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Characterisation of Al-10 wt. % Al_2O_3 Powder Composite: Synthesised via Ball Milling

Mohd Danish
Mechanical Engineering Department
Aligarh Muslim University
Aligarh, India
mdanish.amu@gmail.com

M Arif Siddiqui
Mechanical Engineering Department
Aligarh Muslim University
Aligarh, India
mas_alig@gmail.com

***Abstract*—Al-10 wt. % powder composite synthesised via conventional ball mill. Characterisation has been done at different interval of milling time. X-ray diffraction (XRD) and scanning electron microscope (SEM) techniques are used for characterisation of the powder composite which shows that uniform distribution of Al_2O_3 in Al matrix was successfully obtained after milling the powder for the period of 12 hours. The uniform distribution of Al_2O_3 in the Al matrix was confirmed by characterizing the powders by SEM. The particle size of powder composite is decreasing with increase in milling time. Addition of Al_2O_3 particles in Al powder further accelerates the milling and if further decreases the size of particle which is shown by the SEM micrographs. Crystallite size has got similar trend of decreasing like particle size which is shown by XRD. XRD also shows increase in lattice strain in the powder particle which is induced during the milling.**

***Key words*—Al-Al_2O_3 powder composite; Ball milling; SEM; X-ray diffraction.**

I. Introduction

Metal matrix composites find a varied application in aerospace, automotive and military industries [1,2] owing to their high mechanical properties and good physical behaviour including light weight, electrical and thermal conductivity. There is a whole lot of metallic alloy systems available at hand which can be used as matrix, however, Al in this category have attracted most of the researchers due to its low density, heat treatment capability, and wide range of its alloys and processing flexibility. Reinforcing the ductile aluminium matrix with stronger and stiffer reinforcements like oxides, carbides, borides, and nitrides provides a combination of properties of both the metallic matrix and the ceramic reinforcement components resulting in improved physical and mechanical properties of the composite [3,4].

Addition of ceramic reinforcements into a ductile matrix has a great effect on structural evolution during ball milling [5-11]. Further, enhancement in the mechanical properties of the composite with decrease in the particle size of the reinforcements [12-15] is reported. Among various reinforcements, Al_2O_3 is one of the most widely used dispersoid in Al-based composites [16,17]. Some of the earlier works report the synthesis of Al-Al_2O_3 powder composites. Ball milling is a simple and useful technique for attaining a homogeneous distribution of inert fine particles within a fine grained matrix [18,19]. Most of the earlier works involve high energy ball mill for synthesis of composites, which requires a high energy as well as has low production rate. The present work employs a low energy conventional ball mill for the synthesis of Al-10 wt. % Al_2O_3 powder composite. The characterisations of samples have been done with the help of X-ray diffraction patterns and SEM micrographs. Results show that addition of Al_2O_3 powder markedly influences the structural and morphological evolution of the Al matrix. Homogeneous distribution of Al_2O_3 particles and uniform size distribution is confirmed by SEM micrographs. The Analysis of lattice strain and grain size was also carried out with the help of XRD patterns during milling stages.

II. Experimental Procedures

A. Sample Preparations

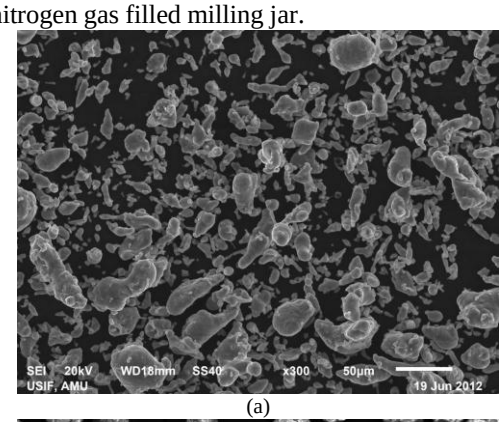
The Al powder of 99.9 % purity and average particle size of 40 μm with maximum 3% particles have size greater than 75 μm was obtained from MEPCO, The Metal Powder Company Limited, India.

Pure aluminium oxide (Al_2O_3) powder was obtained from commercial vendors. Al_2O_3 powder is of 99 % purity with the average particle size of 150 μm. Ethyl alcohol has been used as the process controlling agent. The Figure 1 shows the SEM micrographs of the pure aluminium and alumina powder.

