

B. LESZCZYŃSKA-MADEJ*

THE EFFECT OF SINTERING TEMPERATURE ON MICROSTRUCTURE AND PROPERTIES OF Al – SiC COMPOSITES

WPLYW TEMPERATURY SPIEKANIA NA MIKROSTRUKTURĘ I WŁASNOŚCI KOMPOZYTÓW Al – SiC

Attempts have been made to describe the influence of sintering temperature on the microstructure and properties of Al – SiC composites. Mixtures of 100%Al and Al – 5% SiC, Al – 10% SiC were produced by tumbling for 30 minutes in the Turbula T2F mixer. The powders were subsequently cold pressed at pressure 300MPa in a rigid die on a single action press. The green compacts were sintered in nitrogen at 580°C and 620°C for one hour.

The main objective of this work was to determine influence of chemical composition and the manufacturing parameters on microstructure and properties of Al – SiC composites produced by powder metallurgy technology.

Keywords: Al – SiC composites, sintering, microstructure, cold pressing, tribological properties

W pracy przedstawiono wyniki badań kompozytów aluminiowych umacnianych cząstkami węgla krzemu SiC. Wytworzono spiekane kompozyty o zawartości 5% i 10% SiC. Jako materiał referencyjny stosowano Al99,7 wytworzone w takich samych warunkach, jak kompozyty Al – SiC. Materiały wytworzono na drodze jednostronnego prasowania przy zastosowaniu ciśnienia prasowania 300MPa. Kolejno kształtki poddano spiekaniu w atmosferze azotu w temperaturze 580°C oraz 620°C. Na tak wytworzonych spiekach przeprowadzono obserwacje mikrostruktury, pomiar twardości metodą Brinella, próbę zginania oraz przy zastosowaniu testera T05 badanie odporności na zużycie ściernie.

Celem pracy jest określenie wpływu składu chemicznego oraz parametrów procesu wytwarzania na mikrostrukturę i właściwości kompozytów Al – SiC wytwarzanych technologią metalurgii proszków.

1. Introduction

Metal matrix composites (MMCs) have evolved significantly during the past 30 years. Great interest of this group of the materials is caused because of their attractive properties, such as specific strength and stiffness [1-3]. Most of the commercial works on MMCs are focused on aluminium or aluminium alloy as the matrix material. It is possible to add hard particles such as: Al₂O₃, AlN, SiC, TiC and TiB₂ to primary aluminium alloy powders by the conventional premixing process to improve wear resistance of the sintered aluminium alloy [1, 2]. The most characteristic and positive property of aluminium powders is its excellent compressibility, density level of 90% of theoretical density can be achieved at about 160 MPa uniaxially compacting pressure, and 95% at about 350 MPa [4, 5].

Cold pressing of randomly mixed powders has been used widely for making parts in industry because of the simplicity and efficiency of the method. Although sintering is necessary to strength pressed materials. The presence of a surface oxide makes aluminium difficult to sinter. The thickness of the oxide is dependent on the temperature at which it is formed and the atmosphere in which it is stored, particularly the humidity. The thickness of the oxides on atomized powder (as was used

in the investigations presented in the article) can vary from 50-150 Å. The problems with sintering aluminium or other metals with stable oxides have been explained in terms of the relative diffusion rates through the oxide and the metal [6].

The aim of the presented study is to determine influence of chemical composition and the sintering temperature on microstructure and properties of Al – SiC composites produced by powder metallurgy technology. The following mixtures: 100%Al, 95%Al + 5%SiC, 90%Al+10%SiC were prepared for investigations. The powder mixtures were cold pressed at pressure 300 MPa and then sintered at temperatures: 580°C and 620°C in nitrogen atmosphere for 1 hour.

2. Experimental procedure Initial material

The starting powders are shown in Fig. 1 and characterised in Table 1. The microstructure of aluminium powders is presented in Fig. 2.

