

## MICROSTRUCTURE STUDY ON AL-5%SiC NANOCOMPOSITE POWDERS

T. Rostamzadeh<sup>1</sup> and H. R. Shahverdi<sup>2,\*</sup>

\* shahverdi@modares.ac.ir

Received: July 2010

Accepted: December 2010

<sup>1</sup> Department of Electronic Engineering, Faculty of Engineering, Islamic Azad University, Garmsar Branch, Garmsar, Iran.

<sup>2</sup> Department of Nano-Material Engineering, Faculty of Engineering, Tarbiat Modares University, Tehran, Iran.

**Abstract:** In this study Al-5 (Vol) % SiC<sub>p</sub> nanocomposite powder has been successfully synthesized by high-energy planetary milling of Al and SiC powders for a period of 25 h at a ball-to-powder ratio of 15:1. The changes of the lattice strain, the crystallite size of the matrix phase, and the nanocomposite powder microstructure with time have been investigated by X-ray diffraction (XRD), X-ray mapping, and scanning electron microscopy (SEM) analyses. The morphologies of the nanocomposite powders obtained after 25 h of milling have also been studied by transmission electron microscopy (TEM). The results showed that nanocomposite powders were composed of near-spherical particles and, moreover, the SiC particles were uniformly distributed in the aluminum matrix.

**Keywords:** Microstructure evolution, Al-SiC, nanocomposite, mechanical milling.

### 1. INTRODUCTION

Metal matrix composites (MMCp) are engineering materials in which a hard ceramic component is dispersed in a ductile metal matrix in order to obtain characteristics that are superior to those of conventional monolithic metallic alloys [1- 5]. Among these materials, aluminum-based metal matrix composites (MMCs) are appropriate materials for structural applications in the aircraft and automotive industries because they are lightweight and have a high strength-to-weight ratio [6-12]. Uniform dispersion of the fine reinforcements and a fine-grained matrix improve the mechanical properties of the composite. Incorporation of ceramic particulates into the metallic matrix can be accomplished by several techniques, such as molten-metal routes or solid-state processing [13, 14]. It should be noted that Al-SiC composites are difficult to obtain by conventional melting-based methods due to the poor wettability between molten Al and the SiC. In addition, these methods usually lead to an undesirable reaction between the SiC and molten Al, which produces brittle phases of Al<sub>4</sub>C<sub>3</sub> and Si [15, 16]. Mechanical alloying (MA), as originally developed by Benjamin, has been widely utilized to produce metal or ceramic-based composite powders with fine microstructures. The mechanical alloying process

involves repeated welding and fracturing of a mixture of powder particles to produce an extremely fine microstructure [17- 27]. Recently, several research articles have focused on the production of composite materials, the effect of milling time on the mechanical properties of the products, and optimization of the milling parameters using the mechanical alloying method [28- 31]. As the milling time has considerable effect on morphology and other properties of the mechanically milled powder, this study was designed and undertaken to consider the structural evolution of nanocomposite powders during the course of milling. The current paper presents experimental results relating to the effect of milling time on the morphology, crystallite size, lattice strain, distribution of reinforcement phase, and microstructure of Al-SiC composite powders.

### 2. EXPERIMENTAL PROCEDURE

Aluminum (purity 99.9%) and SiC (purity >99.5%) powders were mixed to give a nominal composition of Al-5%(Vol)SiC. To avoid agglomeration and the cold welding of powder particles, stearic acid amounting to 1.5 wt% of the total powder charge was used as a process control agent. The preparation of the nanocomposite powders by mechanical milling

