

The Influence of SiC on the Properties of Aluminium Powder Obtained from Recycled Materials

M. SUŚNIAK^{1*}, J. KARWAN-BACZEWSKA¹, J. DUTKIEWICZ², K. PIETRZAK³

¹ AGH – University of Science and Technology, Faculty of Non-Ferrous Metals, Cracow, Poland

² Polish Academy of Science, Institute of Metallurgy and Materials Engineering, Cracow, Poland

³ Motor Transport Institute, Centre for Material Testing, Warsaw, Poland

*e-mail: susniak@agh.edu.pl

Abstract

The paper presents the experiments concerning the influence of SiC on the process of mechanical alloying of recycled materials in the form of aluminium chips. Oxidized aluminium chips used for the experiment were derived from waste products. Chemical, physical and engineering properties were characterized in the first step. Study shows repeatable chemical composition of the chips. Technological properties preclude the use of them as material for automatic charge (flow rate about 30 s). Therefore attempts were made to produce a composite powder by using the powder metallurgy method. Mechanical alloying process of aluminium chips with different amounts of SiC (0, 20, 40 wt%) and addition of Zr (2 wt%) was performed in a high energetic mill. Milling time was 20 hours using a milling speed of 200 rpm. Then, the characterization of obtained aluminium matrix composite powder was made by using a light microscope and scanning electron microscopy SEM/EDX. On the basis of the results of the microstructure we can observe that after the MA process, SiC reinforcement is located inside the aluminium particles which confirms the correct course of the process. The experiments showed that 40 wt% amount of SiC and 5 hours of the mechanical alloying allowed us to obtain the particles with an average area less than 2 square micrometers. The composite powders were tested via hot pressing processes (380°C/600 MPa/10 min). In order to determine the effect of addition of SiC reinforcement on the properties for the obtained sinters samples, hardness was studied at small loads by Vickers method. Addition of reinforcement had brought the desired effect of hardness increase in the samples with the SiC content. The influence of SiC on the processing and the properties of aluminium matrix powder was analyzed in details.

Keywords: Aluminium, Recycling, Powder Metallurgy, Silicon Carbide, Composites

WPLYW SiC NA WŁAŚCIWOŚCI PROSZKU ALUMINIUM OTRZYMANEGO Z RECYKLINGU MATERIAŁÓW

W niniejszym artykule przedstawiono wyniki eksperymentów dotyczące wpływu SiC na proces mechanicznej syntezy materiałów pochodzących z recyklingu w formie wiórów aluminium. Utlenione wióry aluminiowe stosowane w eksperymencie zostały uzyskane z odpadów. W pierwszym etapie przeprowadzono badania własności chemicznych, fizycznych i technologicznych wiórów aluminiowych. Wyniki badań wskazują, że skład chemiczny jest powtarzalny. Badania własności technologicznych nie pozwalają na używanie wiórów aluminiowych z recyklingu jako materiału do automatycznego zasypu (sympkość 30 s) do matrycy w procesie prasowania. Dlatego też postanowiono wytworzyć z nich proszek kompozytowy metodami metalurgii proszków. Proces mechanicznej syntezy dla wiórów stopów aluminiowych z różną zawartością wzmocnienia SiC (0, 20, 40 % wag.) oraz dodatkiem Zr (2 % wag.) przeprowadzono w wysokoenergetycznym młynie kulowym. Czas mielenia wynosił 20 godzin przy prędkości mielenia 200 obr./min. Następnie charakterystykę otrzymanych proszków kompozytowych przeprowadzono za pomocą mikroskopu optycznego oraz skaningowego mikroskopu elektronowego SEM / EDX. Na podstawie otrzymanych wyników badań mikrostruktury można zauważyć, że po procesie mechanicznej syntezy faza wzmocnienia SiC znajduje się wewnątrz cząstek aluminium, co potwierdza prawidłowy przebieg procesu. Badania wykazały, że 40 % wag. SiC i 5. godzinny proces mechanicznej syntezy pozwala na uzyskanie cząstek o średniej wielkości powierzchni mniejszej niż 2 mikrometry kwadratowe oraz zmianę kształtu wiórów aluminiowych na sferyczne aglomeraty kompozytowe. Z kolei proszek kompozytowy poddano procesowi prasowania na gorąco (380°C/600 MPa/10 min). W celu określenia wpływu fazy wzmacniającej SiC na właściwości otrzymanych spieków przeprowadzono badania twardości pod niskim obciążeniem metodą Vickersa. Dodatek SiC spowodował pożądany efekt wzrostu twardości w badanych próbkach. W toku badań szczegółowo przeanalizowano wpływ SiC na proces wytwarzania i właściwości proszku kompozytowego.