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF NON-FERROUS METALS, 30-059 KRAKÓW, AL. MICKIEWICZA 30, POLAND

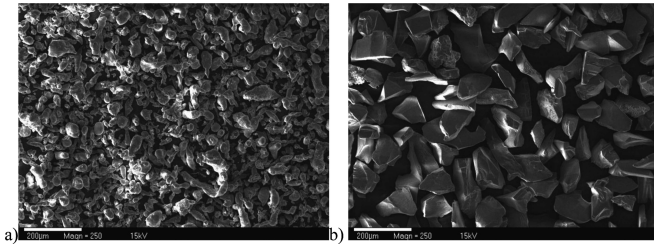


Fig. 1. SEM micrograph of aluminium (a) and SiC (b) powders

TABLE 1

Selected properties of powders and mixtures used for examination

Powder/ Mixture	Particle size μm	Tap density g/cm^3	Flow time 50g/s	Theoretical density g/cm^3
Al	to 63 μm	0,9	–	2,70
SiC	40 – 60 μm	1,37	54,48	3,21
Al+5%SiC	to 63 μm	0,97	–	2,73
Al+10%SiC	to 63 μm	0,98	–	2,75

Presented in Fig. 2 microstructure of aluminium powders show small pores and light particles distributed uniformly on the grain boundary.

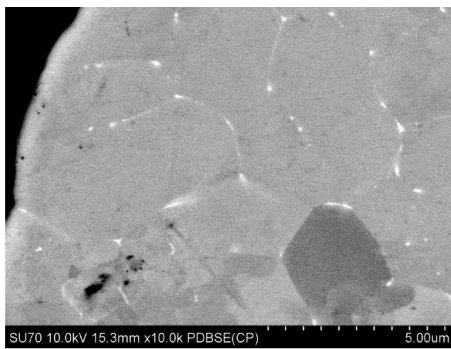


Fig. 2. Microstructure of aluminum powders; SEM

3. Production of Al, Al – 5%SiC and Al – 10%SiC composites

Mixtures of 100%Al and Al – 5% SiC, Al – 10% SiC were prepared by tumbling the powders for 30 minutes in the Turbula T2F mixer. The powders were subsequently cold pressed at 300MPa in a rigid die on a single action press without lubricants application. The green compacts were sintered in nitrogen at 580°C and 620°C for one hour.

The specimens were subsequently tested for Brinell hardness, bending strength and resistance to wear. They were also analyzed by means of both light microscopy (LM) and scanning electron microscopy (SEM). The wear tests were carried out using the block – on – ring tester (Fig. 3).

The sample (1) was mounted in a sample holder (4) equipped with a hemispherical insert (3) ensuring proper contact between the sample and a rotating ring (2). The wear surface of the sample was perpendicular to the pressing direction. Double lever system input the load L , pressing the sample to the ring with the accuracy of $\pm 1\%$. The ring rotated with a constant rotating speed.

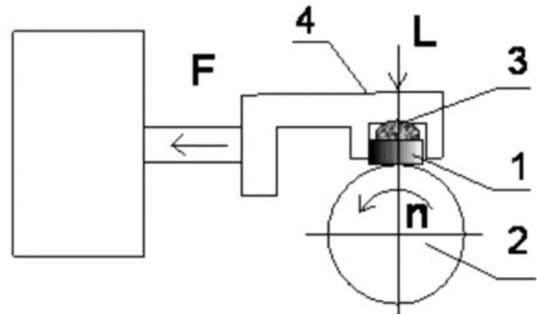


Fig. 3. Schematic view of block – on – ring tester

The wear tests conditions chosen for the current investigations were following:

- tested samples – rectangular specimens 20×4×4 mm,
- counterpart (rotating ring) – φ 49,5×8 mm, heat treated steel 100Cr6, 55 HRC,
- dry sliding,
- rotational speed – 136 rev./min.,
- load – 50 N,
- sliding distance – 250 m, 500 m.

The measured parameters were:

- loss of sample mass,
- friction force F (used to calculate the coefficient of friction).

This tester enabled performing tests in accordance with the methods determined in ASTM D 2714, D 3704, D 2981 and G 77 Standards.

4. Results and discussion

After cold pressed at pressure 300MPa in a rigid die on a single action press, the green compacts with density as summarized in Table 2 were obtained. Green density of compacts decreases with the increasing of SiC content.

TABLE 2

Densities values for cold pressed samples

Material	Density, ρ [g/cm^3]	Theoretical density, ρ_t [g/cm^3]	Relative density [%]
Al	2.505	2.7	92.8
Al – 5%SiC	2.5	2.73	91.59
Al – 10%SiC	2.508	2.75	91.3

After sintering in nitrogen atmosphere at 580°C and 620°C the following densities were obtained (Fig. 4, Table 3).

