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P/M COMPOSITES OF Al-Si-Fe-Cu ALLOY WITH SiC PARTICLES HOT-EXTRUDED AFTER PRELIMINARY COMPACTION

The objective of the research work was to evaluate the possibility of forming high quality composites based on the Al17Si5Fe3Cu1.1Mg0.6Zr alloy, reinforced with silicon carbide particles, by means of preliminary hot compaction and hot extrusion processes. The mixtures were prepared with a matrix alloy powder and reinforcing phase, with a volume fraction of SiC particles maintained at 5, 10 and 15%. The feedstock to be extruded was prepared by preliminary hot compaction of the powders and composite mixtures. Subsequently, the semi-finished products were subjected to forward extrusion in isothermal conditions. For the obtained materials, both after compaction and after extrusion, their relative densities and hardness were determined. For the extruded materials, their compressive strength was determined, stress-strain curves were constructed, and their microstructure was analysed as well. The obtained materials showed high relative density and mechanical properties depending on the amount of deformation and volume fraction of the reinforcing phase. As a result of introducing SiC particles into the matrix or increasing their volume fraction, an increase of hardness was observed. Hot extrusion resulted in decreased hardness as compared to the material after preliminary compaction. With a volume fraction of SiC maintained not higher than 10%, the compressive strength of the extruded materials increased, while at 15% the average compressive strength was lower when compared to the matrix alloy. Based on room temperature stress-strain curves it was found that introducing particles into the matrix or increasing their volume fraction caused a decrease of the strain level at which failure of the specimen occurred. Increasing the volume fraction of SiC particles up to 10% resulted in a strengthening of the matrix, while in the case of a composite containing 15% of particles, a lowering of strength was observed. Specimens subjected to compression at a temperature of 200°C were deformed plastically, and the stress value at which the deformation occurred increased with an increasing volume fraction of the reinforcing phase. The microstructure of the matrix obtained as a result of extrusion realized with the assumed parameters was fine-grained. In the case of composites, the observations revealed a uniform distribution of SiC particles in the matrix. Based on the obtained results of the investigations, it was concluded that in the case of products formed with the assumed parameters, the introduction of SiC particles into the matrix, with a volume fraction maintained not higher than 10%, has a favourable effect, while at 15% a decrease of the analysed material properties at room temperature and their increase at an elevated temperature is observed. The obtained results of investigations allow us to conclude that the decision to introduce SiC particles into the Al17Si5Fe3Cu1.1Mg0.6Zr alloy matrix as a reinforcing phase, as well as of their volume fraction, should depend on the foreseen working conditions of the element made of this material.

Keywords: powder metallurgy, forming, aluminium alloy powder, SiC particles, composites, compaction, hot extrusion, mechanical properties, microstructure