Słowa kluczowe: aluminium, recykling, metalurgia proszków, węgiel krzemu, kompozyty

1. Introduction

Nowadays, composite materials are becoming more and more popular for the sake of different properties. Thanks to the current technology processes such materials can be obtained to produce components with high performances. In scientific publications there are several examples concern-

ing the materials combining low weight and good plastic properties with high hardness and good thermal properties. The combination of such different materials can be made using powder metallurgy (PM). This method allows the manufacturing of parts from powders, without going through the liquid phase. Separate grains of powder are combined into a single mass during heating and strong pressing in

the protective atmosphere. The process of powder metallurgy is the economic method for high-volume production of small elements of simple shapes, and full-density compacts. This technology allows us to obtain a homogeneous microstructure free of inclusions and defects. Application of the PM process including mechanical alloying (MA) for the preparation of composite samples can be realized in different ways. The MA process is one of the technology that depends on the milling of powder mixtures with balls in a high energy ball mill. The repeated milling, cold welding, fracture and rewelding of the powder lead to the homogenized material with a uniform dispersion of particles. This technique is efficient for the preparation of unique alloys without necessity of employing high temperatures [1, 2].

Aluminium matrix composites are usually connected to very hard reinforced materials with high strength properties (SiC, Al₂O₃, B₄C, TiC) [3-5]. SiC is the most important non-oxide ceramic material which started to be produced in 1891 by the Ascheson process. In the MA process, particles of SiC are added to aluminium alloys in order to improve the mechanical properties of resultant composites. The addition of silicon carbide increases significantly yield strength, compressive and tensile strengths, and concurrently hardness. SiC reinforcement allows the Young's modulus and the thermal expansion coefficient to be increased. The aluminium composites with the addition of reinforcement have been used mostly in automotive and aerospace industry for the production of microtools and in general into the engineering sectors [6-8].

The paper presents the results of experiments concerning the influence of SiC on the process of mechanical alloying of recycled materials in the form of aluminium chips.

2. Experimental procedure

The powder characterization for the evaluation of chemical, physical and engineering properties was the first step of the research. Prior to the design of the manufacturing processes, the knowledge of the aforementioned properties is required and for this reason aluminium chips have been tested by bulk density and tap density. The size and shape of the powder particles, their adsorbed gases, the degree of oxidation and several other factors [9-11] are important to evaluate the correct processing method.

Oxidized aluminium chips used for the experiment were derived from waste products, and Atomic Absorption Spectrometry (AAS) was used for the characterization of their chemical composition.

Several important features characterize the properties of the chips, mainly bulk density, tap density, flow rate. Bulk density tests were performed by Scott method, while tap density was evaluated using a manual shaking method. Flow rate was evaluated with a Hall funnel apparatus, the sieve tests

were also performed in accordance with the existing standard. To identify the size and shape of aluminium chips, light microscopy and SEM were also used [11-15].

The next stage of the study was to prepare a mixture of chips with the addition of Zr (2 wt%) and SiC (0, 20, 40 wt%) particles with 1 % of stearic acid (CH₃(CH₂)₁₆COOH) as lubricant. The mixture was handled in a glove box under a purified argon atmosphere; in order to carry out the process of mechanical alloying a Fritsch Pulverisette 5 mill was used. A 15 min of milling was followed by 45 min of pause to cool down and to avoid overheating of powders. The overall milling time was 20 hours using a milling speed of 200 rpm. Metallographic examinations of the powders were carried out by light microscopy and scanning electron microscopy SEM/EDX.

