A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 58 2013 Issue 2
DOI: 10.2478/amm-2013-0014

M. SUŚNIAK*, J. KARWAN-BACZEWSKA*, J. DUTKIEWICZ**, M. ACTIS GRANDE***, M. ROSSO***

STRUCTURE INVESTIGATION OF BALL MILLED COMPOSITE POWDER BASED ON AlSi5Cu2 ALLOY CHIPS MODIFIED BY SiC PARTICLES

BADANIA STRUKTURALNE MIELONEGO PROSZKU KOMPOZYTOWEGO NA OSNOWIE WIÓR STOPU ALUMINIUM MODYFIKOWANYCH CZĄSTKAMI SiC

The paper is focused on the processing of aluminum alloy chips using powder metallurgy. Chips obtained from recycled AlSi5Cu2 alloy were ball milled with the addition of silicon carbide powder with an average size of 2μm. Mechanical alloying process was employed to obtain homogeneous composite powder. The effect of processing time (0 - 40h) on the homogeneity of the system was evaluated, as well as a detailed study of the microstructure of AlSi5Cu2 aluminum chips and SiC particles during MA was carried out. Addition of silicon carbide (10, 20wt%) to recycled aluminium chips and application of MA lead to fragmentation of the homogeneous composite powder down to particle size of about 3μm and spheroidization. The addition of hard SiC particles caused reinforcement and reduced the milling time. Higher content of silicon carbide and longer processing time allowed to obtain AlSi5Cu2/SiC powders with microhardness ~500$HV_{0.025}$. The results of MA were investigated with SEM, EDS, LOM, XRD and showed that relatively homogeneous distribution of SiC reinforcements in the matrix as well as grain refinement of aluminum solid solution down to 50nm can be obtained after 40h of processing.

Keywords: AlSiCu alloy chips, Recycling, Powder metallurgy, Mechanical alloying, Al-SiC composite powder

W artykule przedstawiono metodę otrzymywania proszku kompozytowego na osnowie wiór stopu aluminium AlSi5Cu2 pochodzących z recyklingu z dodatkiem węglika krzemu (SiC-α, 2μm). Określono wpływ czasu mielenia oraz dodatku SiC na mikrostrukturę i właściwości proszku kompozytowego.

Dodatek SiC (10, 20%mas) do wiór stopu aluminium i zastosowanie mechanicznej syntezy pozwala na otrzymanie jednorodnego i drobnoziarnistego proszku o wielkości 3μm i kształcie zbliżonym do sferycznego. Dodatek twardych cząstek SiC powoduje umocnienie proszku oraz skrócenie czasu mielenia. Dodatek SiC i zastosowanie 40h mielenia pozwala otrzymać cząstki proszku o mikrotwardości około 500 $HV_{0,025}$.

Badania proszku kompozytowego przeprowadzone na mikroskopie optycznym, SEM oraz TEM potwierdziły jednorodne rozmieszczenie cząstek SiC w osnowie oraz zmniejszenie wielkości ziarna do 50nm po 40 godzinach procesu.

1. Introduction

Mechanical Alloying (MA) is frequently used for the fabrication of Metal Matrix Composites (MMCs). This fabrication procedure allows to obtain an acceptable uniform dispersion of reinforcing particles in a metallic matrix, with higher specific strength, as compared to conventional composite materials such as casting composites. By MA method it is possible to produce materials with tailored properties for specific applications, by combining the characteristics of reinforcement and metallic matrix [1, 2]. MA can possibly be applied to aluminium scraps which are very difficult to be recycled. Most of them are used in industry, mainly in foundry operations, allowing high tolerances when selecting the chemical composition. However, that forming technique causes a high environmental pollution and forms other scraps in the final stage of processing of generated elements [3, 4]. Therefore alternative processes, more secure and efficient, need to be investigated. Aluminum alloy chips have been used in powder metallurgy (PM), starting from recycled material, milled or cut, adding reinforcement particles and then cold pressed, hot extruded, or hot pressed [4-6]. The use of recycled material directly in a form of chips, without any other additions, shaped by cold or hot extrusion and plastic consolidation additional operation has also been taken into account [7].

In this work the microstructural changes occurring in aluminum alloy chips and the effect of MA on the distribution of SiC reinforcing additions as well as their structure and mechanical properties was investigated.