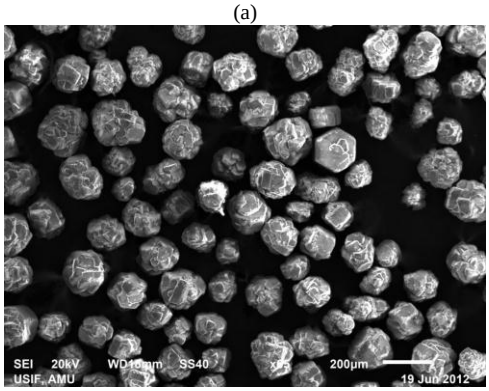
B. Milling of Powder

The pure powders of Al and Al_2O_3 were mixed in the desired weight. After mixing, the powder blend was put inside the milling vial under the atmosphere of nitrogen gas. Nitrogen gas is inert in nature and it will provide an inert atmosphere during milling of powder. About 100 grams of powder mixture is used along with 12 stainless steel balls each of which has a diameter of 20mm and a total weight is 392 grams. Therefore in our experiment the ball to powder ratio is 4:1 which is maintained throughout the experiments. Here we have used a low energy ball mill having vial diameter and length is 115mm and 156 mm respectively. The mill was rotated at constant speed of 104 rpm which is nearly 75% of its

critical speed. The powders along with the balls were loaded into the nitrogen gas filled milling jar.



(a)



(b)

Fig. 1: SEM micrographs of as received (a) Al powder and (b) Al_2O_3 powder.

Milling was carried out for different times until steady-state conditions were achieved. The milled powders were taken out at regular intervals of time i.e. 2, 4, 8 and 12 hours for morphological and structure analysis. To avoid any unwarranted and excessive cold welding of powder particles amongst themselves, onto the internal surfaces of the vial, and to the surface of the grinding medium during milling, few drops of ethyl alcohol was added to the mixture as the process control agent.

C. Characterisation of Powder Composites

The X-ray diffraction patterns were taken using Philips Bruker D8 Advanced X ray Diffractometer with Cu Kα radiations. The peak broadening observed in XRD could be due to physical factors such as crystallite size and lattice strain. The average crystal size and lattice strains were obtained using Williamson–Hall method [20].

$$\beta\cos\theta = \lambda / d + 2\eta\sin\theta \quad (1)$$

The morphological characterisation of powder i.e. distribution of Al_2O_3 particles and particle size distribution was studied by JOEL-JSM 6510 LV scanning electron microscope (SEM). Figure 1 shows the SEM micrographs of as received Al and Al_2O_3 powder.

III. RESULTS AND DISCUSSIONS

A. Morphological Characterisation

The as-received Al powder is irregular in shape as shown in Figure 1. The monolithic aluminium particles were deformed into flake like shapes after 2 hours of milling as shown in Figure 2a. After 4 hours milling, the particle are found to have the largest size (Figure 2b). This is because of the fact that the aluminium is ductile in nature. Due to the ductile nature of aluminium powder, welding occur between the powder particles resulting in an increase in particle size. It is also noted that the maximum size of particles are found to be in this stage. It is because welding is the dominating mechanism in this stage.

Flake like morphology was maintained after 8 hours of milling but the particle size distribution was changed and the average particle size was decreased (Figure 2c). In this time period, the fracture mechanism is activated due to the work hardening of the particles. Large flake like particles are crushed due to the intensive impacts of balls and collision with the walls of ball mill. Further milling up to 12 hours has no considerable effect on the morphology of the particles (Figure 2d). Indeed at milling times longer than 8 hrs, the steady state predominates.

Figure 3 shows the sequence of scanning electron micrographs of the Al-10 wt. % Al_2O_3 powder as a function of milling time. The alumina particle starts distributing in aluminium matrix after 2 h of milling as shown in Figure 3a. It may be noticed from the micrograph that Al_2O_3 particles were appearing bright and the Al matrix appearing grey in the image.