Composites were obtained by hot pressing of the powder mixtures. Compacting was performed under the vacuum of 10⁻² bar at a pressure of 600 MPa and a temperature of 380°C for 10 minutes.

In order to determine the effect of addition of SiC reinforcement on the properties of obtained sinters samples, hardness was studied at small loads. The research was made with a Leica VMHT microhardness tester using 300 G of load for 15 s.

3. Results and discussion

Reproducibility and uniformity of chemical composition of the chips are important aspects in terms of the chips suitability for further technological processes. The average chemical composition of aluminium chips is presented in Table 1; the origin of aluminium chips is from different alloys from casting and forging.

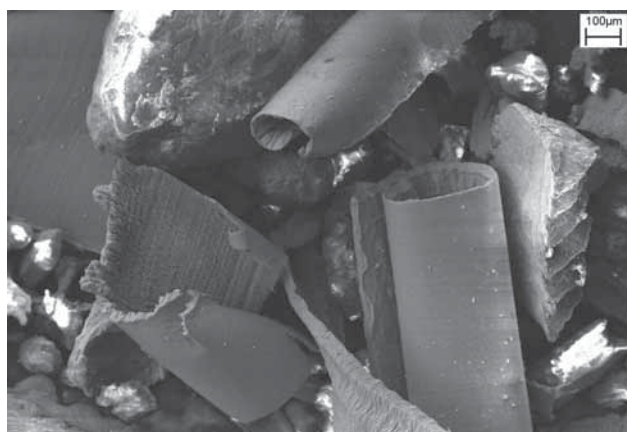
Fig. 1a shows that chips particles have a very irregular shape. The morphology is derived from the production process carried out. In fact, as a result of milling and turning processes, the chips are in the form of round shaped debris (Fig. 1b).

Powder bulk density is 0.783 g/cm³, whereas tap density is 0.984 g/cm³. The reason of the difference is the irregularity of particle shape, which avoids big volume reduction by joggle, but allows smaller parts to fill a free space between the biggest one. The flow properties of aluminium chips are relatively high (29.5 s), showing potential negative effects when using them in the automatic charge process. The sieve analysis showed that chips are divided into two fractions: coarse grained (0.2 to 0.4 mm) and fine grained (greater than 0.4 mm). This has a positive effect during mould charge because the smaller particles fill a free space between the biggest one, and in consequence a compacting process can be more effective [11].

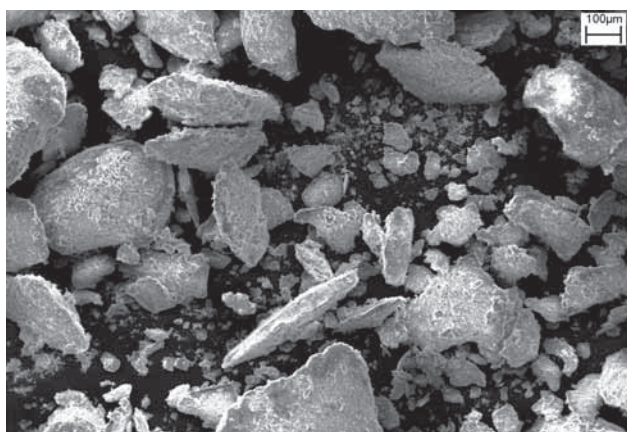
In order to get rid of stress generated during the machining and accumulated grease, aluminium chips were annealed for 3 hours at 500°C, and after that were cleaned with alcohol.

Table 1. Chemical composition (in wt%) of aluminium chips.
Tabela 1. Skład chemiczny wiórów aluminiowych (% wag.).

Si	Zn	Cu	Mg	Sn	Pb	Fe	Sb
7.5	5.2	2.4	0.57	0.6	0.35	0.17	0.1
Ni	Mn	As	Ga	Cr	Cd	Al	
0.045	0.033	<0.30	<0.10	<0.005	<0.001	balance	



a)



b)

Fig. 1. Microstructure of aluminium chips: a) before milling and b) after 20 h of milling.