**Table 1.** Crystallite sizes and lattice strains of Al-5%SiC milled powders after 5, 10, 15, and 25 h of milling.

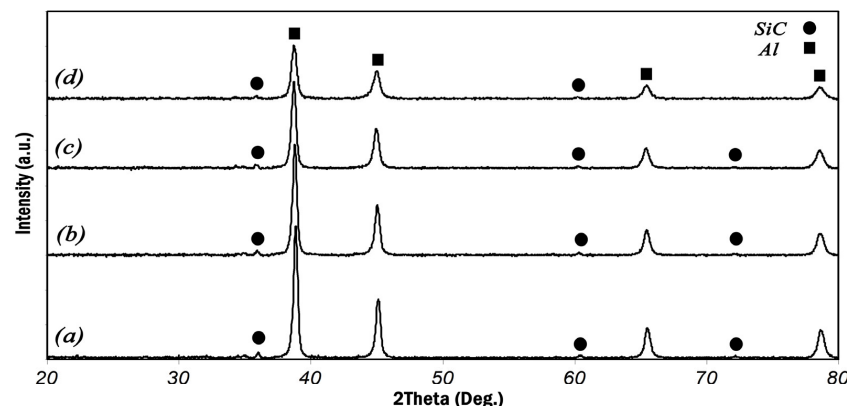
Milling time (h)	Average crystallite size (nm)	Maximum lattice strain (%)
5	71	0.25
10	62	0.33
15	54	0.38
25	45	0.41

of the mixed powder was carried out using a high-energy planetary ball mill with a hardened stainless steel vial and 12 mm diameter hardened steel balls in an argon atmosphere. A rotational speed of 300 rpm and a ball-to-powder weight ratio of 15:1 were employed. In order to avoid oxidation of powders during the MA treatment, all handling was performed in a glove box in an argon atmosphere. Small amounts of the milled powders were collected for microstructure analysis at different stages during the MA process. A Philips XL-30 scanning electron microscope (SEM) coupled with an energy-dispersive spectrometer (EDS) for compositional analysis and a Philips CM200 FEG transmission electron microscope (TEM) were used to characterize the microstructures of the milled powders. X-ray investigations were performed using a Philips PW-1730 diffractometer with  $\text{Cu-K}\alpha$  radiation. To determine the crystallite size  $d$  and the lattice strain  $\varepsilon$ , the Williamson–Hall method was adopted. X-ray mapping was also carried out to observe the distribution of the different elements in the milled powders.

### 3. RESULTS AND DISCUSSION

#### 3. 1. XRD Analysis

Figs. 1 (a)–(d) show the XRD patterns of the Al-5%SiC composite powders after 5, 10, 15, and 25 h of milling time. There was no formation of any other phase, but the X-ray diffraction patterns display increasing broadening of the crystalline peaks as a function of milling time. The XRD peaks were analyzed using the Williamson–Hall method [16] and the results are reported in Table 1. Of significance here is that as the milling time was increased, work hardening rate and matrix deformation were enhanced, and so the lattice strain increased and the average size of the aluminum phase crystallites decreased [16, 17]. Table 1 shows that the Al-5%SiC composite powders acquired the smallest crystallite size (45 nm) with the highest lattice strain (0.41%) after 25 h of milling. Furthermore, it is evident that mechanical milling led to a faster rate of change of the average grain size and lattice strain in the early stage of the MA process as compared to the later stages. A slight shift in the position of the

**Fig. 1.** X-ray diffraction patterns of Al-5%SiC milled powders after (a) 5 h, (b) 10 h, (c) 15 h, and (d) 25 h of milling time.

