

## EFFECTS OF CU AND SOLUTION HEAT TREATMENT ON THE MICROSTRUCTURE AND HARDNESS OF IN-SITU ALUMINIUM MATRIX COMPOSITE CONTAINING AL<sub>4</sub>SR PHASE

K. Tavighi<sup>1</sup>, M. Emamy<sup>\*1</sup> and A. R. Emami<sup>2</sup>

\* emamy@ut.ac.ir

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<sup>1</sup> School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran.

<sup>2</sup> Young Researchers Club, Science and Research Branch, Islamic Azad University, Tehran, Iran.

**Abstract:** This study was undertaken to investigate the effects of Cu and solution heat treatment on the microstructure and hardness of cast Al-Al<sub>4</sub>Sr metal matrix composite. Different amounts of Cu (0.3, 0.5, 1, 3 and 5 wt.%) were added to the composite. Specimens were heat treated at 500 °C for 4 hours followed by water quenching. Microstructural studies were assessed by the use of optical microscope, scanning electron microscope (SEM) and x-ray diffractometry (XRD). The results showed that addition of 5 wt.% Cu reduces the length of large needle-like Al<sub>4</sub>Sr phase and refines the microstructure. In addition, the presence of Cu-intermetallics increases hardness of the composite. Cu mainly forms  $\theta$  phase which segregates at the grain boundaries. Heat treatment partially dissolves Cu-intermetallics and homogenizes the distribution of  $\theta$  phase in the matrix.

**Keywords:** Metal matrix composite (MMC); Microstructure; Intermetallic; Hardness; Heat treatment.

### 1. INTRODUCTION

Metal matrix composites (MMC) are the new class of materials and are rapidly replacing conventional materials in various engineering applications [1]. The intrinsic advantage of MMCs over the unreinforced alloy is the improvement of mechanical properties, e.g., an improved specific stiffness, strength, and wear resistance and a tolerable coefficient of thermal expansion (CTE) [2], due to addition of the reinforcing material [3-5]. Furthermore, the reinforcing phase in MMC has a higher creep resistance than the matrix at a given temperature, thus improving the creep properties of the composite as compared to the unreinforced matrix [6]. However, these materials suffer from poor ductility, low values of fracture toughness and poor low-cycle fatigue properties [3-5].

Of special interest in this regard are particulate reinforced metal matrix composites (PRMMCs), which possess several additional advantages. Firstly, they offer cost effective manufacturing; particulate forms of reinforcement are much cheaper than long fibres. PRMMCs can also be manufactured by conventional metallurgical processes and secondary processing can be

applied. Secondly, PRMMCs have isotropic properties (not the case for continuously reinforced MMCs). Therefore, they can be used for more general applications. Thirdly, they can be produced in large quantities which are required for structural applications [7].

PRMMCs are promising candidates for a number of aerospace and automotive applications due to their higher specific stiffness, specific strength and better wear resistance. It is well known that the mechanical behavior of this class of materials is significantly affected by their microstructure, such as the Young's modulus of the particle, the particle aspect ratio, the particle volume fraction and size effect, as well as the strain-hardening exponent of the matrix material. During the past two decades, many attempts have been made to explore the relationship between microstructure and deformation behavior in PRMMC [8,9]. Many efforts have been focused on SiC as reinforcements in different aluminium based matrix [10-12].

Recently numerous works have been devoted to Mg<sub>2</sub>Si as reinforcement in aluminium. Mg<sub>2</sub>Si has rather high melting point and intrinsically is hard and brittle. Therefore, Mg<sub>2</sub>Si particles are good candidates as reinforcements in MMCs [13-16].

In Al-Sr binary system,  $\text{Al}_4\text{Sr}$  intermetallic forms in 45 wt.% Sr. This intermetallic has the highest melting point (1034 °C) in comparison with other phases in the system.

This fact encouraged some researchers to examine the properties of such composite which contain  $\text{Al}_4\text{Sr}$  intermetallic as a reinforcing phase in the aluminium matrix.

The aim of this study is focused on investigating the effects of Cu on the structural properties of Al-16% $\text{Al}_4\text{Sr}$  composite and its hardness value.