Rys. 1. Mikrostruktura wiórów aluminium: a) przed procesem mielenia oraz b) po 20 h. mielenia.

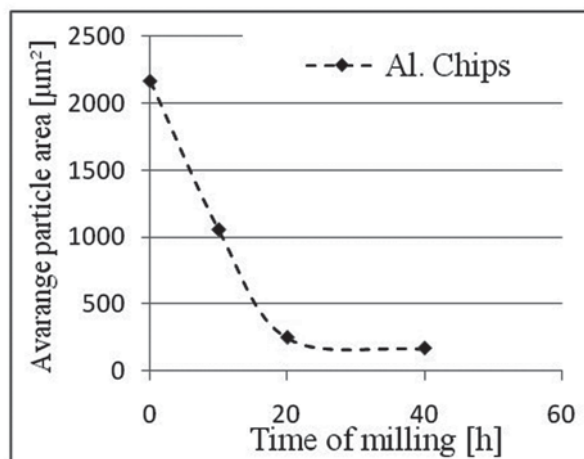
With the starting materials the following compositions were prepared for further processing:

- 1) Aluminium chips,
- 2) Aluminium chips + 2 wt% Zr + 20 wt% SiC,
- 3) Aluminium chips + 2 wt% Zr + 40 wt% SiC.

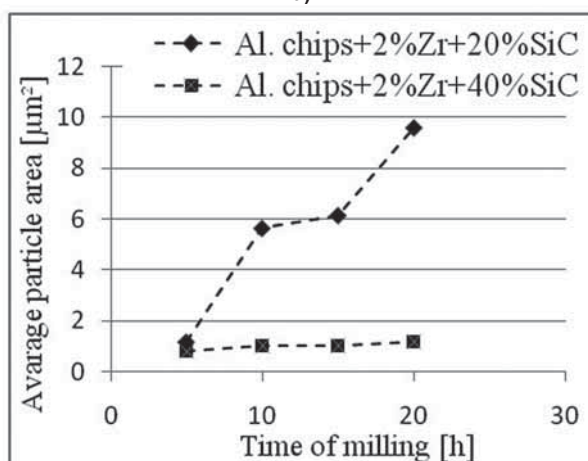
Composition 1 was milled for 40 hours whereas compositions 2 and 3 were milled for 20 hours. Different times of milling were selected because the disintegration process of aluminium alloy chips is slower than that one of aluminium chips mixed with SiC particles.

Image analysis was applied to measure of the average size of Al chips. The following Fig. 2 shows the average surface area as a function of the milling time. Before milling the surface area was bigger than $2000 \mu\text{m}^2$ whereas after 40 h it decreases till $170 \mu\text{m}^2$, with elongated shape (Fig. 3).

Through images 4 and 5 it's possible to observe that in the first stages of milling chips reduce their dimensions till the moment when particles start to agglomerate. After 5 h of mechanical alloying (compositions 2 and 3), the aluminium chips are smaller, and two different fractions can be observed (Figs. 4a and 5a). It is possible to verify that the dimension of chips decreases with increasing amounts of SiC (Fig. 2b). After 10 h of MA, all particles have a nodular shape and the agglomerate of a new composition starts to be generated. The optical microscopy method was used to measure the area of particles and agglomerates after 5, 10, 15, 20 hours of mechanical alloying. The results are reported in Fig. 2b.



a)



b)

Fig. 2. Relationship between time of milling process and average particle area: a) aluminium chips and b) composite powders.

Rys. 2. Zależność pomiędzy czasem mielenia i średnią powierzchnią cząstek: a) wióry aluminium oraz b) proszki kompozytowe.