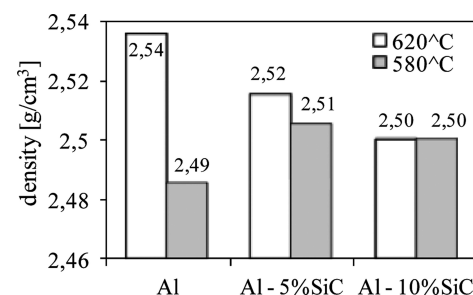


Fig. 4. Density of materials as a function of composition and sintering temperature

TABLE 3

Densities values for sintered samples

Material	density, ρ [g/cm ³]	Theoretical density, ρ_t [g/cm ³]	Relative density [%]
Sintering temperature – 580°C			
Al	2.49	2.7	92.14
Al – 5%SiC	2.51	2.73	91.70
Al – 10%SiC	2.50	2.75	91.01
Sintering temperature – 620°C			
Al	2.54	2.7	93.73
Al – 5%SiC	2.52	2.73	92.15
Al – 10%SiC	2.5	2.75	90.98

The microstructures of as – sintered samples presented in Figs. 5-9 illustrate uniform distribution of SiC particles and pores on the aluminium matrix. The microstructures obtained by the light microscopy look similar for samples sintered both at 580°C and 620°C. SEM observations with (Figs. 8, 9) the maps of the O, Si and Al distributions show occurrence of the Al₂O₃ oxide distributed uniformly in the whole sample on the grain boundary. Probably there are also nitrides inside grains. Confirmations of this thesis are results presented in papers [4, 6]. Results of the TG and DTA analysis presented by T. Pieczonka [4] postulated nitrogen solubility in aluminium. Presented effects of investigations indicate that liquid phase may be formed at temperature 600-620°C. The formation of aluminium nitride is an exothermic reaction which may occur if the freshly exposed aluminium metal surface meets nitrogen [3]. Thermal expansion may cause the oxide layer to fracture. Under N₂, the exposed metal will readily react according to reaction (1) [6]:



G.B.Schaffer and B.J. Hall [6] confirmed presence of Al-Ni by the X – ray diffraction patterns and the energy – dispersive spectra. They also suggest that the quantity of the nitride decreases as the green porosity decreases.

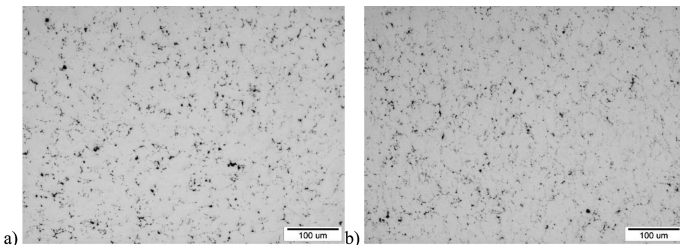


Fig. 5. Microstructure of as – sintered Al; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

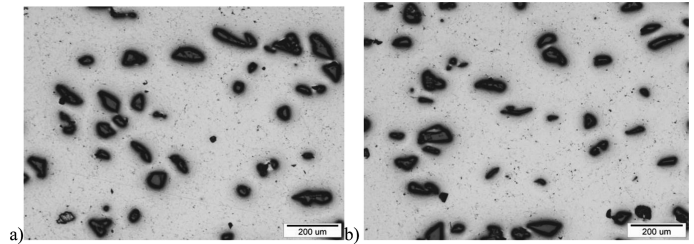


Fig. 6. Microstructure of as – sintered Al – 5%SiC composites; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

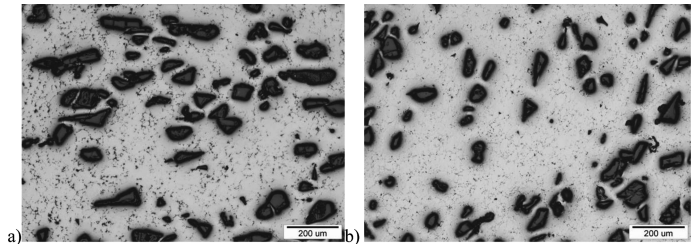


Fig. 7. Microstructure of as – sintered Al – 10%SiC composites; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