## 2. EXPERIMENTAL PROCEDURE

In order to prepare MMC ingots, pure commercial Al (>99.8% purity) metal was heated in an electrical resistance furnace (6 Lit. capacity) in a graphite crucible (10 Kg capacity). After heating up to 800 °C, commercially Al-10Sr master alloy was added to the melt in order to form Al-7Sr which contains 16 wt.%  $\text{Al}_4\text{Sr}$  particles approximately. Ingots were cut to small pieces for remelting process. Then, the chopped ingots were remelted in a small furnace and different amounts of Cu (0.3, 0.5, 1, 3 and 5 wt.%) were added to the molten MMC. To ensure complete mixing, the molten alloy was hand stirred gently with a graphite rod for about 1min. Degassing was conducted by using commercially available  $\text{C}_2\text{Cl}_6$  tablets (0.3 wt% of the molten alloy) for about 2 min. After stirring and cleaning off the dross, the molten composite was cast into the prepared cast iron mould to produce the cylindrical specimens (30 mm in diameter and 45 mm high) at room temperature for microstructural studies. After filling the mould, specimens were prepared by sectioning the castings.

Heat treatment experiments were performed in a high accurate temperature controlled electrical resistance furnace ( $\pm 2$  °C). The specimens were solutionized at 500 °C for 4 h followed by quenching in cold water.

Metallographic specimens were polished using standard routine and etched with Keller's reagent for about 17 s at room temperature. Quantitative data from microstructural studies were determined using an optical microscope equipped

with an image analysis system (Clemex Vision Pro. Ver.3.5.025). The microstructural characteristics of the specimens were also examined by scanning electron microscopy (SEM) in Vega ©Tescan.

In order to characterize the phases in the MMC, X-ray diffractometry by the use of PHILIPS X-ray diffractometer was conducted.

Hardness results were obtained by the use of ESEWAY universal hardness tester. Results were prepared and reported by five measurements for each specimens based on Brinell hardness test (30 Kgf) with 2.5 mm stainless steel spherical indenter.

## 3. RESULTS AND DISCUSSION

Fig. 1-a and 1-b demonstrate the typical microstructure of Al- $\text{Al}_4\text{Sr}$  MMC and its XRD pattern, respectively. The microstructure consists of aluminium matrix accompanying with the second intermetallic phase. XRD pattern reveals that the second phase in the composite is  $\text{Al}_4\text{Sr}$  (Fig. 1-b). As it is seen,  $\text{Al}_4\text{Sr}$  particles are large; in addition, the second phase is not uniform which means that thick and thin particles exist in the microstructure. Average length of  $\text{Al}_4\text{Sr}$  phase is 140  $\mu\text{m}$  with the average diameter of 20  $\mu\text{m}$ .

Due to the unsuitable morphology of  $\text{Al}_4\text{Sr}$  particles, it is necessary to refine the phase. Large particles are favoured sites for stress concentration; therefore, refining the microstructure is known as a target of this study.

### 3. 1. Effect of Cu Addition on The composite:

Fig. 2 (a-e) shows the effect of different weight percentages (0.3-5) of Cu on the second phase morphology. Fig. 3 shows length and thickness of  $\text{Al}_4\text{Sr}$  particles in the composite as functions of different contents of Cu. As it can be seen addition of Cu reduces the length of  $\text{Al}_4\text{Sr}$  particles; however, error bars show high deviation. In the other words, in the composites with different percentages of Cu, large particles still remain beside small ones. Large particles surrounded by stress atmosphere resulting in dislocation/particle interactions. Geometrical dislocations will be generated in the interface in