Concerning composition 2, the agglomerates start to form after 5 hours of milling and their final dimension, at the end of the process, is $9.6 \mu\text{m}^2$. The smallest particles have been obtained for composition 3 with 40 % reinforcement of SiC; after MA, the average particle area does not ex-

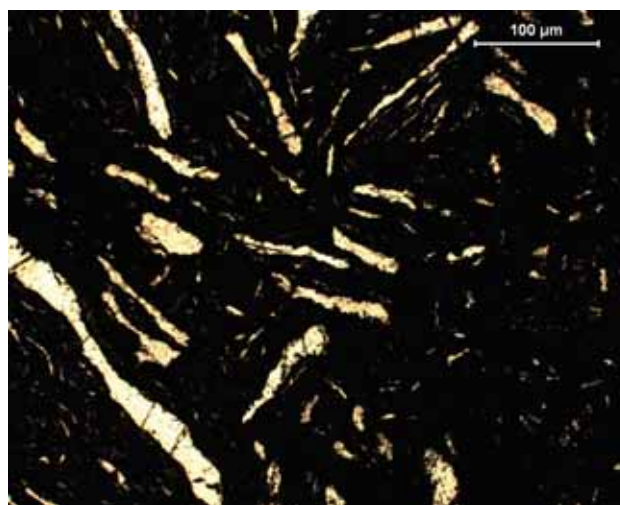
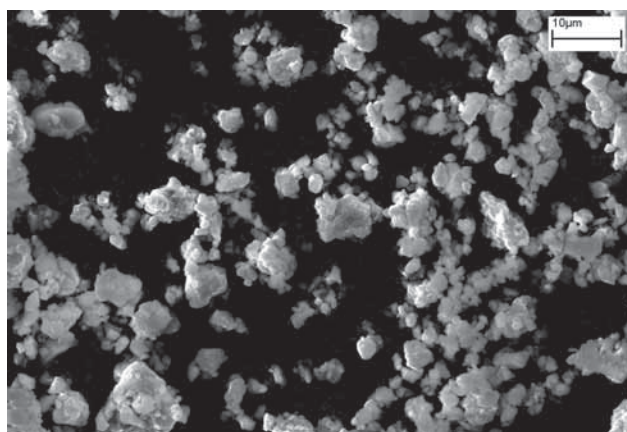
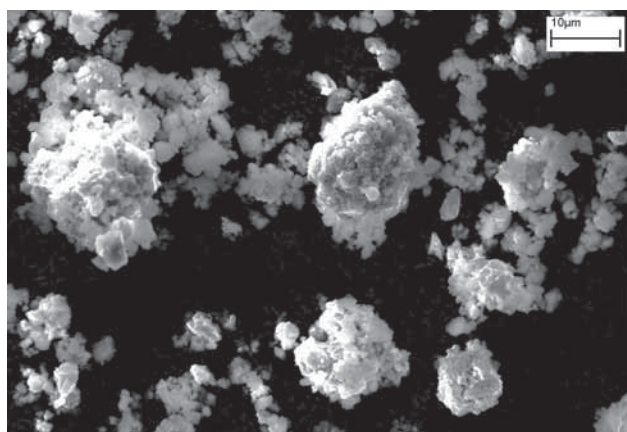


Fig. 3. Shape of aluminium chips particle after 40 hours of milling.
Fig. 3. Kształt cząstek wiórów aluminium po 40 godzinach mielenia.



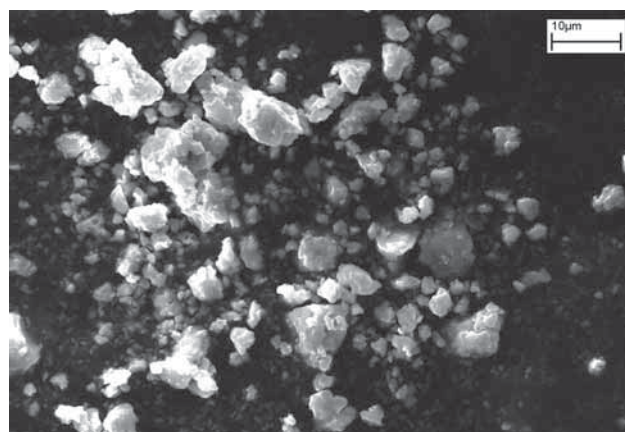
a)



