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 60 2015 Issue 2
DOI: 10.1515/amm-2015-0164

A. SIMON*,♯, D. LIPUSZ*, P. BAUMLI**, P. BALINT**, G. KAPTAY**, G. GERGELY**, A. SFIKAS***, A. LEKATOU***, A. KARANTZALIS***, Z. GACSI**

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-WC COMPOSITES

MIKROSTRUKTURA I WŁAŚCIWOŚCI MECHANICZNE KOMPOZYTÓW Al-WC

The scope of the research work is the production and characterization of Al matrix composites reinforced with WC ceramic nanoparticles. The synthesis process was powder metallurgy. The produced composites were examined as far as their microstructure and mechanical properties (resistance to wear, micro/macrohardness). Intermetallic phases ($Al_{12}W$ and Al_2Cu) were identified in the microstrucutre. Al_4C_3 was not detected in the composites. Adding more than 5 wt% WC to the aluminum, microhardness and wear resistance exceed the values of Al alloy. Composites having weak interface bond performed the highest wear rate.

Keywords: Composite, aluminum, WC, microstructure, mechanical properties

1. Introduction

The constant desire of aerospace and automotive industries to enhance the performance is continually driving the development of improved high-performance structural materials. In recent years, MMCs have emerged as a promising class of materials. Several challenges must be overcome in order to enhance the engineering usage of MMCs. Compared to the conventional aluminum alloys, particle reinforced aluminum matrix composites (Al-MMCp) possess high-specific elastic modulus and strength, light weight, good wear resistance and excellent properties at elevated temperature [1-3]. Because of these excellent mechanical properties, particle reinforced aluminum matrix composites have been widely used in aerospace and infrastructure industries.

The main methods for producing metal matrix composites are casting, powder metallurgy, spray atomization or disintegrated melt deposition technique. During composite molding, the ceramic particles are incorporated into the melted aluminum by continuous stirring. In this method, the particle – melted metal suspension is protected, for example by inert argon and sulfur hexafluoride (Ar-SF6) gas mixture. The continuous stirring ensures homogenous distribution of the particles [4]. The high ratis of the particles in the molten aluminum results high viscosity of the melt [5]. The particles may coagulate [6] because the melted aluminum does not wet the most common reinforcing materials high (e.g. SiC, Al2O3, graphite) [7]. Perfect wettability of carbon or graphite by aluminum has been achieved only recently [8].

Powder metallurgy (PM) is a widely used method for producing metal matrix composites. Pressing into desired shape of form (compacting), and then heating the compressed material under a controlled atmosphere to bond the material (sintering) is a powder metallurgy process to blend fine powdered materials. As processing takes place in solid state, it minimizes the reaction between the constituents of the composite. Although PM allows one to produce components with complex geometries in bulk, there are some disadvantages associated with conventional powder metallurgy such as the segregation of the reinforcing particles between the metal matrix particles and porosity. This often leads to the degradation of the mechanical properties. These problems become more important when the difference in the particle size between the reinforcement and the matrix alloy powders is significant or when the volume fraction of the reinforcement is high. Compacting and sintering are often combined in one step during hot pressing [9]. Several composites can be prepared by powder metallurgy, for example SiC_p/Al [10], Al_2O_3/Al [11] MgO/Al [12] WC/Ni [13].

Al based metal matrix composites were in the main focus of several research groups. One of the main research topic is the WC reinforced Al matrix composite.

Wu Yuying et al. [14] prepared an Al/WC composite coating on Al-12.6Si alloys by high energy milling. After the milling, the composite coating was heat treated. They experienced that under certain conditions, Al powder reacted with WC, and as a result, the strength of the bond between Al and WC increased.

* INSITUTE OF CERAMICS AND POLYMER ENGINEERING, MISKOLC, HUNGARY
** INSTITUTE OF PHYSICAL METALLURGY, METALFORMING AND NANOTECHNOLOGY, MISKOLC, HUNGARY
*** UNIVERSITY OF IOANNINA, IOANNINA, GREECE
♯ Corresponding author: femandi@uni-miskolc.hu

C.Y. Liu [15] has successfully produced aluminum metal matrix composites reinforced with tungsten carbide (WC) particles by warm accumulative roll bonding (ARB). The composite microstructure shows excellent WC particle distribution in the Al matrices, and no reaction was observed between Al and WC. Compared with the ARBed 1060-Al, the Al/WC composites show a higher number of dislocations, as suggested by the introduction of WC particles. The tensile, hardness, and wear properties of the Al/WC composites were also determined.