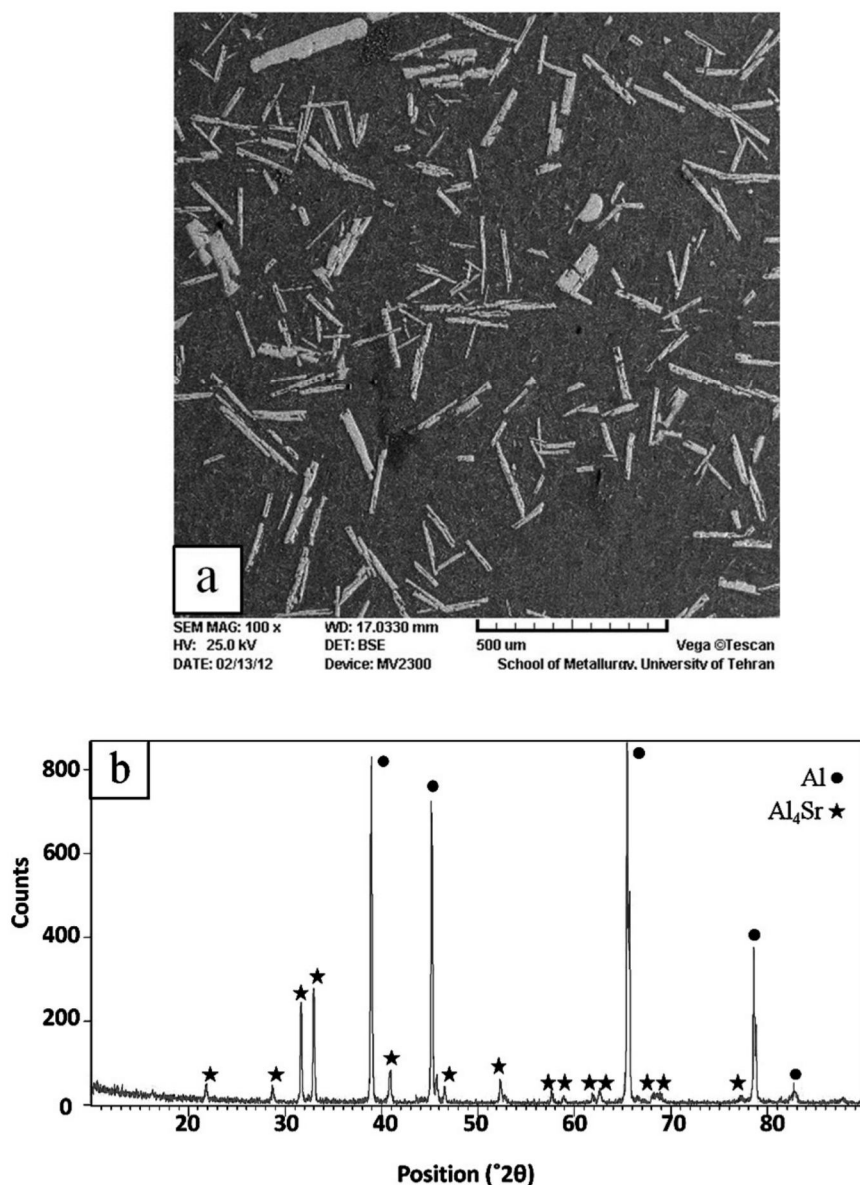
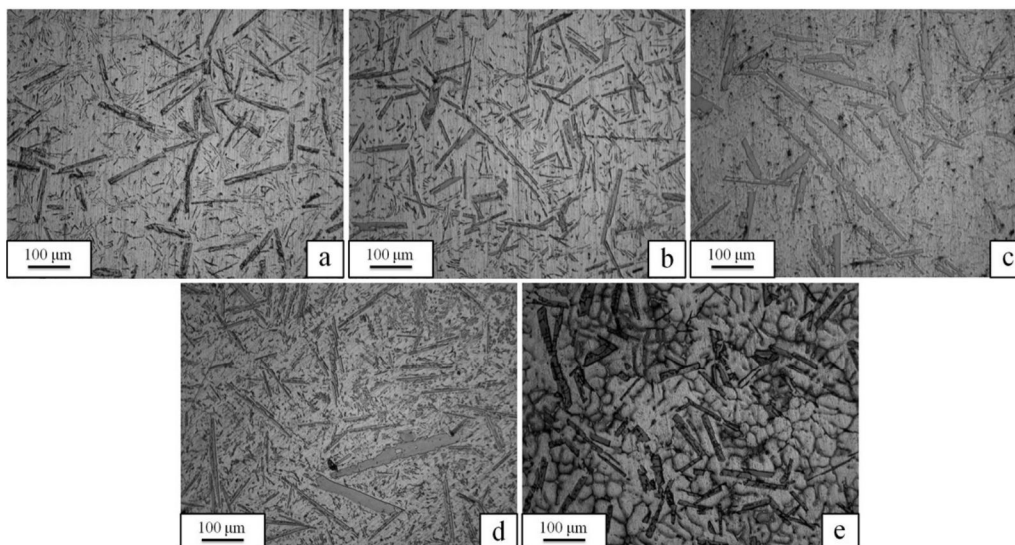