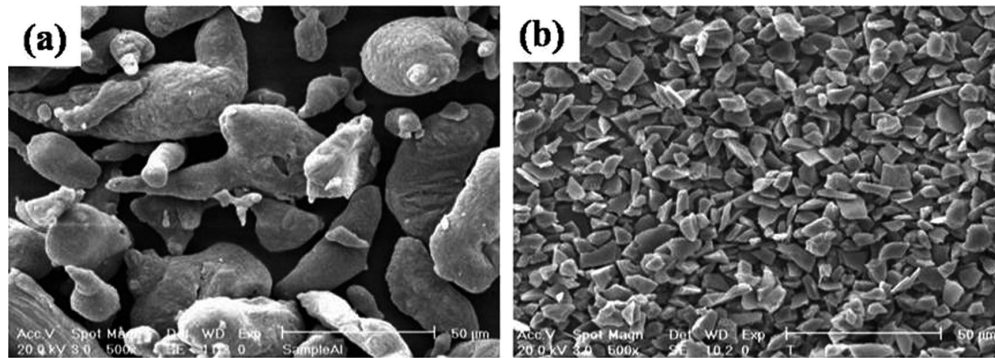


Fig. 2. Primary powders used: (a) Aluminum, (b) SiC

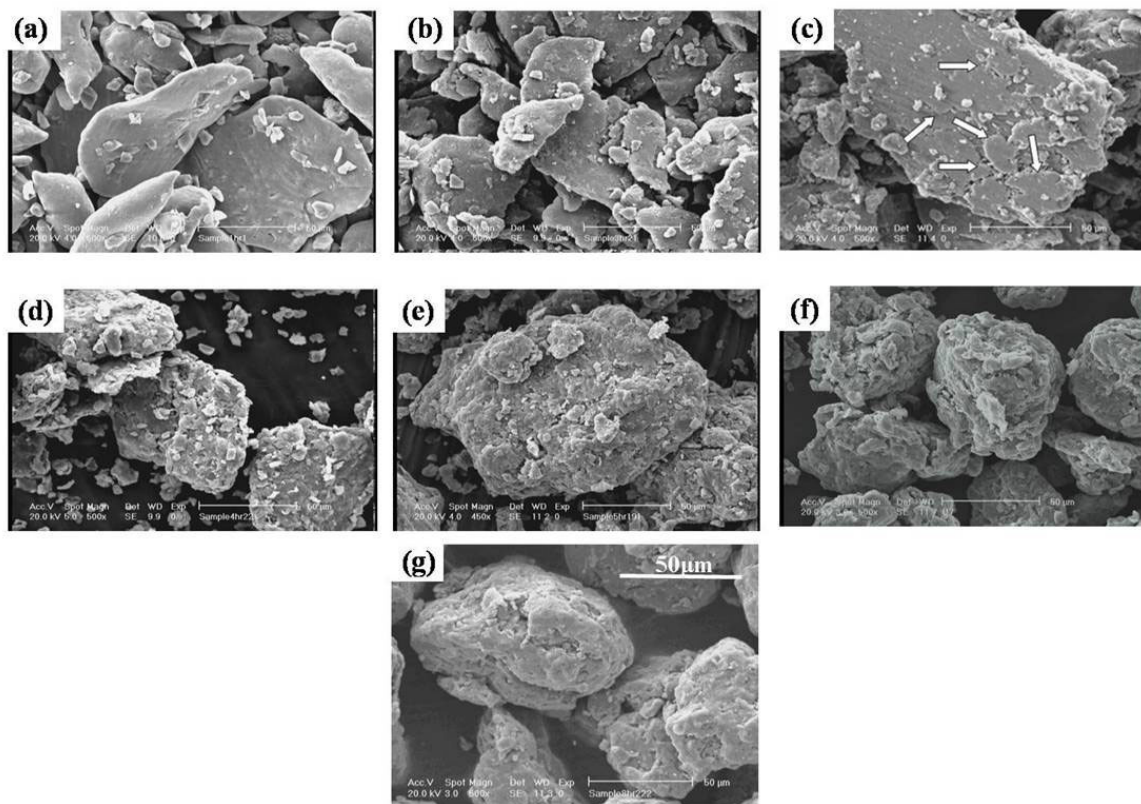


Fig. 3. SEM morphologies of Al-5% SiC after (a) 1 h, (b) 2 h, (c) 5 h, (d) 10 h, (e) 15 h, (f) 20 h, and (g) 25 h of ball-milling time.