* AGH- UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF NON-FERROUS METAL, AL. A. MICKIEWICZA 30, 30-059 KRAKÓW, POLAND
** POLISH ACADEMY OF SCIENCE, INSTITUTE OF METALLURGY AND MATERIALS ENGINEERING, POLAND
*** POLITECNICO DI TORINO, DEPARTMENT OF APPLIED SCIENCE AND TECHNOLOGY, ITALY

2. Experimental procedure

Aluminum alloy chips obtained from recycled AlSi5Cu2 aluminium alloy were used as the matrix. Figure 1a shows that aluminum alloy chips have a very irregular shape. The morphology is a direct result of the production procedure. In fact, as a consequence of machining and turning processes, the chips are in the form of rounded shaped debris. Silicon carbide (SiC) particles (of 2.0μm average particle size) were used as reinforcement phase. The morphology of the SiC particles is angular with sharp edges as a result of crushing and grinding of SiC lumps that occur during the fabrication methods (Fig.1b).

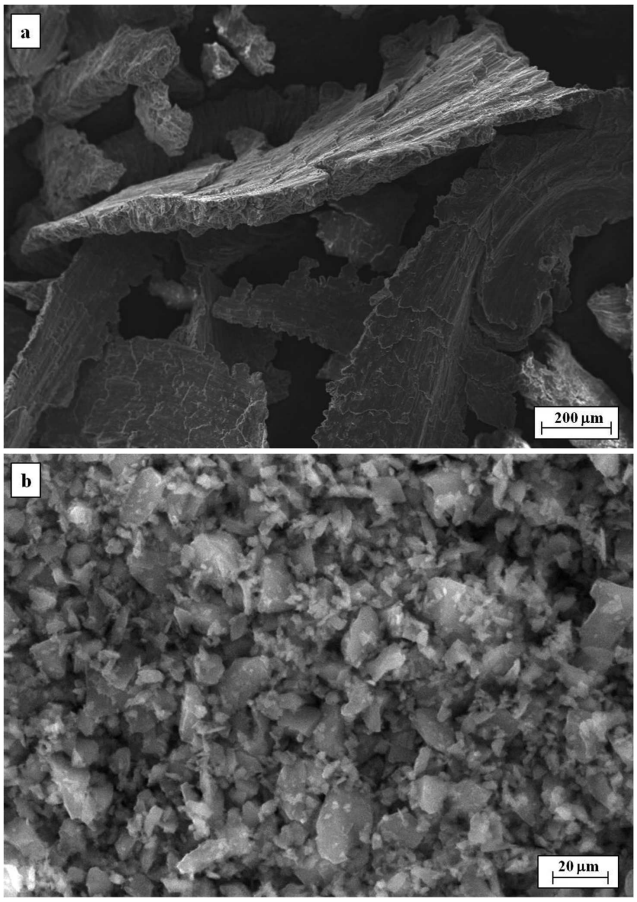


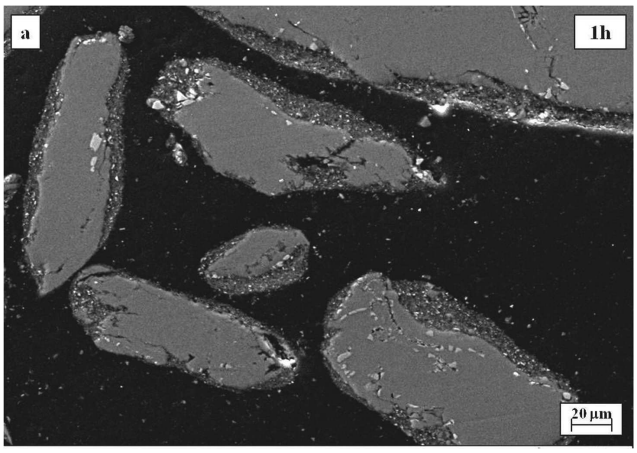
Fig. 1. SEM micrographs of: (a) aluminum chips derived from recycling of AlSi5Cu2 alloy; (b) SiC particles used as reinforcements