Fig. 1. Al-Al<sub>4</sub>Sr MMC: (a) typical microstructure and (b) XRD pattern.

order to support the contiguity of the material. The amount of dislocation density around large particles is higher in comparison with smaller ones which causes crack nucleation at these sites. The results show another scenario in the case of addition of 5 wt.% of Cu. Fig. 3 shows the least deviation both in length and thickness of second phase particles. It is clear that the length of precipitates reduces from 140 μm to 65 μm. Also from Fig. 2-e, it is obvious that the size and

precipitation mode of Al<sub>4</sub>Sr particles are uniform. Another fact, which is apparent in the composite containing 5 wt.% Cu (Fig. 2-e), is the presence of some Cu-rich intermetallic precipitates at the grain boundaries.

Higher amounts of Cu (>1wt.%) results in the formation of Cu-rich intermetallics, mainly θ-CuAl<sub>2</sub> phase. This intermetallic due to its hardness is able to act as a reinforcement agent in the MMC. Cu-rich intermetallics are pushed

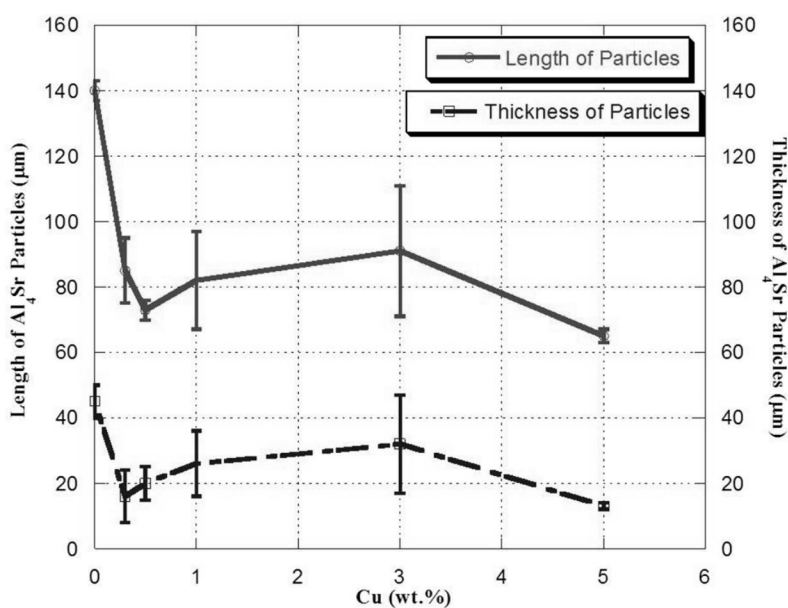


**Fig. 2.** Microstructure of Al-Al<sub>4</sub>Sr MMC containing Cu (wt.%): (a) 0.3, (b) 0.5, (c) 1, (d) 3 and (e) 5.

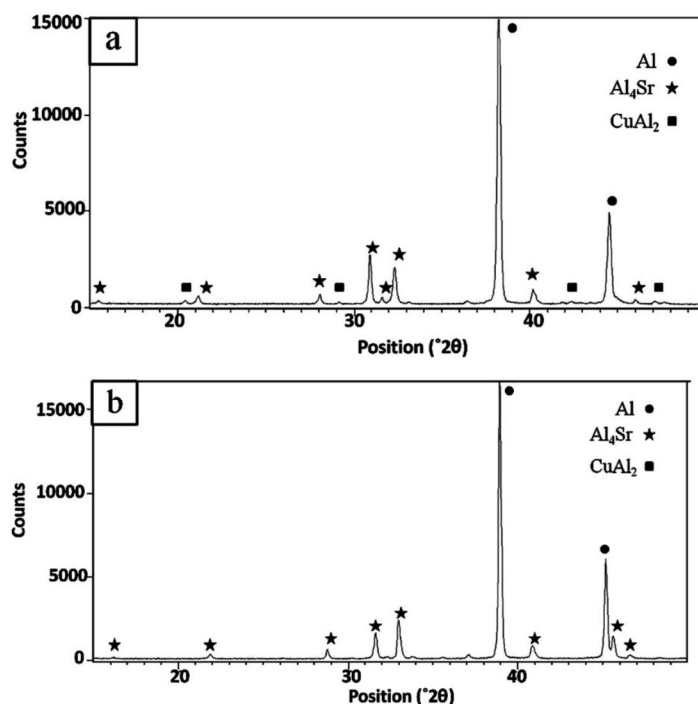
towards grain boundaries at the last stage of solidification. Therefore, grain boundaries, as seen in Fig. 2-e, are recognized as stress-concentrated sites of the system in presence of 5 wt.% of Cu in the MMC which are favored path for both crack initiation and crack propagation [13].