The particle size is changing with milling time, as a result of the two opposing factors of cold welding and fracturing of powder articles. While cold welding increases the particle size, fracturing reduces the size. In the early stages of milling, the powder particles are still soft and cold welding predominates. Consequently, an increase in particle size is observed after 4h of milling as shown in Figure 3b. Note that at this milling stage, particles have been under deformation and cold welding therefore flattened particles with high aspect ratio were formed. With continued milling, the particles get work hardened and become brittle and the rate of fracturing tends to increase resulting in a reduction in particle size after 8 hours of milling. This can be clearly seen in Figure 3c. Bigger particles are still visible after 8 hours of milling. It is because once fracturing had occurred, fresh particle surfaces are produced and due to the high reactivity of these surfaces, cold welding again becomes predominant leading to an increased particle size after a milling time of 8 h, as depicted in Figure 3c. Eventually, a balance is established between the cold welding and fracturing events and a steady-state situation is obtained. That is, the particle size gets stabilized, and does not change with further milling (Figure 3d).

It can be clearly see that as we increase the milling time, the uniform distribution of reinforcement particles increases.

The distribution of Al_2O_3 particles is very uniform after 12 hours of milling.

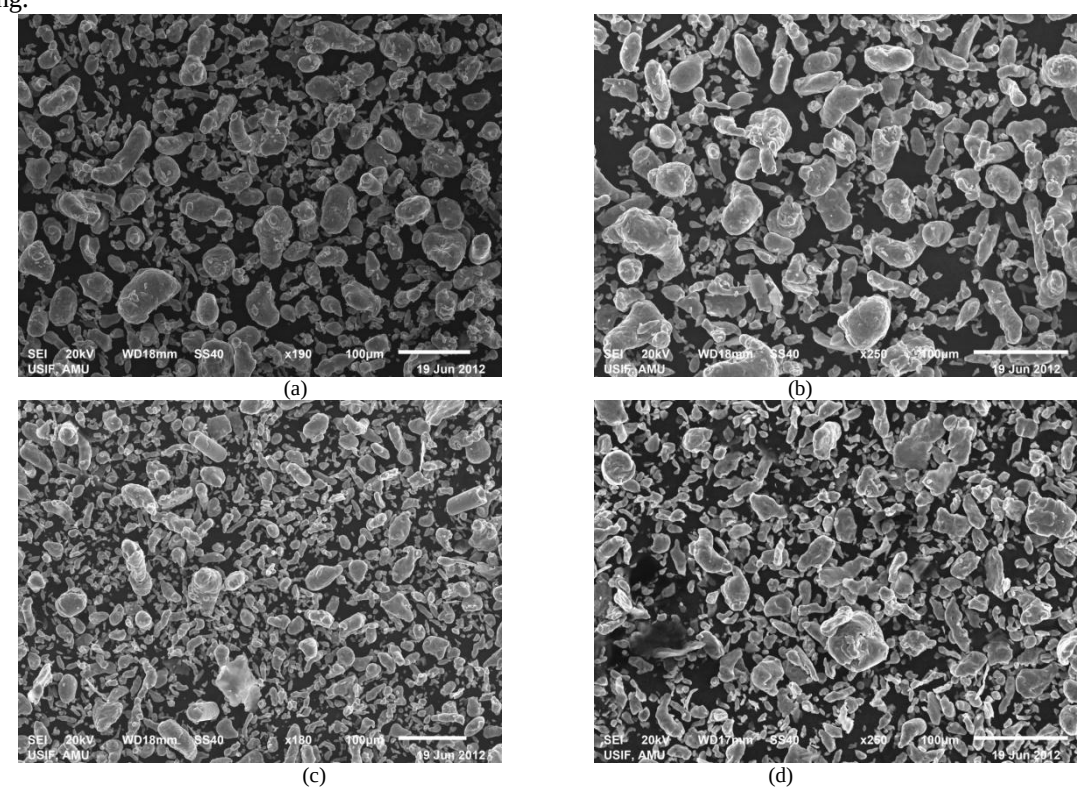


Fig. 2: SEM Micrographs of monolithic Al powder particles at different milling time: (a) 2 h, (b) 4 h, (c) 8 h and (d) 12h