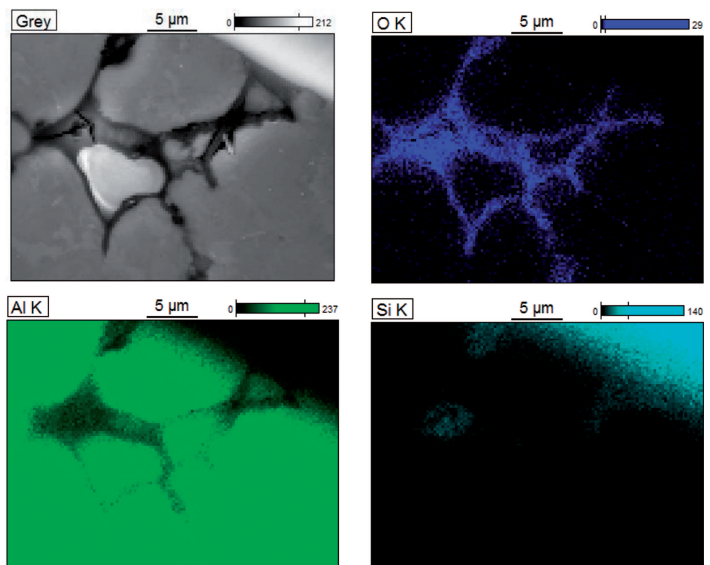


Fig. 8. Microstructure and the maps of the O, Si and Al distributions of as – sintered Al – 10%SiC composite; sintering temperature – 620°C

The Brinell hardness of the investigated materials increases with the increasing of SiC carbide contents (Fig. 10), whereas the bending strength seems to be adversely affected by the addition of silicon carbide (Fig. 11). Increasing the sintering temperature has a positive effect on hardness and bending strength of as – sintered materials. Hardness effect is a result of higher density level obtained by sintering at temperature 620°C and formation of dispersed Al₂O₃ particle distributed uniformly in whole sample.