### 3. 2. Effect of Solution Heat Treatment:

As mentioned earlier, Cu-rich intermetallic precipitates are formed at the grain boundaries as a result of segregation. Fig. 4 illustrates the distribution mode of Cu-rich intermetallics before and after heat treatment. As it is seen in the enlarged image of Fig. 4-a, CuAl<sub>2</sub> phase forms at



**Fig. 3.** Length and thickness of Al<sub>4</sub>Sr particles as functions of Cu wt.%.



**Fig. 4.** SEM back-scattered image of the MMC containing 5 wt.% Cu: (a) before solution heat treatment, (b) after solution heat treatment.

the grain boundaries (bright white colour). After solutionizing, the named intermetallics seem to be separated and uniformly distributed in the matrix (Fig. 4-b). It is expected that fine and uniform particles in the matrix encourage strain hardening of the material [17].

XRD patterns demonstrate the re-distribution mode of Cu-rich intermetallics after heat treatment. The experiments were done with step size and time per step of  $0.02^\circ$  and 2 s/step respectively. Because the maximum concentration of Cu in the MMC is 5 wt.%, it is not possible to track Cu-rich intermetallics in lower time per step. At higher time per step and at higher amounts of Cu a little intensity can be observable. Figures 5 a and b are XRD patterns of Al-Al<sub>4</sub>Sr composites containing 5 wt.% Cu, before and after solution heat treatment.

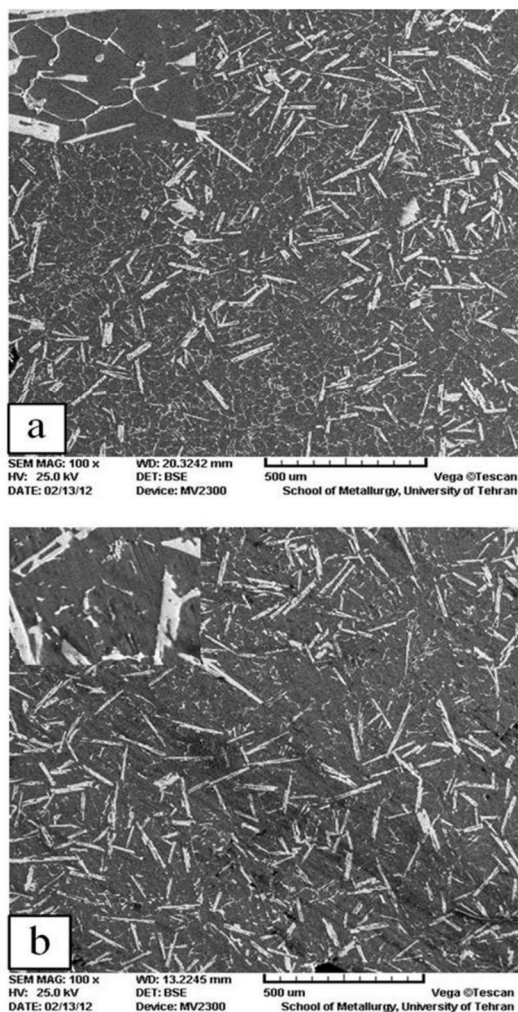
Before heat treatment,  $\theta$  phase peaks are clearly seen in the XRD pattern (Fig. 5-a). From Fig. 5-b, which shows the XRD pattern of the same material but after solution heat treatment, it is interesting to note that  $\theta$  phase peaks are absent. This can be explained by dissolution of  $\theta$