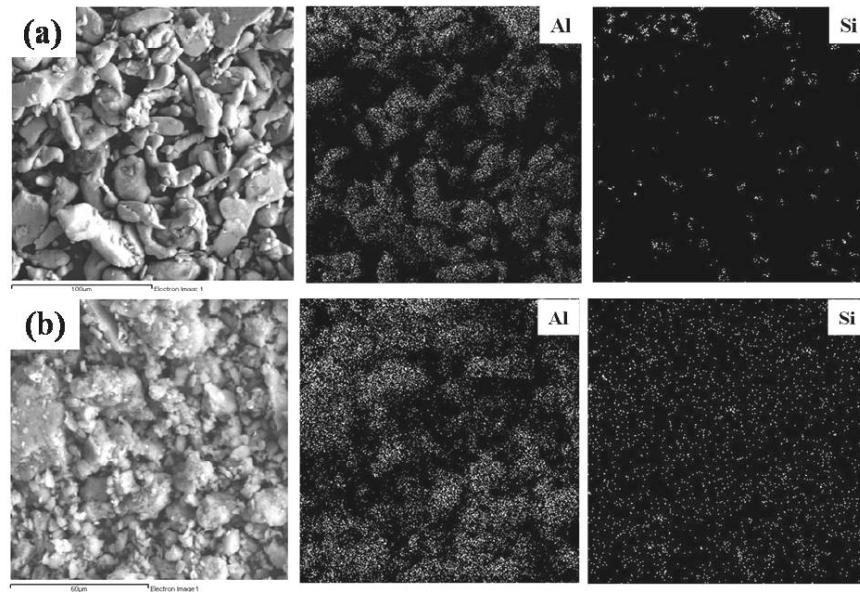
XRD peaks was also noticed and could be related to the dissolution of impurities, mostly iron, in the lattice of the matrix phase [16].

### 3. 2. Microstructural Observations

Fig. 2 shows the particle morphologies of the Al and SiC initial powders. Fig. 3 shows the effect of milling time on the morphology of the

Al/5%SiC powders, while Fig. 4 depicts X-ray map results showing the distribution of the elements Al and Si in the nanocomposite powders milled for 2 h and 5 h.

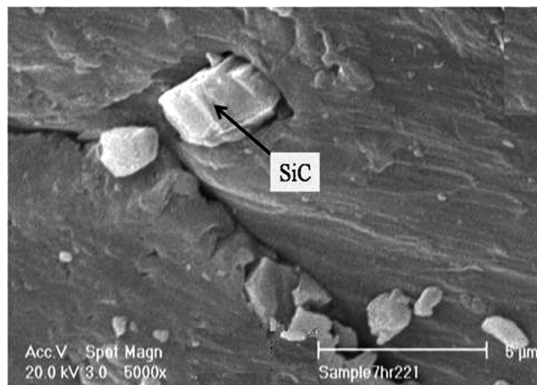
From inspection of these figures, it is clear that the powders obtained after the short milling time were bulky with random shape and size and the distribution of the reinforcing particle was not uniform (Fig. 4(a)). Also, the distance between



**Fig. 4.** X-ray maps showing the distributions of the elements Al and Si for the Al-5%SiC powders milled for (a) 2 h and (b) 5 h.

SiC particles was rather large (Fig. 3(a)). It can be seen from Figs. 3(b), (c) that the aluminum particles were first flattened by the cold working and shear forces generated by the ball-powder-ball collisions in the milling medium. Detailed inspection of the particle surface microstructure after 5 h of milling revealed the presence of SiC uniformly distributed on the surfaces of the flakes. Moreover, it is evident from Fig. 4 (b) that the dispersal of SiC in the aluminum matrix was greatly improved after 5 h of milling. Fig. 5 shows a broken SiC particle embedded in an

aluminum matrix after 5 h of milling. Indentation of the hard reinforcing phase into the ductile matrix (Fig. 5) led to an increase of work hardening and therefore the increase of fracture rate of Aluminum matrix. As a result, the aluminum particles started to laminate and became finer. With increasing milling time, new flake surfaces were created and the particles welded together and agglomerated to form larger powders. Agglomerated powders have been shown and marked with arrows in Fig (3c). This occurred because of the tendency for decreasing surface free energy. On the other hand, increasing the MA time increases the hardness and this leads to fracturing of the agglomerated powders to form smaller particles [17]. However, there are differences in average particle size when different milling times are used, which indicates that milling time and other processing phenomena, such as competition between fracturing and agglomeration rate, have an influence on the morphology and size of the agglomerated powders (Figs. 3 (d)–(f)). It is clear that the increased milling time resulted in refining the SiC particles and decreasing the distance between them. Also, the aluminum particles were laminated and the broken SiC



**Fig. 5.** SiC powder embedded within an aluminum matrix after 5 h of milling.



Fig. 6. SEM morphologies of Al-5% SiC after 25 h of ball-milling at different magnifications.