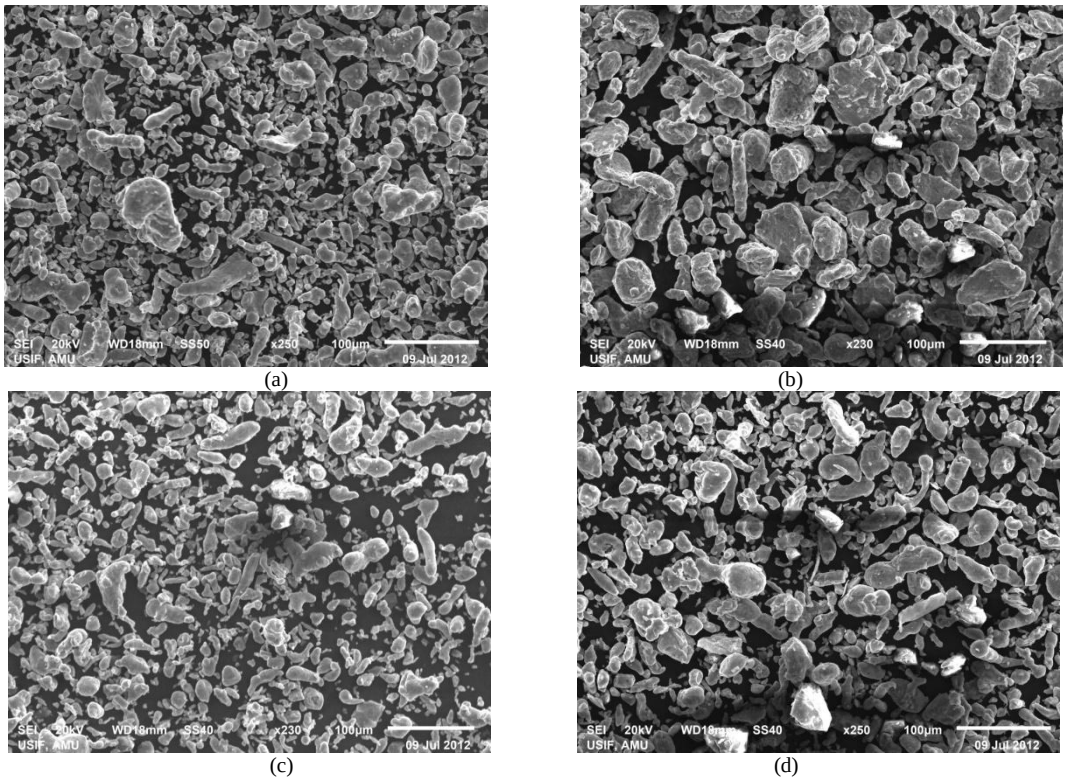


Fig. 3: SEM micrographs of Al-10 wt. % Al_2O_3 powder milled for different times: (a) 5h, (b) 4h, (c) 8h and (d) 12h

On comparing Figure 2 and 3 we can notice that on addition of brittle particle of Al_2O_3 in Al matrix, the particle size decreases more rapidly with increase in milling time than in the case of monolithic Al. It is because addition of Al_2O_3 particle it accelerated the milling process, leading to faster work hardening rate and fracture of the aluminium powder.

B. Microstructure Characterisation

X-ray diffraction experiments were carried out on the milled powders for crystal structure analysis. X-ray diffraction patterns are taken at different milling time. Figure 4 shows the x-ray diffraction patterns of Al-10 wt. % Al_2O_3 at different milling time which are 2, 4, 8 and 12 hours. The peaks of Al are clearly shown in the Figure 4 but the peaks of alumina are of very low intensity. Further, on increasing the milling time, peaks tend to increase its broadness which is due to grain refinement of the powder and lattice strain induced during milling.

The crystallite size and lattice strain of monolithic aluminium and Al-10 wt. % Al_2O_3 powder composite during milling time is calculated by Williamson-Hall method i.e. Eq. 1. Figure 5 shows the variation of crystallite size and lattice strain as a function of milling time. It is very clear that the crystallite size is decreasing with increasing milling time.

It is also noted from the graph that the crystallite size decreases rapidly in early milling stages that is up to 4 hours of milling. After 4 hours, the grain size decrease at smaller rate and grain size reached a minimum. After 8 hours of milling, it nearly become stable as the milling reaches to steady state. With addition of Al_2O_3 the grain size decreases at more rapid rate it could be due to its brittle nature induced in the Al matrix due to its brittleness.