phase during heat treatment and diffusion of Cu atoms into the matrix. As it can be seen in Fig. 4-b, dissolution is not completed and  $\theta$  intermetallics (bright-coloured phases) are distributed uniformly in the matrix. Therefore, Cu-rich intermetallics are partially dissolved after the applied heat treatment.

#### 4. HARDNESS RESULTS

Hardness results of the Al-Al<sub>4</sub>Sr composite containing different Cu weight percentages are illustrated in Fig. 6.

The figure reveals that the changes in hardness values are marginal by adding Cu up to 1 wt.%, but it rises sharply in the presence of higher amounts of Cu (>1 wt.%) in the MMC. This enhancement is directly related to the presence of  $\theta$  phase in the MMC. As it is clear in Fig. 6, after adding 5 wt.% Cu to the composite, the hardness values increase from 35 HB to 56 HB. The presence of intrinsically hard and brittle Cu-rich intermetallics at the grain boundaries before heat treatment, prepares favoured path for stress



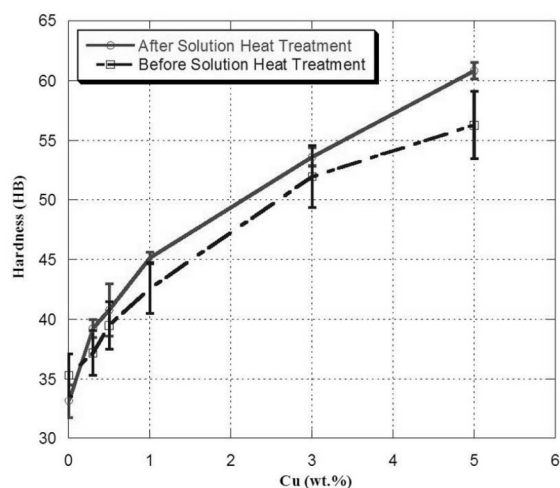
**Fig. 5.** XRD patterns of Al-Al<sub>4</sub>Sr containing 5 wt.% Cu: (a) before solution heat treatment and (b) after solution heat treatment at 500°C for 4 hours.

concentration. During heat treatment, dissolution of  $\theta$  phase intermetallics occur and uniformly distributes in the matrix.

## 5. CONCLUSIONS

The effect of Cu addition and solution heat treatment on the microstructure and hardness of Al-Al<sub>4</sub>Sr composite was studied. The following conclusions can be drawn:

1. Al<sub>4</sub>Sr intermetallics were found to be large and needle-like in the composite matrix.
2. The addition of Cu reduced the size of



**Fig. 6.** Brinell hardness of Al-Al<sub>4</sub>Sr composite as a function of Cu concentration.

- Al<sub>4</sub>Sr particles from 140  $\mu$ m to 65  $\mu$ m.
- Hard and brittle  $\theta$  phase was found in the MMC which segregates at the grain boundaries. This brittle phase can prepare favoured path for stress concentration and crack propagation through the boundaries.
- Solution heat treatment was found to be useful for partial dissolution of  $\theta$  phase and uniform distribution of Cu-rich intermetallics in the matrix.

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## REFERENCES

1. Seeman, M., Ganesan, G., Karthikeyan, R., Velayudham, A., "Study on Tool Wear and Surface Roughness in Machining of Particulate Aluminum Metal Matrix Composite-Response Surface Methodology Approach", International Journal of Advanced Manufacturing Technology, 2010, Vol. 48, pp. 613-624.
2. Clyne, T. W., Withers, P. J., "An Introduction to Metal Matrix Composites", Cambridge: Cambridge University Press, 1993.
3. Manoharan, M., Kamat, S. V., "On the Fracture Toughness of Particulate Reinforced Metal-Matrix Composites", Journal of Scripta Metallurgica, 1991, Vol. 25, pp. 2121-2125.