In this research, the aim was to characterize the microstructure (density, porosity and the reinforcing phase distribution through optical microscopy and SEM), the matrix-ceramic reinforcement interface and some mechanical properties (wear resistance, hardness).

2. Experimental

In this study, Al-WC composites were produced via powder metallurgical route. Al-Cu alloy powder (ECKA Alumix 123) was used as matrix material, which was given by ECKA Granules Germany GmbH. The WC powder – as reinforcement phase – was provided by Alfa Aesar. Al powder has a 4.5 wt% Cu, 0.7wt% Si and 0.5wt% Mg as alloying elements and 1.5 wt% lubricant. The ceramic powder contains 99.5 wt% tungsten-carbide. Mean particle sizes of the powders are 24 μm for the Al-Cu alloy and 1 μm for the WC powder. The Al-powder contains rounded, elongated and spherical particles. More information is available in [16, 19].

TABLE 1
Experimental composition of the composites

	WC [wt%]	Mixing time [min]
Al	0	-
Al5WC-15	5	15
Al10WC-15	10	
Al15WC-15	15	
Al5WC-30	5	30
Al10WC-30	10	
Al15WC-30	15	

The experimental parameters are summarized in Table 1. Composite mixtures containing 0, 5, 10 and 15 wt% WC as reinforcement phase were prepared. Each mixture was homogenized in a Retsch PM400 planetary ball mill for 15 or 30 minutes in order to achieve more uniform reinforcement distribution. After homogenization, powder mixtures were cold pressed. Cylindrical specimens with a 1 cm diameter were pressed at 500 MPa, and then sintered under nitrogen atmosphere at 580°C for 20 minutes. The heating rate was 10°C/min. Each sample was held at a temperature of 400°C for 20 min in order to remove lubricant. Then they were heated further to the sintering temperature and held at this temperature for 20 min. Sintering temperature was set at around the solidus temperature in order to maintain liquid phase sintering.

Density, porosity, hardness and wear properties of the composites were determined. Densities of the sintered samples were measured by Archimedes' principle immersion method. Samples were weighed at room temperature, in open air atmosphere, than they were immersed in water to calculate real (ρ) and apparent densities (ρ_{app}) by using the following equations:

$$\rho = \frac{W_a}{W_a - W_{im}} \cdot \rho_w \tag{1}$$

$$\rho_{app} = \frac{W_a}{W_{sp} - W_{im}} \cdot \rho_w \tag{2}$$

where W_a is the weight of sample in air, W_{im} is the weight of the immersed sample, W_{sp} is the weight of the sample which has water filled surface porosity, and finally ρ_w is the density of water at room temperature (ρ_w =1 gcm^{-3}). Mean size of the pores was measured on the micrographs of the samples with Leica Qwin image analyzer.

Microstructures of the samples were observed with Zeiss Axioimager M1m optical and 1830I Amray scanning electron microscope. X-ray diffraction measurements were also carried out using Philips PW1830 equipment (CuKα, 40 kV, 30 mA, 2θ range: 20-90°, step size: 0.05°) to identify the phases in the composites.

Hardness was measured using a Otto-Wolpert Werke-type (Dia Testor 2Rc) hardness tester with a load of 50 N (HV5) and a dwell time of 10 s. Microhardness measurements were performed using Mitutoyo MVK-H1 equipment, with a load of 100 g and a dwell time of 10 s. Three indentations were made for each sample.

Wear test were carried out using a pin-on-disc tester, under a load of 700 g, 100 rpm and dwell time of 10 minutes.

3. Results and discussion

Figure 1. shows the micrographs of the mixtured Al15WC-30 composite powder. The constituents of the composite are distinguished well. The Al alloy consist of two phases, one contains mainly Al while the dendrite grains contains mainly Cu. One can conclude from the micrographs, that even in this case the mixing time (30 min) was not enough to maintain a homogenous reinforcement distribution. As the particle size ratio (PSR, the ratio of the matrix and the reinforcement particle size) is quite high, we had to increase the mixing time next time.