KOMPOZYTY STOP Al-Si-Fe-Cu - CZĄSTKI SiC FORMOWANE NA GORĄCO PRZEZ WYCISKANIE WSTĘPNIE ZAGĘSZCZONYCH MIESZANIN

Celem badań była ocena możliwości formowania wysokiej jakości tworzyw na osnowie stopu Al17Si5Fe3Cu1.1Mg0.6Zr umocnionych cząstkami węgla krzemu, przy wykorzystaniu procesów wstępnego zagęszczania na gorąco i wyciskania na gorąco. Przygotowano mieszaniny proszków stopu osnowy i fazy umacniającej, przy udziałach objętościowych wynoszących odpowiednio 5, 10 i 15% cząstek węgla krzemu. Proces mieszania składników prowadzono w temperaturze pokojowej, przy prędkości obrotowej mieszalnika $0,9 \text{ s}^{-1}$ i w czasie 60 minut. Wsad do wyciskania przygotowano przez wstępne zagęszczenie na gorąco proszków i mieszanin kompozytowych. Proces realizowano przy temperaturze 485°C oraz nacisku 150 MPa, który przetrzymywano przez 5 minut. Tak przygotowane półwyroby były wyciskane współbieżnie z prędkością $0,15 \text{ mm}\cdot\text{s}^{-1}$ i przy współczynniku wyciskania 4,1. Podczas wyciskania stopu osnowy i kompozytów rejestrowano przebieg zmiany siły w funkcji przemieszczenia stempla. Dla półwyrobów po zagęszczaniu i dla wyciskanych tworzyw wyznaczono ich gęstość względną i twardość. Dla materiałów w stanie po wyciskaniu określono wytrzymałość na ściskanie i opracowano krzywe naprężenie - odkształcenie. Równomierność rozłożenia węgla krzemu w osnowie kompozytów oceniano przez obserwacje ich mikrostruktury. W efekcie zastosowania proponowanej technologii formowania otrzymano tworzywa o wysokiej gęstości względnej oraz o właściwościach mechanicznych zależnych od stopnia przetworzenia materiału i udziału cząstek fazy umacniającej w osnowie. Wzrost twardości próbek obserwowano w wyniku wprowadzenia do osnowy cząstek węgla krzemu lub zwiększenia ich udziału objętościowego, proces wyciskania na gorąco przy przyjętych parametrach powodował spadek twardości wsadu. W wyniku wprowadzenia do osnowy 5 i 10% cząstek węgla krzemu stwierdzono zwiększenie wytrzymałości na ściskanie tworzyw wyciskanych, wyznaczona dla kompozytu o zawartości 15% średnia wartość R_c była niższa niż dla materiału osnowy. Na podstawie wyznaczonych w temperaturze pokojowej krzywych naprężenie - odkształcenie stwierdzono, iż wprowadzenie do osnowy cząstek lub zwiększanie ich udziału objętościowego w osnowie powodowało spadek wielkości odkształcenia, przy którym następowało zniszczenie próbek. Podwyższenie zawartości cząstek węgla krzemu w osnowie do

10% prowadziło do umocnienia osnowy, dla kompozytu o zawartości 15% cząstek obserwowano spadek wytrzymałości. Próbki ściskane przy temperaturze 200°C odkształcały się plastycznie, wartość naprężenia, przy którym następowało odkształcenie materiału, rosła wskutek wprowadzenia do osnowy cząstek fazy umacniającej i zwiększania jej udziału w osnowie. Otrzymana w wyniku wyciskania przy przyjętych parametrach mikrostruktura osnowy była drobnoziarnista, w przypadku kompozytów obserwacje wykazały równomiernie rozlokowanie cząstek węgla krzemu w osnowie. Na podstawie otrzymanych wyników badań stwierdzono, że w przypadku wyrobów formowanych przy przyjętych parametrach wprowadzenie do osnowy cząstek węgla krzemu do ich udziału objętościowego wynoszącego 10% jest korzystne, zastosowanie 15% fazy umacniającej powoduje spadek badanych właściwości materiału w temperaturze pokojowej oraz ich polepszenie w podwyższonej temperaturze. Dlatego decyzja o zastosowaniu cząstek węgla krzemu do umocnienia wykonanej ze stopu Al17Si5Fe3Cu1.1Mg0.6Zr osnowy oraz o ich udziale objętościowym w osnowie powinna zależeć od przewidywanych warunków pracy wykonanego z tego materiału elementu.

Słowa kluczowe: metalurgia proszków, przeróbka plastyczna, proszek stopu aluminium, cząstki węgla krzemu, kompozyty, zagęszczanie, wyciskanie na gorąco, właściwości mechaniczne, mikrostruktura

INTRODUCTION

Aluminium-silicon type alloys show a number of favourable properties qualifying these materials for responsible structural components. These properties include low specific gravity, high strength, good heat conduction and abrasion resistance. It is possible to improve and modify these properties according to the needs, by introducing additional elements into the chemical composition of the alloy. Iron, copper and magnesium retard silicon grain growth [1]. Additionally, iron causes hardening of an alloy. Copper and magnesium additions enable precipitation hardening of aluminium-silicon alloys, resulting from the formation of CuAl_2 or Mg_2Si precipitates. However, the addition of copper results in lowering the corrosion resistance, while the addition of about 1% nickel improves it. Finally, a material can be obtained showing a coefficient of thermal expansion, microstructure and mechanical properties being stable up to a temperature of about 300°C, which is important in the case of working at elevated or variable temperatures. Additional stabilization of the properties in such conditions can be achieved by introducing reinforcing phase particles into the alloy matrix, provided that no reactions proceed between them [2]. In the case of the discussed group of materials, ceramic phase particles such as aluminium oxide or silicon carbide can be used. These materials can be produced with the application of powder metallurgy techniques. This technology, when compared to casting methods, requires significantly lower temperatures and allows one to obtain a more fine-grained microstructure of the matrix. However, its application involves the necessity to overcome some technological problems. Due to the low ductility of Al-Si-Fe-Cu alloys, the preparation of a feedstock to be formed realized by the compaction of mixtures at room temperature often does not lead to obtaining a stable P/M compact, regardless of the unit pressure level. This especially concerns powders of small and spherical particles, which decreases their formability significantly. The problem can be solved by the application of the hot forming of mixtures [3]. The pilot testing of selected properties of composites based on an Al17Si5Fe3Cu1.1Mg0.6Zr alloy matrix, reinforced