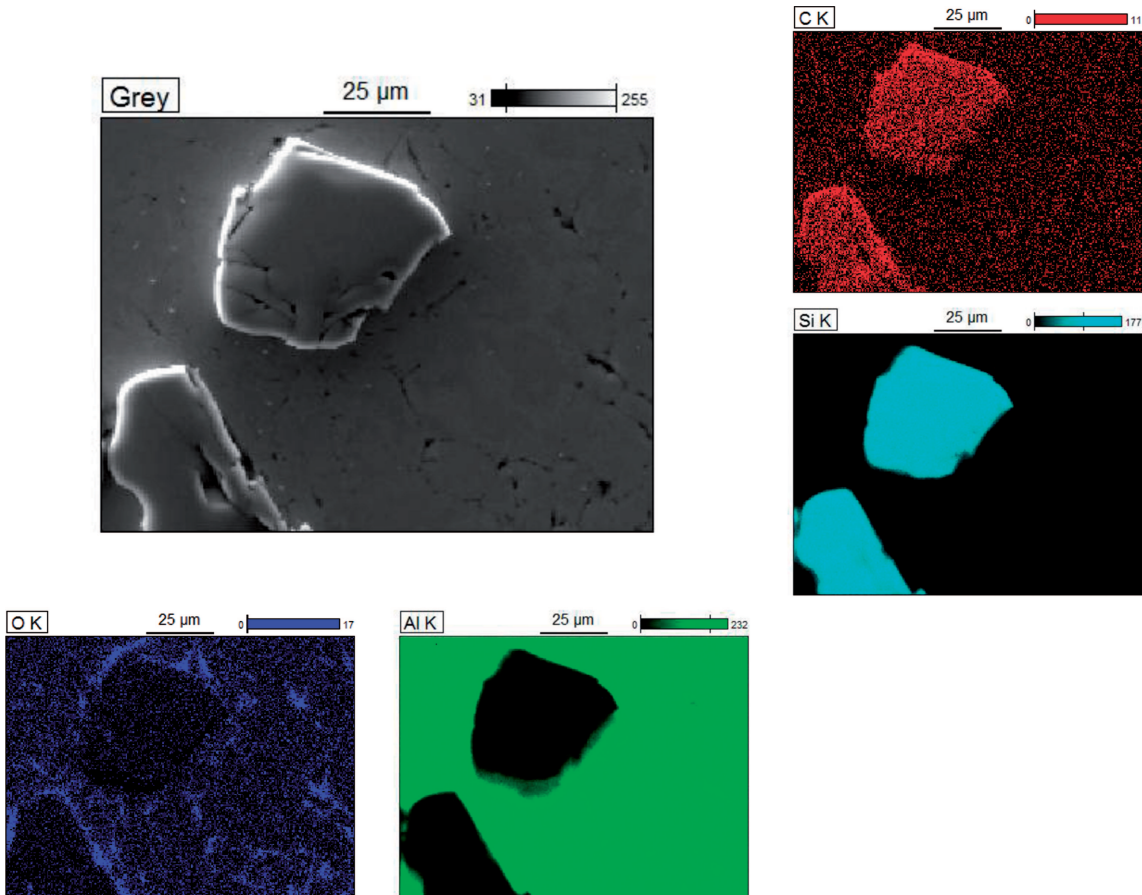


Fig. 9. Microstructure and the maps of the O, Si, Al and C distributions of as – sintered Al – 10%SiC composite; sintering temperature – 580°C

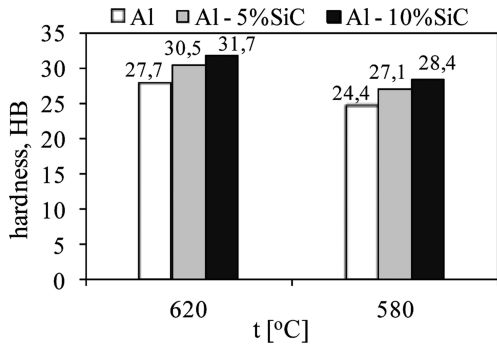


Fig. 10. Brinell hardness of as – sintered aluminum, Al – 5SiC and Al – 10SiC composites

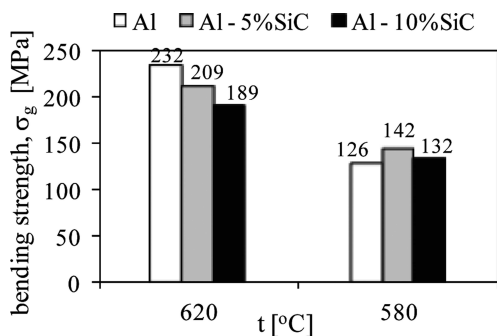


Fig. 11. Bending strength of as – sintered aluminum, Al – 5SiC and Al – 10SiC composites

It is evident that the as – sintered properties of the investigated materials are a complex function of the silicon carbide content and the sintering temperature.

Fractography of as – sintered at temperature 620°C aluminium sample pointed out transcrystalline ductile character with elevations and hollows formed during plastic deformation (Fig.12a). After sintering at lower temperature – 580°C fractography show loose powders particles. This phenomenon is a consequence of sintering in to low temperature (Fig.12b). Presented in Figures 13a and 14a fractography pointed out transcrystalline ductile character under the aluminium matrix. Also characteristic in the composites are observed cracking of the SiC phase and voids, which forms during pulling of the SiC particle or are the pores. Characteristic feature of the as – sintered at temperature 580°C composites is cohesion lack between aluminium matrix and SiC particles and simillary as in aluminium sample loose powders particles (Figs.13b, 14b).

The Al₂O₃ surface oxides could be breaking during sintering of aluminium at temperature 620°C, what was described by the authors [4, 6] and small diffuse connections forms between aluminium particles. Confirmation of this are results of the bending strength, which after sintering at temperature 620°C are higher about 50-80% with the comparison to the sintering at the 580°C.

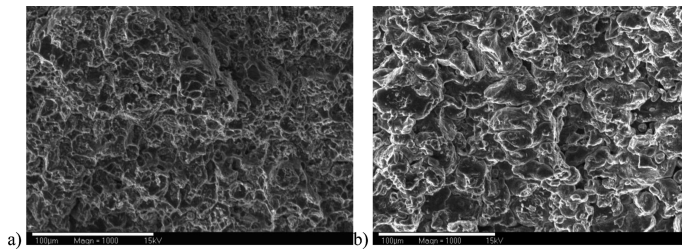


Fig. 12. Fractography of as – sintered Al; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