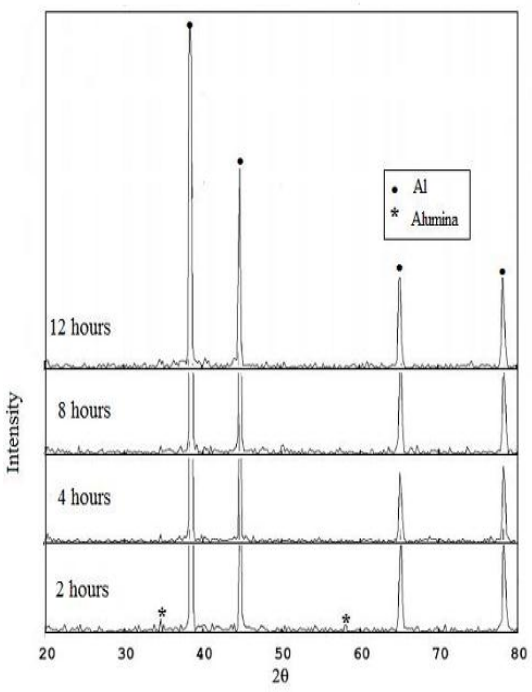
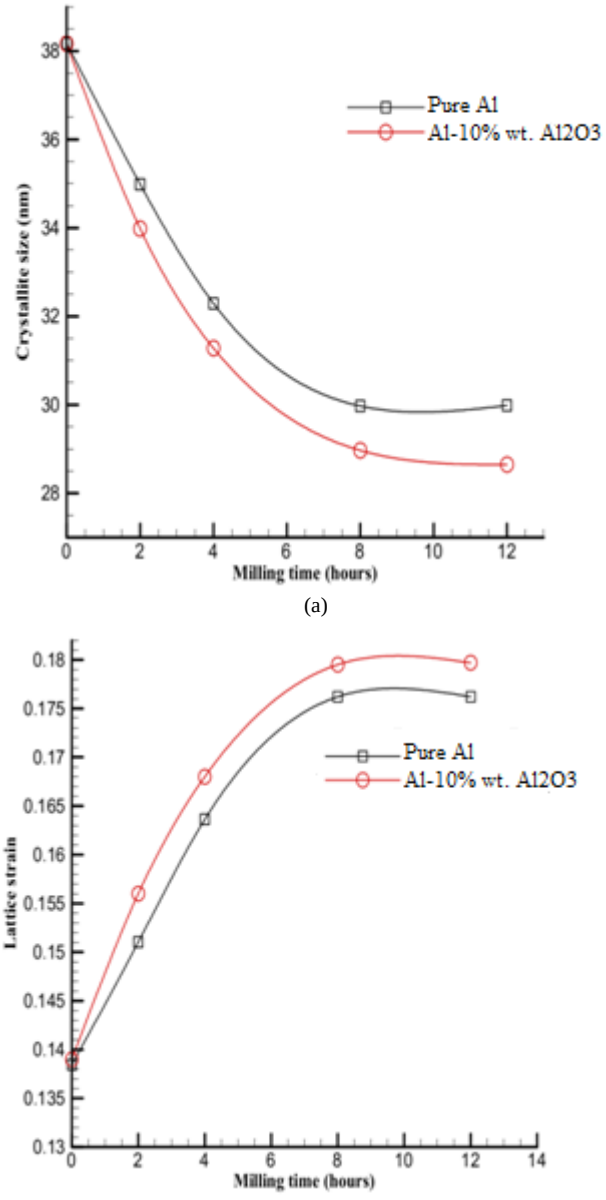


Fig. 4: XRD patterns of Al-10 wt. % Al_2O_3 obtained for different milling time.



(a)

(b)

Fig. 5: Variation of (a) Crystallite size and (b) Lattice strain as a function of milling time.

Lattice strain is increases with milling time as shown in Figure 5b. It can be seen in that lattice strain is increases up to maximum value that is after 8 hours in our case and after that it remains constant. The increase in lattice strain with time is due to distortion effect caused by dislocation in lattice. Increase in lattice strain is more rapid in the case of Al-10 wt. % Al_2O_3 powder composite during milling.

IV. CONCLUSIONS

Al-10 wt. % Al_2O_3 powder composite was successfully synthesised by mechanical alloying. Characterisation of milled powder by SEM has confirmed the uniform distribution of

alumina particles in Al matrix after12 hours of milling. Homogeneity of particle size is increases with increase in milling. It was found that the milling stages include plastic deformation, welding and fracture of particles.