AlSi5Cu2-SiC powder mixtures (10, 20 wt% SiC) were prepared by 40 hours MA. In order to get rid of accumulated grease and stress generated during the machining, aluminum chips were annealed for 3 hours at 500°C, and then were cleaned in alcohol. The powder blends were mixed in a high-energy ball mill (Fritsch, Pulverisette 5/4) for up to 40h. The ball's/powder weight ratio was 10:1 and the rotation speed was 200 rpm. 1% by weight of $(CH_3(CH_2)_{16}COOH)$ stearic acid was added as process control agent (PCA). Handling and milling steps were performed under a high purity argon atmosphere in a glove box. AlSi5Cu2 aluminum alloy chips (without addition of SiC particles) were also processed according to the same procedure for comparison. At successive MA time, a small amount of powder was collected for the observation of microstructure, distribution of particle size and microhardness measurements.

The composite powders were embedded in resin and polished for scanning electron microscopy (SEM) observation. Secondary electron and backscattered electron images were taken for evaluating microstructural features such us porosity, particle size and distribution of SiC phase. SEM analysis were also made for the initial powder in order to observe shape evolution during milling. Light microscopy was applied for evaluating the distribution of SiC particles inside of the grit as well as for measurement of the particle average size. The evolution of morphologies and microstructure inside powder particles were also observed by SEM on the polished metallographic specimens. The transmission electron microscopy (TEM) was applied using Philips CM20 electron microscope using samples as thin slices of powders cut using Leica microtome. Vickers microhardness tests were performed using Innovatest hardness testing instrument, with 25g load and dwell time of 15s.

3. Results and Discussion

Figure 2 shows development of the microstructure of AlSi5Cu2+10wt%SiC powder particles during MA; at the beginning of the process AlSi5Cu2 chips are rimmed by a surface layer of SiC particles (Fig. 2a). That effect is related to a large size differences between the matrix and the reinforcement powder and is independent on the amount of SiC. Since the size of the chips tends to decrease after MA, it is clear that during the process particles harden and fracture. Cracks form on the interfaces of aluminum matrix and intermetallics particles: hard ceramics penetrate into the ductile matrix, leading to fast particle breakdown, forming a stratified structure (Fig. 2b, second stage of MA). An increase of milling time leads to a composite powder with plastic matrix and homogeneously dispersed hard particles (Fig. 2c; final stage of MA).



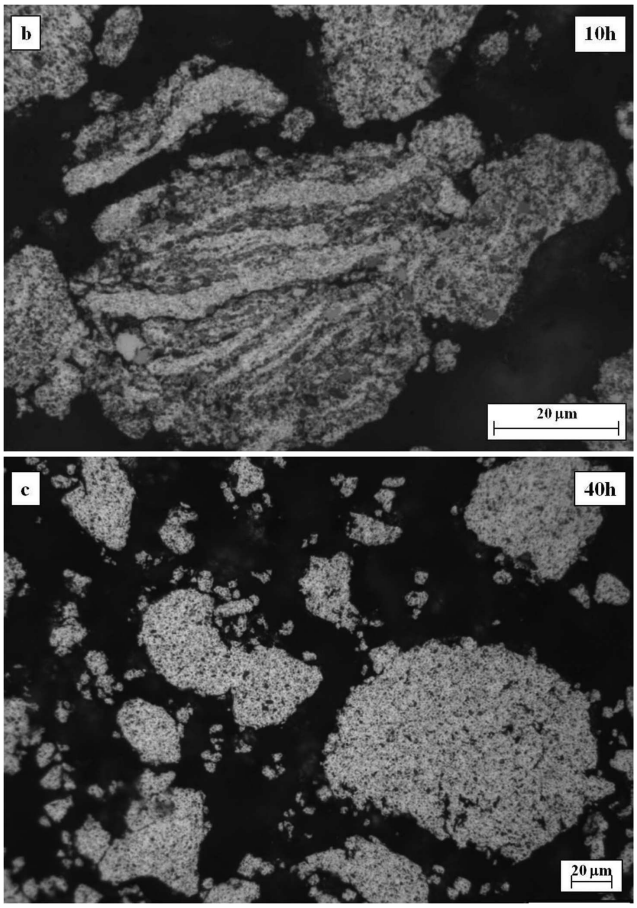
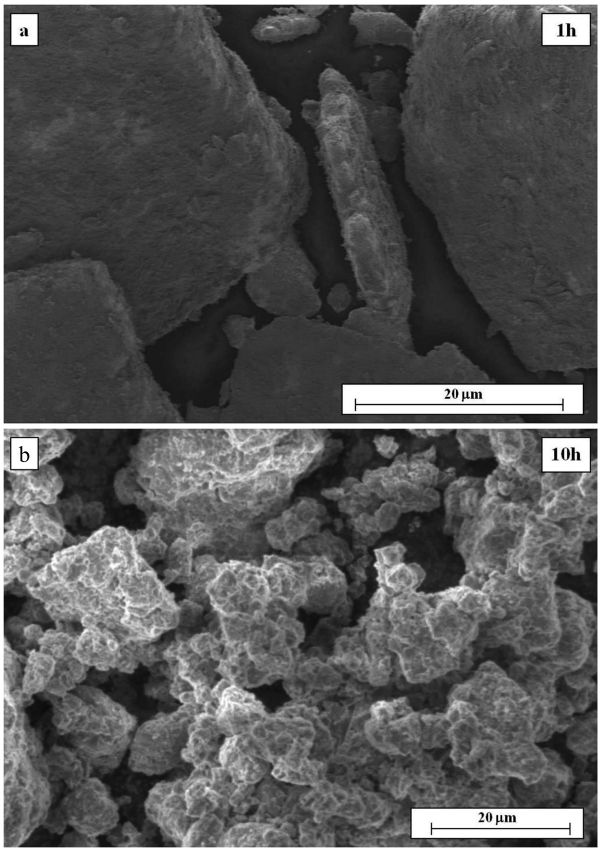