with 10% SiC particles, obtained by hot compaction and extrusion, is presented in [4]. Since, in the case of the application of powder metallurgy techniques, one of the conditions of obtaining high-quality composites is uniform distribution of the reinforcing phase particles in the matrix, it is also important to develop effective methods making it possible to quickly select the proper mixing parameters. The methods based on knowledge engineering are fairly suitable for this purpose, which was shown in [5, 6]. Finally, the selection of suitable forming technology and proper technological parameters leads to obtaining composites showing a favourable microstructure and high mechanical properties. The above mentioned features resulted in the application of these materials as structural components in such fields as the automotive industry, aviation, sports equipment production and medicine.

EXPERIMENT

Objective and scope of research

The objective of the investigations was the assessment of the possibility of forming high-quality materials based on an Al17Si5Fe3Cu1.1Mg0.6Zr alloy matrix, reinforced with silicon carbide particles, with the application of preliminary hot compaction of a powder or a mixture and hot extrusion processes. The scope of research included the manufacturing of matrix alloy materials and composites with various volume fractions of SiC particles, as well as the determination of their selected properties.

Material under investigation and sample preparation

As a matrix material, an atomized Al17Si5Fe3Cu1.1Mg0.6Zr alloy powder was applied, while silicon carbide SiC_{600} was used as a reinforcing phase. The particle size of both components did not exceed 40 μm .

The forming process was realized with parameters which were determined in previous experiments as being favourable for a matrix material. Selected results

of those investigations were presented in [7]. The parameters of mixing of composite components were estimated with application of the fuzzy logic method. Examples of such a type of applications were published in [6].

The mixtures of a matrix alloy powder and reinforcing phase were prepared, with volume fractions of silicon carbide particles amounting to 5, 10 and 15%. The mixing process was realized using a two-cone mixer, at room temperature, with a 0.9 s^{-1} speed of rotation, during 60 minutes. Semi-finished products destined for extrusion were produced by hot compaction of the aluminium alloy powder and mixtures in a closed die. Compaction was realized at 485°C , applying 150 MPa unit pressure and a duration of 5 minutes. The semi-finished products were then subjected to forward extrusion, applying a tool speed of $0.15 \text{ mm}\cdot\text{s}^{-1}$ and an extrusion ratio amounting to 4.1. The extrusion forces were registered as a function of punch displacement. As a result of the introduction of SiC particles into the alloy matrix during the preliminary stage of the process, a more intensive increase of force is observed at smaller punch displacements (Fig. 1, curves 2-4), when compared to the matrix material (Fig. 1, curve 1). In each case, a slight decrease of force is evident within this stage, which is caused by a decrease of feedstock length in the die and, in consequence, a smaller feedstock-tool contact surface. The force required for the realization of a laminar extrusion stage increases slightly, as a result of the introduction of reinforcing phase particles into the matrix or resulting from an increase of their volume fraction.

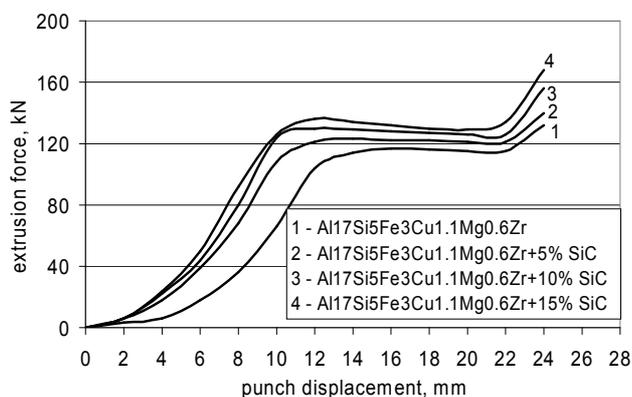


Fig. 1. Influence of chemical composition on extrusion force variations as function of punch displacement

Rys. 1. Wpływ składu chemicznego na przebieg zmian siły wyciskania w funkcji przemieszczenia stempla

For semi-finished products after compaction as well as for extruded materials, their relative densities were determined. For the materials after extrusion, their hardness and compressive strength were evaluated and stress-strain curves were constructed as well. Assessment of the uniformity of SiC distribution in the composite matrix was performed by means of microstructure examination.