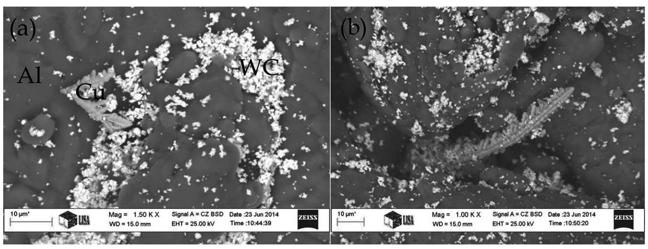

Fig. 1. SEM micrographs of the Al15WC-30 composite powder in a) 1500x b) 1000x magnification

The micrographs of the sintered samples can be seen in Figure 2. Al-Cu alloy sample has a denser microstructure containing less porosity then the composites. Pores are usually situated along or amongst the Al grains, only some of them is at the matrix-reinforcement interphase. Adding more reinforcement to the Al powder increases the amount and size of the pores. Additionally, increasing the mixing time also seems to result more porosity.

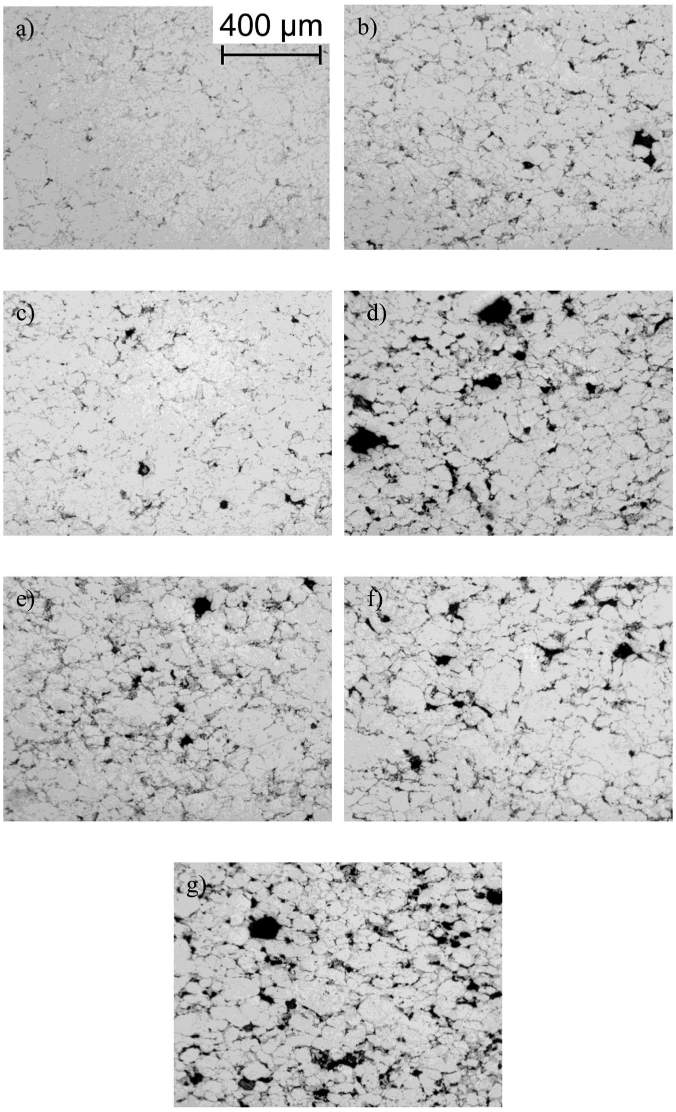

Fig. 2. The micrographs of the sintered samples a) Al b) Al5WC-15 c) Al10WC-15 d) Al15WC-15 e) Al5WC-30 f) Al10WC-30 g) Al15WC-30

Same conclusions can be interpreted from measuring the density and the porosity of the samples by the Archimedes method (see Figures 3-4.). While the apparent density includes both closed and opened porosity, real density shows only closed porosity. As a consequence, the difference between the two densities gives the opened porosity. Adding WC as reinforcement to the Al alloy results a slightly higher density. Increasing the mixing time also results denser structure. As for the pore sizes, the highest values belongs to the composites with 15 wt% WC. Increasing the mixing time is advantageous to homogenize the reinforcement distribution, however at the same time it's also detrimental as it generates more porosity in the powder.

Microstructure of the composites was also investigated by SEM and XRD to identify the intermetallic phases in the composites (Figure 5.). From the results it can be concluded that aluminum reacts with tungsten to form $Al_{12}W$ metastable phase [17-18]. According to [17], the formation of this phase starts at 450°C. $Al_{12}W$ is identified at the matrix-reinforcement interface or close to the WC particles. From the reaction of aluminum and copper, needle-shaped Al_2Cu developed. Al_4C_3, which really impairs the mechanical properties of the composite, was not detected in the samples.