Fig. 2. The distribution of SiC particles in the matrix mixed blends (AlSi5Cu2+10wt%SiC): (a) after 1 h of MA (SEM); (b) after 10 h of MA (LOM); (c) after 40 h of MA (LOM)



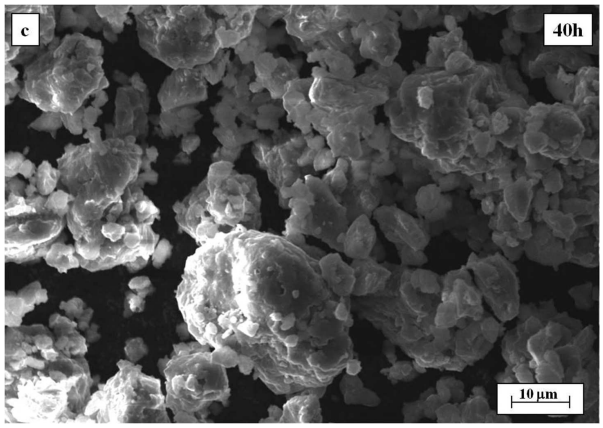
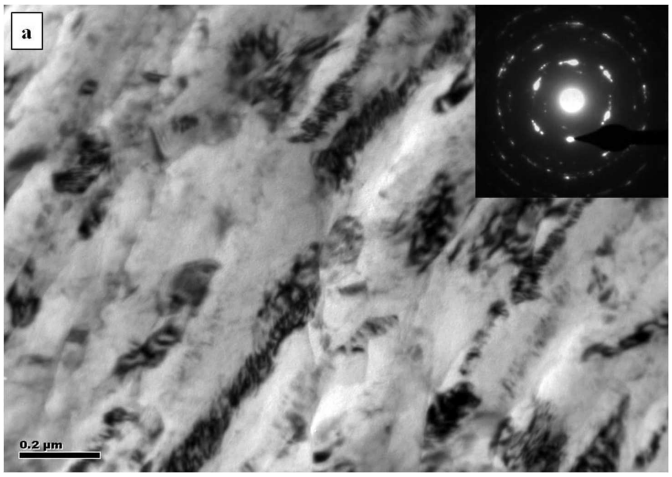


Fig. 3. (a – c) SEM micrographs of composite powders of composition AlSi5Cu2+20wt%SiC after different milling time

As discussed in [8] composition with 20 wt% SiC provides a faster response to MA than composition with 10 wt% SiC. In the former case, five hours of MA allow to obtain composite powder with homogenous distribution of SiC. Composition with lower amount of SiC needs 10 hour of milling to obtain a similar result.