RESULTS OF INVESTIGATIONS

Relative density. The density testing of the semi-finished products after hot compaction was realized using the geometrical technique, while in the case of extruded samples, the Archimedes method was applied. The obtained results are presented in Figure 2. The compaction of powder, realized with the assumed testing parameters, resulted in obtaining highly compacted materials, with a volume fraction of pores not exceeding 2%. In the case of the composites containing 10 and 15% silicon carbide particles, slightly lower average densities were observed, when compared to the matrix material and to a composite containing 5% SiC. Hot extrusion resulted not only in the mechanical processing of the material, but also in its densification. In the case of the matrix material and composites containing up to 10% silicon carbide particles, specimens were obtained showing a density close to that of a solid material. For a composite reinforced with 15% SiC particles, the relative density was slightly lower, amounting to $98.76 \pm 0.32\%$.

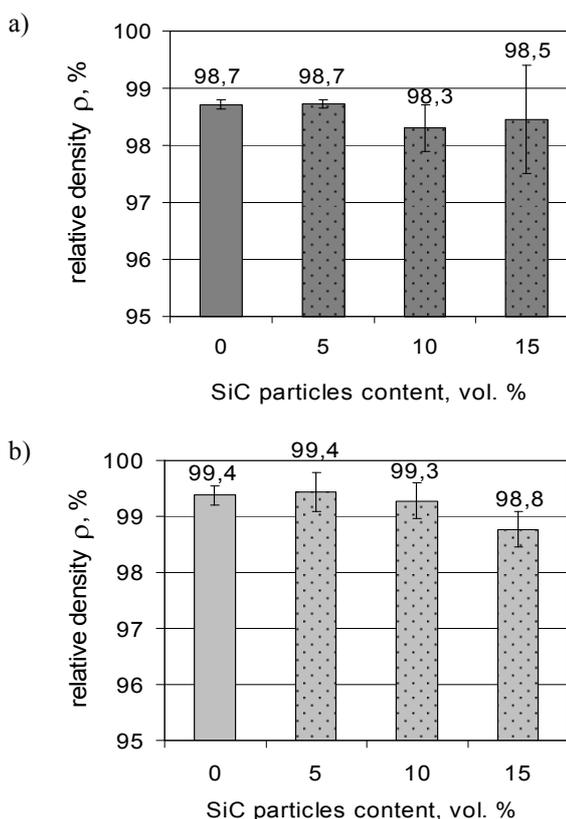


Fig. 2. Influence of chemical composition on relative density of Al17Si5Fe3Cu2Mg0.6Zr alloy and composites based on this alloy, reinforced with silicon carbide particles. Material: a) after preliminary hot compaction, b) after hot extrusion

Rys. 2. Wpływ składu chemicznego na gęstość względną stopu Al17Si5Fe3Cu2Mg0.6Zr i kompozytów na jego podstawie umocnionych cząstkami węgla krzemowego. Materiał a) po zagęszczaniu na gorąco, b) po wyciskaniu na gorąco

Hardness. The hardness was measured for the materials after hot compaction as well as after hot

extrusion. The hardness measurements were performed using the Brinell method. The results of the measurements are presented in Figure 3. The HB hardness of the materials under investigation depends on the degree of processing and the volume fraction of the reinforcing phase particles in the matrix. As a result of the introduction of silicon carbide particles into the matrix or increasing their volume fraction, an increase of hardness is observed. The hot extrusion process realized with the assumed parameters resulted in a lowering of the HB level for all the materials under investigation, regardless of their chemical composition.