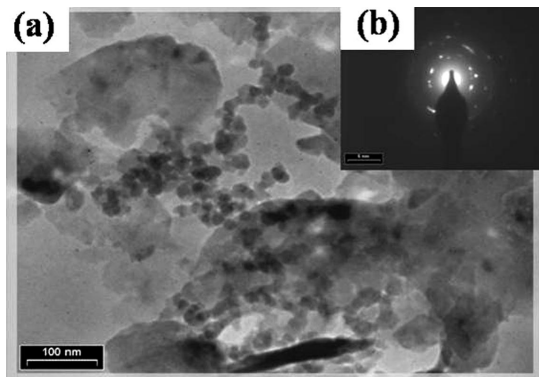


Fig. 7. (a) Bright-field image (BFI) and (b) selected-area diffraction pattern (SADP) of Al-5%SiC nanocomposite powder obtained after 25 h of ball-milling.

particles became embedded within the aluminum matrix lamina. Fracturing and agglomerating continued throughout the milling time competitively. Finally, a near-spherical nanostructured composite powder with a uniformly distributed reinforcing phase in a laminated aluminum matrix was obtained when fracturing and cold welding were balanced (Fig. 3(g)). The final powder particles were well-rounded and composed of a multitude of layers.

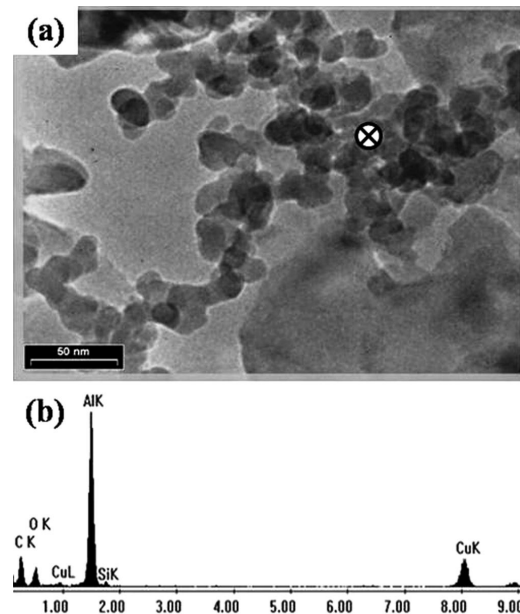


Fig. 8. (a) BFI of Al-5%SiC nanocomposite powder obtained after 25 h of ball-milling and (b) EDS analysis of the region marked in (a).

Fig. 6 shows the microstructure of the agglomerated spherical nanocomposite powder obtained after 25 h of MA (Fig. 3(g)) at different



**Fig. 9.** (a) Bright-field image and (b) dark-field image of Al-5%SiC layered-structure powder obtained after 25 h of ball-milling, and (c) EDS analysis of the region marked by (1) in (a).

magnifications. It is obvious that the agglomerated powder included much laminate (Figs. 6(b), (c)) and contained embedded spherical nanoparticles (Fig. 6(d)).

Transmission electron microscopy (TEM) allowed a better understanding of the structure of the Al-5%SiC nanocomposite. Figs. 7 (a) and (b) show a bright-field image (BFI) and a selected-area diffraction pattern (SADP), respectively, of the nanocomposite powder obtained after 25 h of ball-milling. The bright-field image indicates that there are some nearly spherical nanoparticles and some layered structures of greater size (Fig. 7 (a)). The SADP shows a reasonably sharp ring/spot pattern, which is characteristic of diffracting polycrystalline components (Fig. 7 (b)).

Fig. 8 shows that the nearly spherical particles were approximately 5–20 nm in diameter. EDS analysis of the region marked in Fig. 8 (a) proved the existence of Al and SiC components. Bright- and dark-field images in conjunction with EDS analysis of 1, 2 points proved that the SiC nanoparticles, the numbered points, were appropriately distributed in the laminated matrix after 25 h of milling, as shown in Fig. 9.

Dissimilar brightnesses of different points in the dark-field image (Fig. 9 (b)) can be attributed to different orientations and diverse crystallite structures of SiC nanoparticles.