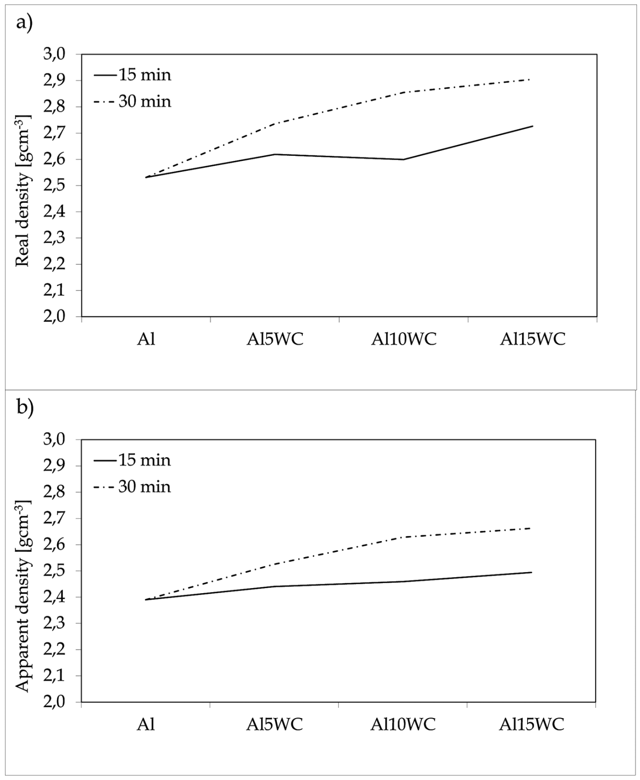

Fig. 3. Real and apparent density of the samples

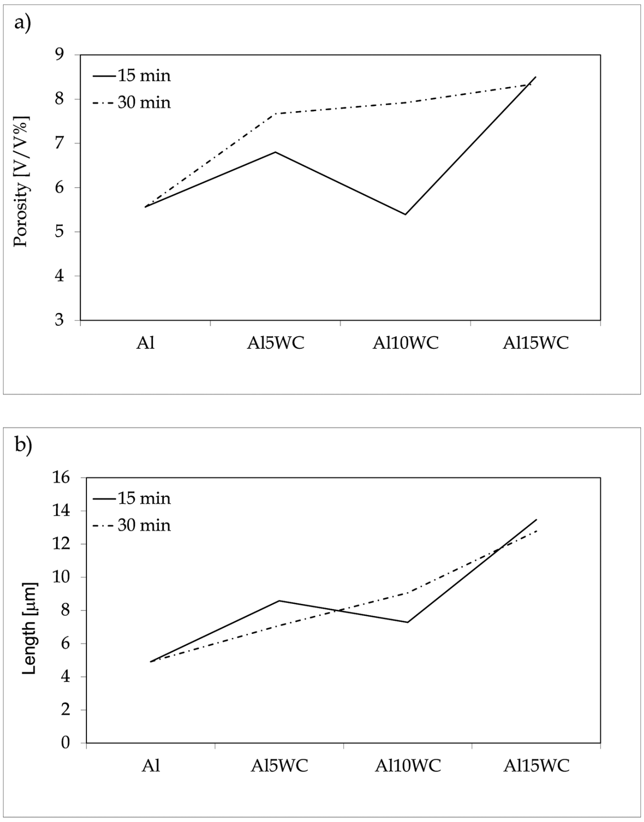

Fig. 4. a) Amount and b) mean size of the pores

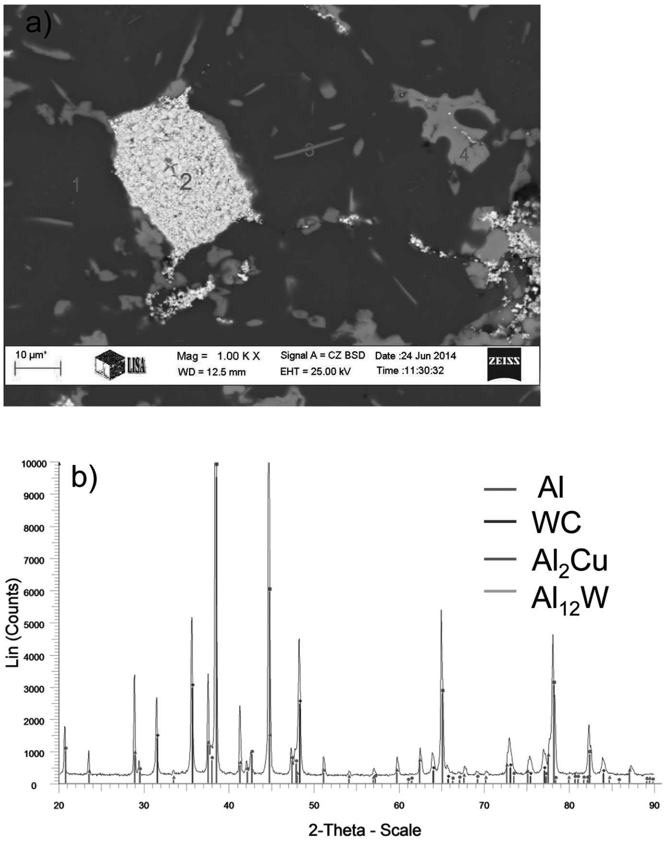

Fig. 5. a) Microstructure (1=Al, 2=WC, 3=Al2Cu, 4=Al12W) and b) XRD pattern of Al15WC-30 composite

Measuring microhardness (Figure 6.) is an easy method to characterize the matrix-reinforcement interphase. A strong, adherent bond between the two phases hinders the movement of the ceramic particles. In case of weak bond, this effect is missed. Consequently, the goodness of interfacial bonding can be concluded by using microhardness tester. Except from the samples containing only 5 wt% WC, all composites had better microhardness than the Al alloy. Al10WC-15, Al10WC-30 and Al15WC-30 composites had quite the same microhardness. Same conclusions can be written for the Vickers hardness test (with a load of 50 N). Composites containing only 5 wt% reinforcement phase don't achieve even the hardness of the Al alloy. However, increasing the amount of WC resulted only the same value as the Al sample had.

Figure 6. shows the results of the wear and hardness tests. Composites with a low microhardness (and a weak interface bond) had the highest wear rate. During the wear test, first the matrix is removed from the surface leaving ceramic particles protuberant. The acting forces break these particles into smaller pieces. In case of weak bonding, the reinforcement particles can even turnout from their original place. Comparing the microhardness and wear rate values, the stronger interfacial bond between the phases leads to lower wear rate.