Figure 4 shows TEM micrographs and Selected Area Diffraction Patterns (SADP) as an insert from the powder after 40 hours of milling from the milled chips without additions (4a) and from chips milled with 20wt% SiC (4b). One can see in Fig. 4a elongated subgrains of length above 1 μm formed during milling by multiple ball hitting and welding. The SADP shows spreading of reflections along the Debye-Scherrer rings due to misorientation of subgrains. Slightly different micrograph can be seen from the chips milled with SiC powder. The welded elongated particles can be seen with a fine rather spherical subrains of size below 50 nm in accordance with X-ray diffraction where significant broadening of α(Al) reflections can be seen. SADP shows reflections diffused along rings characteristic for nanomaterials. The SiC particles can be distinguished as no defects can be seen there. They are located between the welded particles.



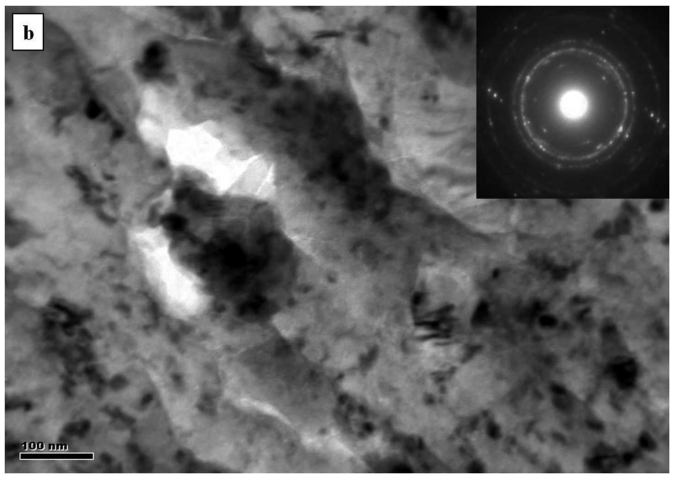


Fig. 4. a) TEM micrograph from the 40 hours milled chips from the alloy AlSi5Cu2 and SADP as an insert; b) TEM micrograph from the 40 hours milled chips of the alloy AlSi5Cu2 with 20wt% SiC and SADP as an insert

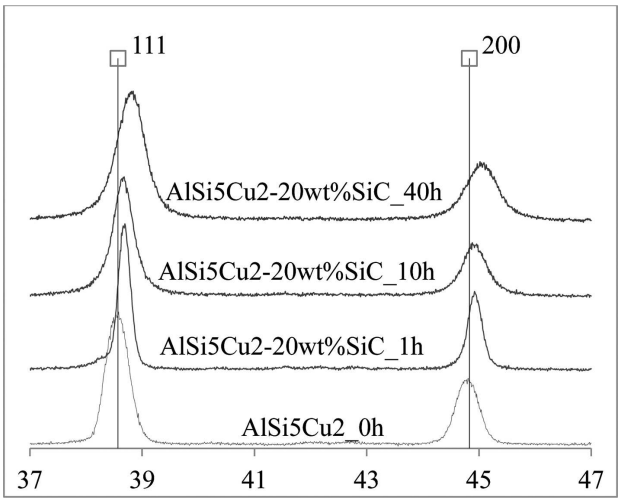


Fig. 5. X-ray diffraction of (111) and (200) of AlSi5Cu2 (1) as received powder; 2-4 MA AlSi5Cu2-20wt%SiC powder in different time of milling

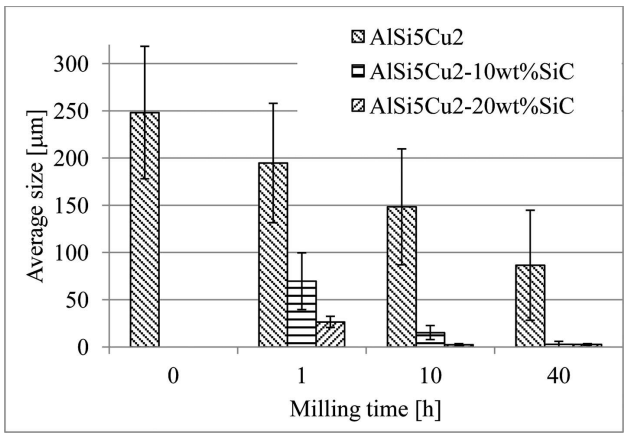


Fig. 6. Average size of powder as function of milling time for 0, 10 and 20wt% SiC