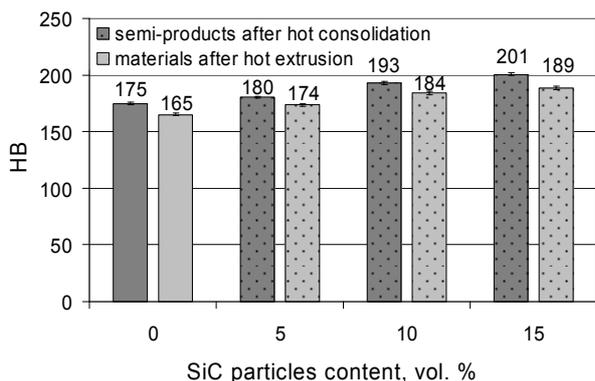


Fig. 3. HB hardness of Al17Si5Fe3Cu2Mg0.6Zr alloy and composites based on this alloy, reinforced with silicon carbide particles. Materials after hot compaction and after hot extrusion

Rys. 3. Twardość HB stopu Al17Si5Fe3Cu2Mg0.6Zr i kompozytów na jego osnowie umocnionych cząstkami węgliku krzemu, Materiały po zagęszczaniu na gorąco i po wyciskaniu na gorąco

The compressive strength was evaluated for the materials after hot extrusion. The compression test was performed at room temperature, using cylindrical specimens of a height to diameter ratio amounting to 1.1 and with a traverse speed of $0.15 \text{ mm}\cdot\text{s}^{-1}$. The results of the tests are collected in Table 1. The introduction of 5 and 10% silicon carbide particles into the matrix results in a slightly increased compressive strength. For a composite containing 15% SiC, a drop of the average R_c value occurred at a level lower than that of the matrix material.

TABLE 1. Influence of chemical composition on compressive strength of hot-extruded Al17Si5Fe3Cu2Mg0.6Zr alloy and composites based on this alloy, reinforced with silicon carbide particles

TABELA 1. Wpływ składu chemicznego na wytrzymałość na ściskanie wyciskanych na gorąco stopu Al17Si5Fe3Cu2Mg0.6Zr i kompozytów na jego osnowie umocnionych cząstkami węgliku krzemu

Chemical composition	$R_c \pm \Delta R_c$, MPa
Al17Si5Fe3Cu1.1Mg0.6Zr	643.79 ± 23.12
Al17Si5Fe3Cu1.1Mg0.6Zr + 5% SiC	654.34 ± 04.78
Al17Si5Fe3Cu1.1Mg0.6Zr + 10% SiC	662.08 ± 34.05
Al17Si5Fe3Cu1.1Mg0.6Zr + 15% SiC	625.18 ± 11.66

Stress-strain curves. Based on the results obtained from the compression test performed at room temperature as well as at 200°C , stress-strain curves were constructed. The curves are presented in Figure 4.

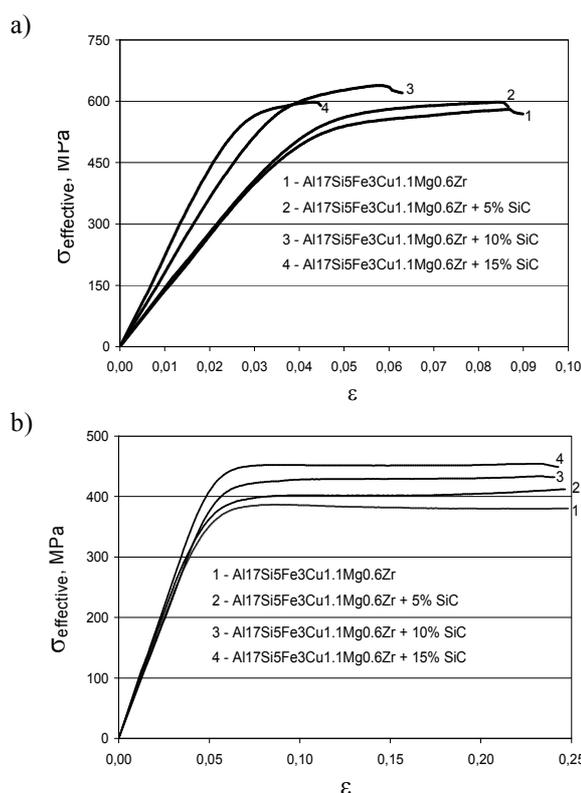


Fig. 4. Influence of chemical composition on character of stress-strain curves obtained in compression test at room temperature (a) and at 200°C (b), for specimens produced by means of hot extrusion of preliminary compacted semi-finished products