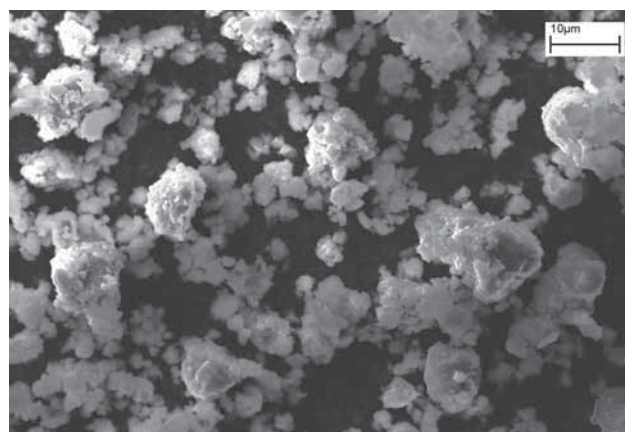
b)

Fig. 4. Aluminium chips + 2 wt% Zr + 20 wt% SiC: a) after 5 h and b) after 20 h of milling.

Rys. 4. Wióry aluminiowe + 2 % wag. Zr + 20 % wag. SiC: a) po 5 h i b) po 20 h mielenia.



a)



b)

Fig. 5. Aluminium chips + 2 wt% Zr + 40 wt% SiC: a) after 5 h and b) after 20 h of milling.

Rys. 5. Wióry aluminiowe + 2 % wag. Zr + 40 % wag. SiC: a) po 5 h i b) po 20 h mielenia.

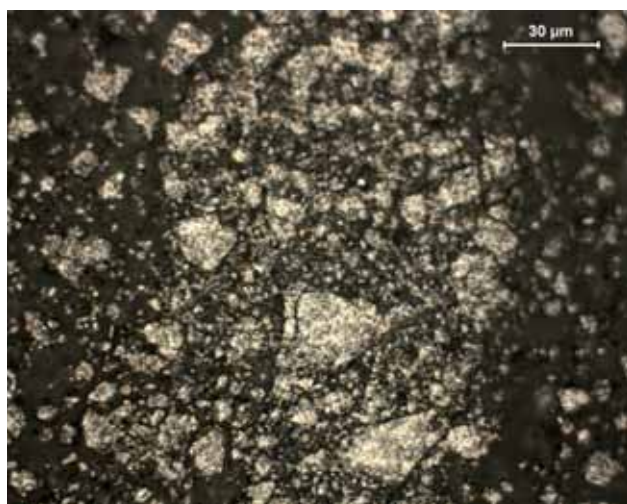


Fig. 6. Microstructure of composite powder obtained in the MA process for aluminium chips + 2 wt% Zr + 40 wt% SiC.

Rys. 6. Mikrostruktura proszku kompozytowego wytworzonego w procesie mechanicznej syntezy w przypadku wiórów aluminiowych + 2 % wag. Zr + 40 % wag. SiC.

ed $1.2 \mu\text{m}^2$. As shown in Figs. 4 and 5 the average particle area depends on the agglomerate fraction, and the fraction lower than $0.5 \mu\text{m}^2$ is observed.

On the basis of the results of the microstructure studies we can observe that after the MA process, SiC reinforce-

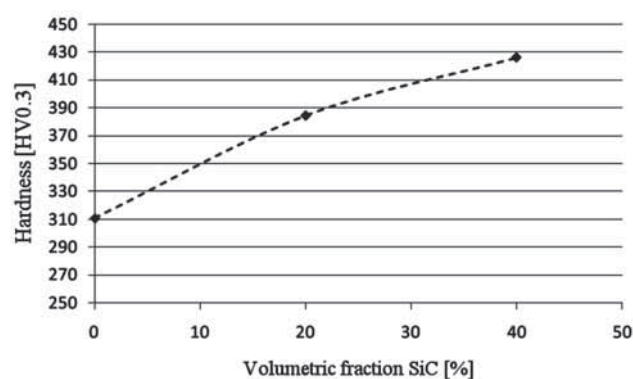


Fig. 7. The influence of volumetric fraction of SiC on Vickers hardness of sintered samples at small loads ($\text{HV}_{0.3}$).