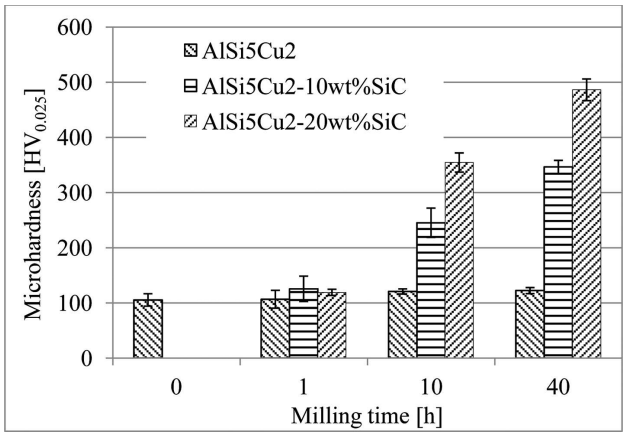


Fig. 7. Microhardness as function of milling time for 0, 10 and 20wt% SiC

4. Conclusions

1. Addition of silicon carbide (10, 20 wt%) to recycled AlSi5Cu2 aluminum alloy chips and subjected to 40 hours of MA lead to fragmentation chips down to particle size of about 3 μm and granular shape. The milled chips show elongated subgrains of length above 1 μm and thickness of about 0.1 μm. The additions of SiC causes significant refinement of aluminum solid solution grains below 50 nm.
2. The addition of hard particles caused reinforcement of powder particles and reduced the milling time. The chips milled 40 hours without the addition of SiC resulted softer (~120 $HV_{0.025}$) as compared to particles mechanically alloyed with SiC which microhardness is ~500 $HV_{0.025}$.
3. Analysis of the results of X-ray diffraction (XRD) confirmed a change in crystallites size measured during TEM studies of the structure of powders during the mechanical alloying process as well as change of the lattice parameter due to formation of a metastable solid solution as an effect of mechanical alloying.

Acknowledgements

Experiments were carried out under the statutory activity at AGH in Cracow (11.11.180.510.) as well as "Doctus – Małopolski fundusz stypendialny dla doktorantów" co-financed by the European Union under the European Social Fund.

Some parts of work was supported in the frame of Italian-Polish Intergovernmental Agreement for Cooperation in the domain of Science and Technology and the authors are grateful to ing. D. Pezzini and to the technical staff of the Department of Applied Science and Technology – Alessandria Campus for their contribution to materials testing.

REFERENCES

[1] N. Z h a o, P. N a s h, X. Y a n g, The effect of mechanical alloying on SiC distribution and properties of 6061 aluminum composite, Journal of Materials Processing Technology **170**, 586-592 (2005).

[2] C. Suryanarayana, Mechanical alloying and milling, Progress in Materials Science **46**, 1-184 (2001).
[3] M. Samuel, A new technique for recycling aluminum scrap, Journal of Materials Processing Technology **135**, 117-124 (2003).
[4] J.B. Fogagnolo, E.M. Ruiz-Navas, M.A. Simón, M.A. Martinez, Recycling of aluminum matrix composite chips by pressing and hot extrusion, Journal of Materials Processing Technology **143-144**, 792-795 (2003).
[5] J. Gronostajski, A. Matuszak, The recycling of metals by plastic deformation: an example of recycling of aluminum and its alloys chips, Journal of Materials Processing Technology **92-93**, 35-41 (1999).
[6] W. Chmura, J. Gronostajski, Mechanical and tribological properties of aluminum-base composites produced by recycling of chips, Journal of Materials Processing Technology **106**, 23-27 (2000).
[7] H. Dybiec, K. Pieła, J. Piotrowska, Recykling wiórów AlCu3,8 metodą konsolidacji plastycznej, Rudy i Metale Nieżelazne **54**, 676-684 (2009).
[8] M. Suśniak, J. Karwan-Baczewska, J. Dutkiewicz, M. Actis Grande, Structural analysis of composite powders obtained from recycled material, Inżynieria Materiałowa **186**, 86-89 (2012).

Received: 20 January 2013.