Rys. 4. Przebieg krzywych naprężenie - odkształcenie, otrzymanych w próbie ściskania w temperaturze pokojowej (a) oraz przy temperaturze 200°C (b) próbek otrzymanych w procesie wyciskania na gorąco wstępnie zagęszczanych półwyrobów

In the case of specimens compressed at room temperature, brittle fracture occurred regardless of the chemical composition. As a result of the introduction of reinforcing phase particles into the matrix and increasing their volume fraction, the value of the strain at fracture decreased. The values of true stress corresponding to a given strain increased with an increasing volume fraction of SiC, up to its level of 10%. In the case of a composite containing 15% SiC particles, the corresponding values of true stress dropped. Specimens compressed at 200°C showed plastic deformation. The stress at which the deformation of a material occurred increased as a result of the introduction of silicon carbide particles into the matrix and also with an increasing volume fraction of SiC.

Microstructure. The microstructure of the matrix and composites was examined with the application of an optical microscope. The micrographs made on polished and etched sections are presented in Figure 5. The arrows visible on longitudinal sections designate the direction of material flow during extrusion.

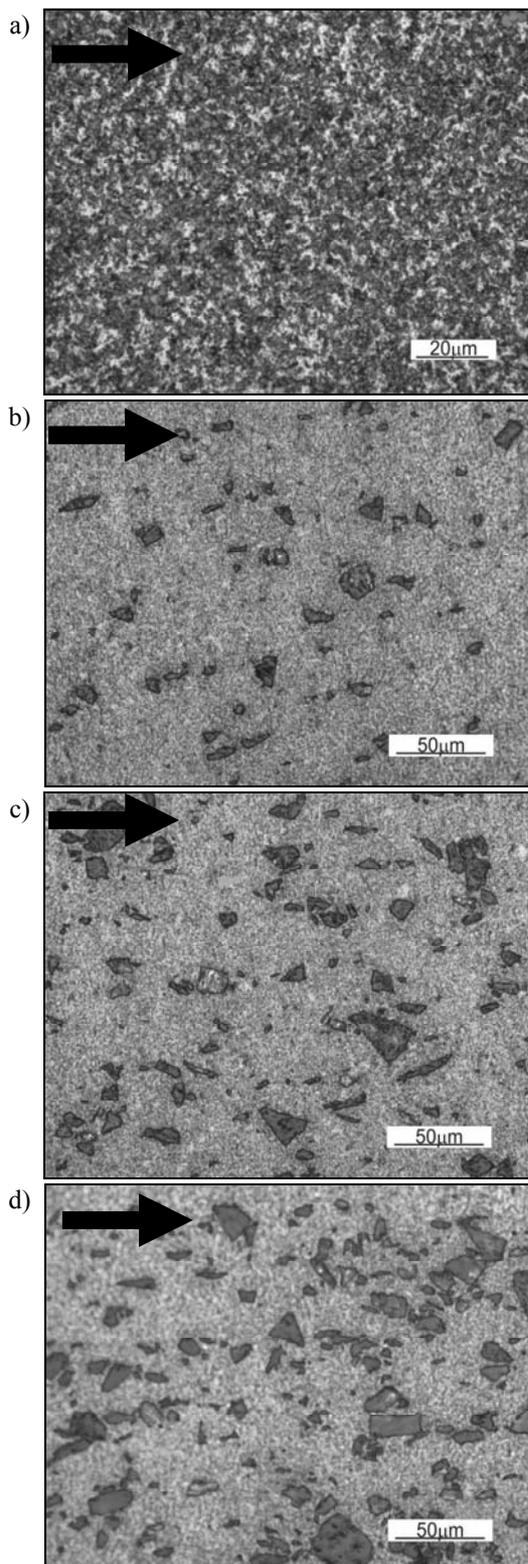


Fig. 5. Microstructures of materials obtained by means of extrusion at temperature 485°C, after preliminary hot compaction; a) Al17Si5Fe3Cu1.1Mg0.6Zr alloy powder, b-d) composite mixtures based on this alloy, reinforced with silicon carbide particles. Volume fraction of SiC particles: b) 5%, c) 10%, d) 15%. Longitudinal sections, a) etched, b-d) polished