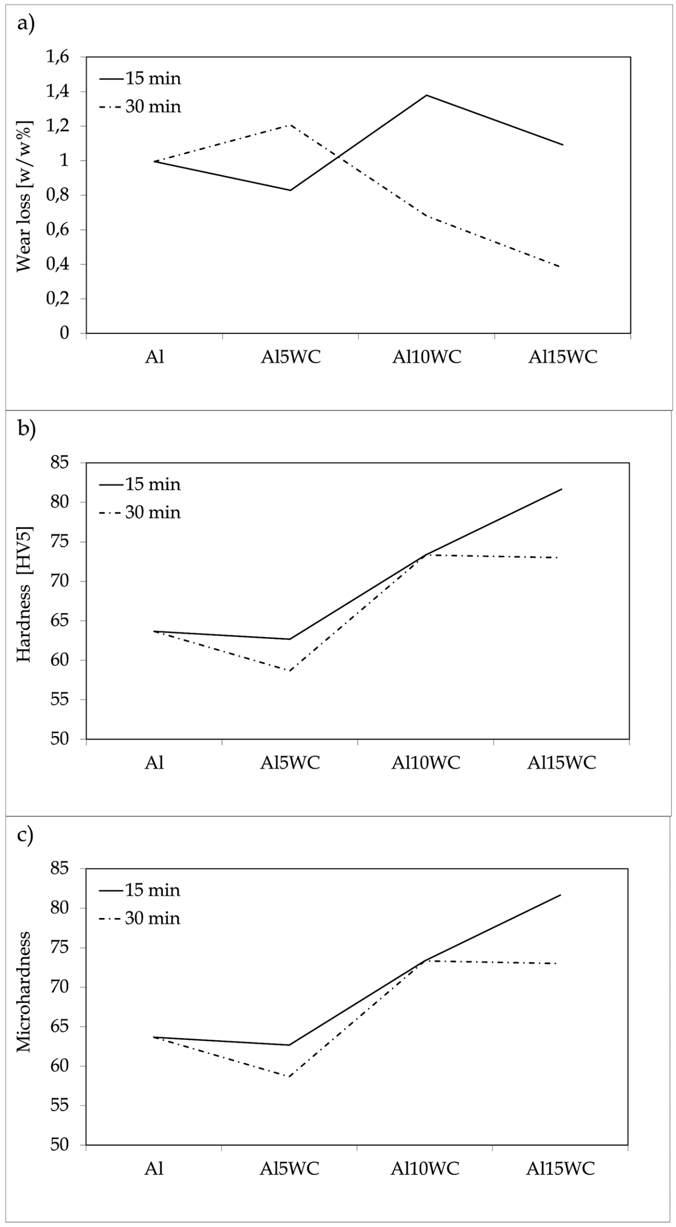

Fig. 6. a) Wear rate b) hardness and c) microhardness of the samples

4. Conclusions

In this study, Al matrix composites reinforced with 0-5-10-15 wt% WC were produced via powder metallurgical route. Density, porosity, hardness and wear properties of the composites were measured to investigate the effect of the reinforcement's amount and the mixing time. In the sintered samples, pores were found mainly along or amongst the Al grains. The amount and size of the pores was also increased by increasing the amount of reinforcement particles and the mixing time. During sintering, $Al_{12}W$ and Al_2Cu develop from the reaction of aluminum with tungsten and copper. Al_4C_3 was not detected in the composites.

Composites containing only 5 wt% reinforcement phase had worse mechanical properties than the unreinforced Al matrix. However, adding more WC to the aluminum, microhardness and wear resistance exceed the values of Al alloy. Comparing the microhardness and wear rate, composites having low microhardness (and weak interface bond) performed the highest wear rate while the stronger the interfacial bond between the phases the lower was the wear rate of the composite.

Acknowledgements

The research work presented in this paper based on the results achieved within the TÁMOP-4.2.1.B-10/2/KONV-2010-0001 project and carried out as part of the TÁMOP-4.2.2.A-11/1/KONV-2012-0019 project in the framework of the New Széchenyi Plan. The research work is also part of the TÉT_10-1-2011-0541 (NSRF 2007-2013) project. The realization of this project is supported by the European Union, co-financed by the European Social Fund. Authors are grateful to Agnes Csurillane and Arpad Kovacs for their kind contribution.

REFERENCES

[1] N. Nemati, R. Khosroshahi, M. Emamy, A. Zolriasatein, Mater Des **32**, 3718-29 (2011).
[2] K.L. Tee, L. Lu, M.O. Lai, Mater Sci Technol **17**, 201-6 (2001).
[3] M.O. Shabani, A. Mazahery, J Mater Sci **46**, 6700-8 (2011).
[4] J.R. Davis (Davis & Associates): Aluminum-Matrix Composites, Aluminum and Aluminum Alloys, ASM Specialty Handbook (1993).
[5] Jun Wang et al., Journal of Processing Technology **136**, 60-63 (2003).
[6] P. Janardan, Scandinavian Journal of Metallurgy **22(5)**, 260-265 (1993).
[7] B. Sarina, T. Kai, A. Kvithyld, T. Engh, M. Tangstad, Trans. Nonferrous Met. Soc. China **22**, 1930-1938 (2012).
[8] P. Baumli, J. Sytchev, G. Kaptay, J Mater Sci **45**, 5177-90 (2010).
[9] M.J. Tan, X. Zhang, Materials Science and Engineering A **244**, 80-85 (1998).
[10] H. Izadi et al., Journal of Materials Processing Technology **213**, 1900-1907 (2013).
[11] Mehdi Rahimian, Nader Parvin, Naser Ehsani, Materials and Design **32**, 1031-1038 (2011).
[12] Hossein Abdizadeh et al., Composites: Part B **56**, 217-221 (2014).
[13] E. Taheri-Nassaj, S.H. Mirhosseini, J. of Materials Proc. Technology **142**, 422-426 (2003).
[14] Wu Yuying et al., Journal of Alloys and Compounds **497**, 139-141 (2010).
[15] C.Y. Liu et al., Materials and Design **43**, 367-372 (2013).
[16] Andrea Simon et al., Materials Science Forum **752**, 48-56 (2013).
[17] R.C. da Silva et al., Nuclear Instruments and Methods in Physics Research Section B Beam Interactions with Materials and Atoms **50(01)**, 423-427 (1990).
[18] Sami Franssila: Introduction to Microfabrication, 2010, Wiley, ISBN 978-0-470-74983-8.

Received: 20 November 2014.