#### 4. CONCLUSIONS

- By using mechanical milling, nanometer-sized SiC particle reinforced Al–SiC composite powder can be synthesized.
- Progressive milling from 0 to 25 h changes the morphology of the powder particles from flaky to near-spherical.
- Morphological study has revealed that the agglomerated powders consist of many layers and that they contain embedded spherical nanoparticles.
- X-ray mapping and TEM analyses have indicated that the SiC nanoparticles are appropriately distributed in the metal matrix.
- It has been established that with increasing milling time, the lattice strain increases and the average size of the aluminum phase crystallites decreases. Moreover,



mechanical milling improves the distribution of SiC throughout the aluminum matrix, thus enhancing the characteristics of the composite.

## 5. ACKNOWLEDGEMENTS

Experimental support of the Tarbiat Modares University and financial support of Iranian Nanotechnology Initiative are gratefully acknowledged.

## REFERENCES

1. Miracle, DB, "Metal matrix composites - From science to technological significance", *Composites Science and Technology*, 2005, Vol. 65, 2526–2540
2. Rostamzadeh, T., Shahverdi, H. R., Sarraf-Mamoory, R., "Investigation the effect of volume fraction reinforcement phase on Microstructure of Al-SiC nanocomposite powder prepared via mechanical alloying", *Advanced Materials Research*, 2010, Vol. 83, 764-770.
3. Hassan, S. F., Gupta, M., "Effect of Type of Primary Processing on the Microstructure, CTE and Mechanical Properties of Magnesium/Alumina Nanocomposites", *Composite Structures*, 2006, Vol. 72, 19–26
4. Bhaduri, A., Gopinathan, V., Ramakrishnan. P., and Miodownik, A. P., "Processing and properties of SiC particulate reinforced Al-6.2Zn-2.5Mg-1.7Cu alloy (7010) matrix composites prepared by mechanical alloying", *Materials Science and Engineering A*, 1996, Vol. 221, 94-101
5. Tham, L. M., Gupta, M., Cheng, L., "Effect of limited matrix-reinforcement interfacial reaction on enhancing the mechanical properties of aluminium-silicon carbide composites", *Acta mater*, 2001. Vol. 49, 3243–3253
6. Prabhu, B., Suryanarayana, C., An, L., Vaidyanathan, R., " Effect of Reinforcement Volume Fraction on the Mechanical Properties of Al—SiC Nanocomposites Produced by Mechanical Alloying and Consolidation", *Material Science Engineering A*, 2006. Vol. 425, 192-200
7. Ogel, B. and Gurbuz, R., "Microstructural Characterization and Tensile Properties of Hot Pressed Al-SiC Composites Prepared From Pure Al and Cu Powders", *Materials Science and Engineering A*, 2001, Vol. 301, 213-220
8. Ozcatbas, Y., "Investigation of the machinability behaviour of Al4C3 reinforced Al-based composite produced by mechanical alloying technique", *Compos. Sci. Technol.* 2008, Vol. 63, 53-61
9. Yang, Y., Lan, J., Li, X., "Study on bulk aluminum matrix nano-composite fabricated by ultrasonic dispersion of nano-sized SiC particles in molten aluminum alloy", *Materials Science and Engineering A*, 2004. Vol. 380, 378–383
10. Babu, A. S., Jayabalan, V., "Weibull Probability Model for Fracture Strength of Aluminium (1101)-Alumina Particle Reinforced Metal Matrix Composite", *J. Mater. Sci. Technol.* 2009, Vol. 25, 341-343
11. Wu, J. M., Li, Z. Z., " Nanostructured composite obtained by mechanically driven reduction reaction of CuO and Al powder mixture", *Journal of Alloys and Compounds*, 2000. Vol. 299, 9–16
12. Bakshi, S. R, Lahiriand, D., Agarwal, A., "Carbon nanotube reinforced metal matrix composites—areview", *International Materials Reviews*, 2010, Vol. 55, 41-64
13. Sherif El-Eskandarany, M., " Mechanical solid state mixing for synthesizing of SiCp/Al nanocomposites", *Journal of Alloys and Compounds*, 1998, Vol. 279, 263-271
14. Lee, K. B., Sim, H. S., " Fabrication and characteristics of AA6061/SiCp composites by pressureless infiltration technique", *Jornal of Material Science*, 2001, Vol. 36. 3179 – 3188
15. Rostamzadeh, T., Shahverdi, H., and Shanaghy A, "EIS study of balk Al-SiC nanocomposite prepared by mechanical alloying and hot press method", *Advanced Materials Research*, 2010, Vol. 83, 1297-1305
16. Razavi Hesabi Simchi, A., Seyed Reihani, M., " Structural Evolution during Mechanical Milling of Nanometric and Micrometric Al<sub>2</sub>O<sub>3</sub> Reinforced Al Matrix Composite" *Material Science Engineering A*, 2006, Vol. 428. 159-