4. Ma, Z. Y., Liu, J., Yao, C. K., "Fracture Mechanism in a SiCw-6061 Al Composite", *Journal of Materials Science*, 1991, Vol. 26, pp. 1971-1976.
5. Srivatsan, T. S., "Cyclic Strain Resistance and Fracture Behaviour of  $Al_2O_3$ -Particulate-Reinforced 2014 Aluminium Alloy Metal-Matrix Composites", *International Journal of Fatigue*, 1995, Vol. 17, pp. 183-199.
6. Dunand, D. C., Derby, B., "In Fundamentals of Metal Matrix Composites", S. Suresh, A. Mortensen, A. Needleman Eds, Boston, MA: Butterworth-Heinemann, 1993.
7. Park, B. G., Crosky, A. G., Hellier, A. K., "Material Characterisation and Mechanical Properties of  $Al_2O_3$ -Al Metal Matrix Composites", *Journal of Materials Science*, 2001, Vol. 36, pp. 2417-2426.
8. Chen, S. H., Wang, T. C., "Size Effects in the Particle-Reinforced Metal-Matrix Composites", *Journal of Acta Mechanica*, 2002, Vol. 157, pp. 113-127.
9. Yueguang, W., "Particulate Size Effects in the Particle-Reinforced Metal-Matrix Composites", *Journal of Acta Mechanica Sinica*, 2001, Vol. 17, pp. 45-58.
10. Zhang, Z. H., Wang, F. C., Luo, J., Lee, S. K., Wang, L., "Microstructure and Mechanical Properties of Spark Plasma Sintered Al-SiC Composites Containing High Volume Fraction of SiC", *Materials Science and Engineering A*, 2010, Vol. 527, pp. 7235-7240.
11. Beffort, O., Long, S., Cayron, C., Kuebler, J., Buffat, P. A., "Alloying Effects on Microstructure and Mechanical Properties of High Volume Fraction SiC-Particle Reinforced Al-MMCs Made by Squeeze Casting Infiltration", *Journal of Composite Science and Technology*, 2007, Vol. 67, pp. 737-745.
12. Song, M., "Effect of Volume Fraction of SiC Particles on Mechanical Properties of SiC/Al Composites", *Transactions of Nonferrous Metals of Society of China*, 2009, Vol. 19, pp. 1400-1404.
13. Emamy, M., Nemati, N., Heidarzadeh, A., "The Influence of Cu Rich Intermetallic Phases on the Microstructure, Hardness and Tensile Properties of Al-15%Mg<sub>2</sub>Si Composite", *Materials Science and Engineering A*, 2010, Vol. 527, pp. 2998-3004.
14. Malekan, A., Emamy, M., Rassizadehghani, J., Emami, A. R., "The Effect of Solution Temperature on the Microstructure and Tensile Properties of Al-15%Mg<sub>2</sub>Si Composite", *Materials and Design*, 2011, Vol. 32, pp. 2701-2709.
15. Khorshidi, R., Honarbakhsh Raouf, A., Emamy, M., Campbell, J., "The Study of Li Effect on the Microstructure and Tensile Properties of Cast Al-Mg<sub>2</sub>Si Metal Matrix Composite", *Journal of Alloy and Compounds*, 2011, Vol. 509, pp. 9026-9033.
16. Zhang, J., Fan, Z., Wang, Y. Q., Zhou, B. L., "Microstructural Evolution of the In Situ Al-15wt.%Mg<sub>2</sub>Si Composite with Extra Si Contents", *Journal of Scripta Materialia*, 2000, Vol. 42, pp. 1101-1106.
17. Dieter, G. E., "Mechanical Metallurgy", SI Metric Ed, London: McGraw-Hill Book Company, 1988.
18. American Society of Mechanical Engineers, "Mechanical Engineering", Vol. 42, Michigan: The University of Michigan, 1920.