Rys. 7. Wpływ udziału objętościowego SiC na twardość spiekanych próbek określoną przy małych obciążeniach ($\text{HV}_{0.3}$).

ment is located around and inside the aluminium particles which confirms the correct course of the process. The average area, measured by image analysis, is $0.6 \mu\text{m}^2$.

Following Fig. 6, we can observe that after the MA process, hard SiC reinforcement is located inside the plastic aluminium particles which confirms the correct course of the process.

Fig. 7 reports how hardness of the sintered samples elevates with the increasing of SiC content. The results up to

426 HV_{0.3} are obtained for the composites with 40 wt% SiC after sintering process, whereas aluminium chips milled without additional components and sintered show the value of 310 HV_{0.3}.

4. Conclusions

Mechanical alloying procedure showed a new concept of producing a composite powder starting from aluminium chips with the addition of Zr, and SiC reinforcement.

Extension of milling time of aluminium chips with the addition of Zr and SiC in the MA process allowed us to obtain the fine-grained composite powder.

Hardness at small loads of aluminium matrix composites grows with the increasing of SiC volumetric fraction.

Acknowledgements

Experiments were carried out under the Project: Działalność Statutowa ITS No 6709/CBM that was supported by the MNiSW in Warsaw and in the frame of Italian-Polish Intergovernmental Agreement for Cooperation in the domain of Science and Technology.

The authors wishes to acknowledge the support and helpful advice of Prof. M. Rosso, Dr M. Actis Grande, Dr A. Zago as well as technical staff of Mechanical Laboratory of Alesandria Campus of the Politecnico di Torino for their contribution to materials testing.

References

- [1] German R.M: Powder Metallurgy Science, Princeton, New Jersey: American Powder Industries Federation, (1984).
- [2] Kumpfert J., Staniek G., Kleinekathöfer W., Thumann M.: „Mechanical Alloying of Elevated Temperature Al-Alloys”, Proc. ASM International Conference “Structural Applications of Mechanical Alloying”, ed. by F. M. Froes, J. J. de Barbadello, ASM Int., Metals Park, Ohio 44037, (1990).
- [3] Harris S.J.: Developments in particulate and short fibre composites in new light alloys, AGARD Lecture Series, 144, (1990), 1–21.
- [4] Christman T., Needleman A., Suresh S.: „An experimental and numerical study of deformation in metal–ceramic composites”, Acta Metall., 37, (1989), 3029–50.
- [5] Aylor D.M.: Metals handbook V-13, ASM Metals Park (OH), (1982), 859–863.
- [6] Senthilkumar J., Balasubramanian M., Balasubramanian V.: „Effect of Metallurgical Factors on Corrosion Behavior of Al-SiCp Composites Fabricated by Powder Metallurgy”, Journal of Reinforced Plastics and Composites, 28, 9, (2009), 1087-1098.
- [7] Thummler F., Oberacker R.: Introduction to Powder Metallurgy, The Institute of Materials, (1994).
- [8] Tandler M., Šuštaršič B., Vehovar L., Torkar M.: „Corrosion of Al/SiC Metal-Matrix Composites. Korozija Kompozitov Al/SiC”, Materiali In Tehnologije, 34, (2000), 353.
- [9] Nowacki J.: „Spiekane metale i kompozyty z osnową metaliczną”, WNT, Warszawa, (2005).
- [10] Ogel B, Gurbu R.: „Microstructural characterization and tensile properties of hot pressed Al-SiC composites prepared from pure Al and Cu powders”, Mater. Sci. Eng. A, 301, (2001), 213-220.
- [11] Suśniak M., Dutkiewicz J., Pietrzak K., Karwan-Baczewska J.: „The influence of mechanical alloying on the properties of fragmentation of aluminium powder obtained from recycled material”- accepted on 6th International Conference on Engineering and Education 2010, 3-5 November, 2010, Białka Tatrzańska.
- [12] PN - EN 24497, July 1999.
- [13] PN - EN ISO 3927, July 2003.
- [14] PN - EN ISO 3953, October, 1997.
- [15] PN-82 H-04935, July 1982.



Received 6 December 2010; accepted 10 January 2011