase TiAl) achieved significant linear correlation relationships.
12. In the dry reciprocal sliding wear of the composite-ball system, the wear rate of the composite increased with the sliding load. The wear mechanism of the composite was predominantly adhesive. With further sliding, delamination and abrasive wear occurred in the composites as a result of the fracture and debonding of the particles than acting as third body particles. It can be stated that during the sliding wear, the main mechanisms deciding about wear of the composites are adhesive delamination and abrasive wear initiated at the surface layers of the worn surface.

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