Rys. 5. Mikrostruktury tworzyw otrzymanych przez wyciskanie przy temperaturze 485°C wstępnie zagęszczonych na gorąco; a) proszku stopu Al17Si5Fe3Cu1.1Mg0.6Zr, b-d) mieszanin kompozytowych na jego osnowie umocnionych cząstkami węgla krzemowego (b-d). Udział cząstek: b) 5%, c) 10%, d) 15%. Zglądy wzdłużne, a) trawiony, b-d) polerowane

The etched section was prepared for a non-strain-hardened Al17Si5Fe3Cu1.1Mg0.6Zr alloy (Fig. 5a), while in the case of composite materials, the observations were made using polished sections (Fig. 5b-d), which were aimed at making it easier to investigate qualitatively the distribution of silicon carbide particles in the matrix. The microstructure of the matrix is highly fine-grained (Fig. 5a). A uniform distribution of reinforcing phase particles in the composite matrix was observed (Fig. 5b-d). Microstructure examination performed on the matrix and composite sections did not show the occurrence of pores, which confirms the obtained results of density testing. The silicon carbide particles are properly situated in the matrix, no discontinuities were observed at the particle-matrix boundaries, and no cavities formed in the matrix as a result of the falling out of reinforcing phase particles during metallographic specimen preparation were observed (Fig. 5b-d). In the case of SiC particles of an oblong shape, the observations show their partial orientation in accordance with the direction of material flow during extrusion, designated by arrows on individual sections.

CONCLUSIONS

Based on the results of the testing of selected properties of the materials obtained by means of extrusion at a temperature of 485°C, where the feedstock are semi-finished products in the form of a hot-compacted Al17Si5Fe3Cu1.1Mg0.6Zr alloy powder as well as the mixtures of this powder and silicon carbide particles, with a volume fraction of SiC amounting to 5, 10 and 15%, the following conclusions can be formulated:

1. The introduction of a reinforcing phase into the matrix and increasing its volume fraction resulted only in a slight increase of force required for the realization of extrusion.
2. The process of powder compaction realized with the assumed parameters resulted in obtaining highly compacted materials, with a slight final densification achieved also during hot extrusion. For the matrix and for composites containing up to 10% silicon carbide particles, samples were obtained showing densities close to that of a solid material. In the case of a composite containing 15% SiC, the relative density was slightly lower.
3. The mechanical properties of the materials under investigation depend on the forming method and the volume fraction of the reinforcing phase particles in the matrix. An increase of material hardness was found as a result of the introduction of SiC particles into the matrix or following the increase of their volume fraction. Hot extrusion realized with the assumed parameters caused a drop of hardness of all the materials, when compared with the semi-finished products after preliminary compaction.

In the case of extruded materials, the introduction of 5 and 10% silicon carbide particles into the matrix resulted in an increased compressive strength, while the average value of R_c determined for a composite containing 15% SiC was lower than that of the matrix material.

4. Based on the character of stress-strain curves it was found that the introduction of particles into the matrix and increasing their volume fraction leads to a decrease of the strain at fracture value. The values of true stress corresponding to a given strain increased with an increasing volume fraction of SiC, up to its level of 10%. In the case of a composite containing 15% SiC particles, the corresponding values of true stress dropped. Specimens compressed at 200°C showed plastic deformation. The stress at which the deformation of the material occurred increased as a result of the introduction of silicon carbide particles into the matrix and also with an increasing volume fraction of SiC.
5. Observation of the composite microstructures showed a uniform distribution of silicon carbide particles in the matrix. No discontinuities were observed at the reinforcing phase-matrix boundaries. The matrix microstructure visible on etched sections is fine-grained.

Based on the obtained results of the investigations, it can be concluded that the properties of composites based on the Al17Si5Fe3Cu1.1Mg0.6Zr alloy can be modified by means of the degree of processing and the volume fraction of the reinforcing phase. In the case of hot-extruded products, with the assumed technological parameters, the introduction of silicon carbide particles into the matrix, up to their volume fraction of 10%, has a favourable effect, while with a 15% reinforcing phase, a drop of the investigated material properties at room temperature and their increase at an elevated temperature is observed. The decision to introduce SiC

particles into the Al17Si5Fe3Cu1.1Mg0.6Zr alloy matrix as a reinforcing phase, as well as of their volume fraction, should depend on the foreseen working conditions of the element made of this material.

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