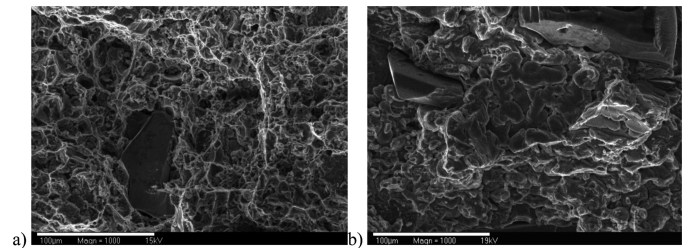


Fig. 13. Fractography of as – sintered Al – 5%SiC composites; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

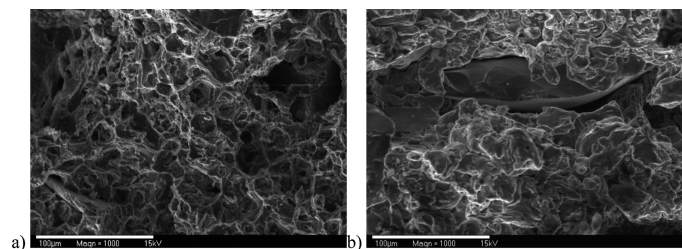


Fig. 14. Fractography of as – sintered Al – 10%SiC composites; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

The wear results are given in Figures 15 and 16. It was found that the tribological properties of the investigated materials depends on the sintering temperature and the amount of SiC particles. Determined friction coefficients values are high, on the sliding distance 250 m they are in the range of 0.38 – 0.87 and adequately 0.46 – 1.02 on the sliding distance 500 m (Fig.15). Friction coefficient of the as – sintered at 620°C samples tends to attain constant value after sliding distance 500m. Similar behavior in friction has been found earlier in Al – MMCs composites [7,8].

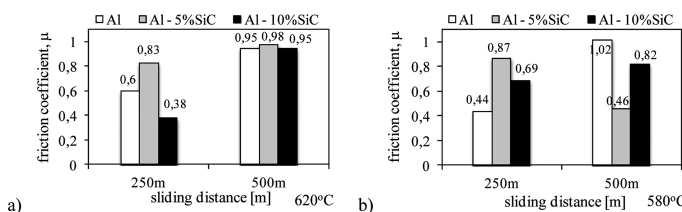


Fig. 15. Friction coefficient as a function of sliding distance; samples sintered at temperature 620°C (a), samples sintered at temperature 580°C (b)

The lowest loss of mass was observed in the materials sintered at temperature 620°C (Fig. 16a). Characteristic is considerable increase loss of mass in the aluminium sintered at the temperature 580°C with the comparison to aluminium

sintered at the temperature 620°C – loss of mass increased above 8 times (Fig.16b).

Higher loss of mass after sintering at the temperature 580°C can be affected by the insufficient cohesion of the particles and lack of the stable joints between aluminium matrix – silicon carbide.

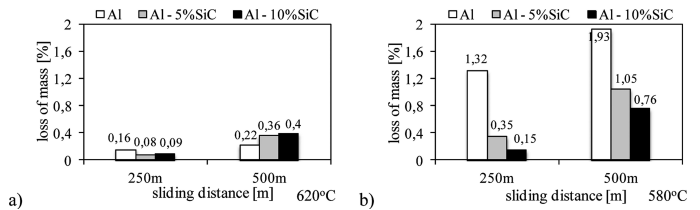


Fig. 16. Loss of mass as a function of sliding distance; samples sintered at temperature 620°C (a), samples sintered at temperature 580°C (b)

Characteristic surface topographies after the wear tests are presented in Figs. 17-19.

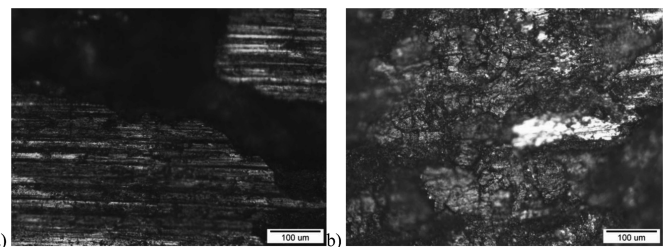


Fig. 17. The surface topographies of as – sintered aluminium, sliding distance 500 m; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