- 168
17. Suryanarayana, C., "Mechanical Alloying and Milling", *Progress in Materials Science*, 2001, Vol. 46, 1-184
18. Zhang, D. L., "Processing of Advanced Materials Using High-Energy. Mechanical Milling", *Progress in Materials Science*, 2004, Vol.49, 537–560
19. Witkin, D. B., Lavernia, E. J., "Synthesis and mechanical behavior of nanostructured materials via cryomilling", *Progress in Materials Science*, 2006, Vol. 51, 1–60
20. Zebarjad, S. M., Sajjadi, S. A., "Microstructure evaluation of Al-Al<sub>2</sub>O<sub>3</sub> composite produced by mechanical alloying method", *Materials and Design*, 2006, Vol. 27, 684–688
21. Woo, K. D., Zhang, D. L., "Fabrication of Al-7wt%Si-0.4wt%Mg/SiC nanocomposite powders and bulk nanocomposites by high energy ball milling and powder metallurgy", *Current Applied Physics*, 2004, Vol. 4, 175–178
22. Ruiz-Navas, E. M., Fogagnolo, J. B., Velasco, F., Ruiz-Prieto, J. M., Froyen, L., "One step production of aluminium matrix composite powders by mechanical alloying", *Composites: Part A*, 2006, Vol. 37, 2114–2120
23. Morsi, K., Esawi, A., "Effect of Mechanical Alloying Time and Carbon Nanotube (CNT) content on the evolution of Aluminum (Al)-CNT Composite Powders", *Journal of Material Science*, 2007, Vol. 42, 4954–4959
24. Bermudez, M. D., Mart nez-Nicolas, G., Carrion, F. J., Martinez-Mateo, I., Rodriguez, J. A., Herrera, E. J., "Dry and lubricated wear resistance of mechanically-alloyed aluminium-base sintered composites", *Wear*, 2001, Vol. 248, 178–186
25. Guang, R., Jing-En, Z., Shengqi, X., Pengliang, L., "Formation of nanocrystalline and amorphous phase of Al-Pb-Si-Sn-Cu Powder during mechanical alloying", *Material Science Engineering A*, 2006, Vol. 416, 45-50
26. Sankar, R., Singh, P., "Synthesis of 7075 Al/SiC Particulate Composite Powders by Mechanical Alloying", *Materials Letters*, 1998, Vol. 36, 201–205
27. Lu, L., Lai MO, Ng CW, "Enhanced mechanical properties of an Al based metal matrix composite prepared using mechanical alloying", *Materials Science and Engineering A*, 1998, Vol. 252, 203–211
28. Abdoli, H., Salahi, E., Farnoush, H., Pourazrang, K., "Evolutions during synthesis of Al–AlN-nanostructured composite powder by mechanical alloying", *Journal of Alloys and Compounds*, 2008, Vol. 461, 166–172
29. Zhang, D. L., Liang, J., Wu, J., "Processing Ti3Al/SiC nanocomposite using high energy mechanical milling", *Materials Science and Engineering A*, 2004, Vol. 375, 911–916
30. Dashtbayazi, M., Shokuhfar, R., "Statistical modeling of the mechanical alloying process for producing of Al/SiC nanocomposite powders", *Computational Materials Science*, 2007, Vol. 40, 466–479
31. Rao, K. P., Zhou, J. B., "Thermal Stability and Phase Evolution of Mechanically Alloyed Ti-Al-Si-C Powders", *Journal of Material Science*, 2004, Vol.39, 5471–5476