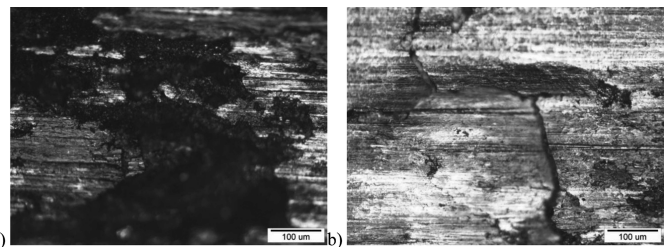


Fig. 18. The surface topographies of as – sintered Al – 5%SiC composites, sliding distance; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

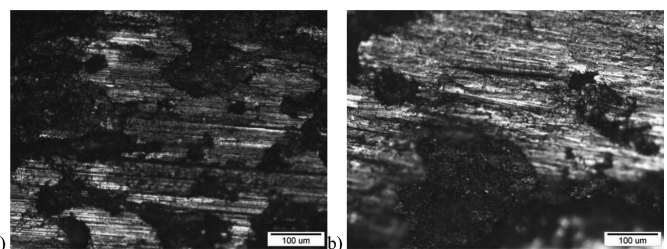


Fig. 19. The surface topographies of as – sintered Al – 10%SiC composites, sliding distance; sintered at temperature 620°C (a), sintered at temperature 580°C (b)

The surface topographies of as – sintered aluminium and Al – SiC composites show, that the main wear mechanism is an abrasive wear, mainly through the microcutting and drawing

and also pulling parts of the materials (Fig. 17-19). Also there are holes, which correspond to traces of added SiC particles detached from the matrix. The detached SiC particles seem to cause abrasive wear. Elongation of the sliding distance to 500 m results in increasing part of adhesive wear (Fig. 17b, 19b). Also characteristic are cracks placed perpendicular to the sliding distance observed in the samples sintered at the temp. 580°C (Fig. 17b, 18b). Probably these cracks form because temperature 580°C is insufficient for good consolidation, confirmation of this are observed in Figs. 12b, 13b, 14b fractography showing cohesion lack between aluminium matrix and SiC particles and loose aluminium powders particles.

5. Conclusions

Cold pressing of aluminium powders and Al – SiC composites at pressure 300MPa in a rigid die on a single action press and subsequently sintering at the temperature 620°C make possible obtain samples with the density above 90%. The density of the composites precisely depends on the mixture chemical composition and the sintering temperature.

The Brinell hardness of the investigated materials increases with the increasing of SiC contents, whereas the bending strength seems to be adversely affected by the addition of silicon carbide. The highest sintering temperature influenced on the increasing of hardness and bending strength values.

Temperature 580°C is too low for forming stable diffusion connection between powders particles. Fractography shows loose powders particles and cohesion lack between aluminium matrix and SiC particles.

Increase of the Al – SiC composites properties is connected with the sintering mechanism at temperature 620°C, what will be subject of the further investigations.

Acknowledgements

The Statutory Activities no 11.11.180.449 supported this work. Author wish thank PhD M.Madej for help in composites preparation, wear tests and fruitful discussion and also PhD T. Tokarski for his help in the SEM investigation.

REFERENCES

- [1] Z. Gnjidic, J. Grbovic, M. Mitkov, D. Bozic, *Powder Metallurgy* **46**, 1, 21 (2003).
- [2] K. Kondoh, Y. Takeda, *Powder Metallurgy* **43**, 1, 69 (2000).
- [3] M. Madej, *Archives of Metallurgy and Materials* **55**, 1, 61 (2010).
- [4] T. Pieczonka, *Powder metallurgy processing of aluminium in Polish Metallurgy 2006-2010 in time of the worldwide economic crisis*, Committee of Metallurgy of the Polish Academy of Sciences (2010).
- [5] K. Shin, D. Chung, S. Lee, *Metallurgical and Materials Transactions A* **28A**, 1997 (1996).
- [6] G.B. Schaffer, B.J. Hall, *Metallurgical and Materials Transactions A* **33**, 3279 (2001).
- [7] B. Venkataraman, G. Sundararajan, *Acta Mater.* **44**, 451 (1996).
- [8] H. Akbulut, M. Durmam, F. Yilmaz, *Wear* **215**, 170 (1998).

Received: 10 January 2012.