Addition of alumina powder has great influence on the morphological and structural characteristics of powder composite. The addition of hard particles accelerates the milling process, leading to faster work hardening rate and fracture of the aluminium powder.

X-ray patters shows increase in broadness of peaks with increase in milling which is due to increase in lattice strain induced due to plastic deformation and decrease in crystallite size. The crystallite size decreases with milling time and addition of alumina increase the rate of reduction in crystallite size. With increase in milling time lattice stain increases and after getting a maximum value it remain constant. Addition of alumina increases the lattice strain as compared to monolithic Al which is due to distortion effect caused by dislocation of lattice and brittleness induced in the matrix due to alumina particles.

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REFERENCES

[1] F. Y. C. Boey, Z. Yuan and K. A. Khor , "Mechanical alloying for the effective dispersion of sub-micron SiCp reinforcements in Al–Li alloy composite", Materials Science and Engineering A, 1998, Vol. 252, pp. 276-287.

[2] N. Parvin , R. Assadifard, P. Safarzadeh, S. Sheibani and P. Marashi, "Preparation and mechanical properties of SiC-reinforced Al6061 composite by mechanical alloying", Materials Science and Engineering A, 2008, Vol. 492, pp. 134-140.

[3] T.W. Clyne and P.J. Withers, "An Introduction to Metal Matrix Composites", Cambridge, UK, 1995.

[4] T. Graziani, A. Bellosi and D.D. Fabbriche, "Effects of some iron-group metals on densification and characteristics of TiB2", International Journals of Refractory Metals and Hard Materials, 1992, Vol. 11, pp.105–112.

[5] G. Jiang, G. Daehn, and R. H.Wagoner, "Inclusion Particle Size Effects on the Cyclic Compaction of Powder Composites", Scripta Materialia, 2001, Vol. 44, pp. 1117-1123.

[6] N. A. Travitzky, "Effect of Metal Volume Fraction on the Mechanical Properties of Alumina/ Aluminium Composites", Journals of Material Science, 2001, Vol. 36, pp. 4459.

[7] H. Lu, J. Hu, C. Chen, H. Sun, and X. Hu,, "Characterisation of Al2O3-Al Nano-Composites Powder Prepared by a Wet Chemical Method", Ceramics International, 2005, Vol. 31, pp. 481.

[8] M. Ferry and P. R. Munroe, "Enhanced Recovery in a Particulate-Reinforced Aluminium Composite", Material Science and Engineering A, 2003, Vol. 358, pp.142-151.

[9] X. X. Yu and W. B. Lee, "The Design and Fabrication of an Alumina Reinforced Aluminium Composite Material", Composites A, 2000, Vol. 31, pp. 245-258.

[10] V. K. Lindroos and M. J. Talvitie, "Recent Advances in Metal Matrix Composites", Journals of Material Processing Technology, 1995, Vol. 53, pp. 273.

[11] C. William Jr and T. Harrigan, "Commercial Processing of Metal Matrix Composites", Material Science and Engineering A, 1998, Vol. 244, pp.75.

[12] H. Mahboob, S. A. Sajjadi and S. M. Zebarjad: Proc. ICMN'08 Conf., Kuala Lumpur, Malaysia, May 2008, ICMN, 127–132.

[13] S.S. Razavi, R. Yazdani Rad and E. Salahi, "Structural Evolution of Al-20% (Wt.) Al2O3 System during Ball Milling Stages", Metal Transaction B, 2009, Vol. 22, No. 2, pp. 169-178.

[14] Y. Mazaheri, F. Karimzadeh and M.H. Enayati, "Nanoindentation Study of Al356-Al2O3 Nanocomposite Prepared by Ball Milling", Material Science and Applications, 2010, Vol. 1, pp. 217-222.

[15] S.A. Sajjadi, H. Mahboob and S.M. Zebrjad, "Influence of Nano-sized Al2O3 Weight Percentage on Microstructure and Mechanical Properties of Al-matrix Nanocompostie", Powder Metallurgy, 2011, Vol. 54, No. 2, pp. 148-152.

[16] K. M.Shorowordi, T. Laoui, A.S.M.A. Haseeb and J.P. Celis, "Microstructure and Interface Characteristics of B4C, SiC and Al2O3 Reinforced Al Matrix Composite: a Comparative Study", Journals of material process technology, 2003, Vol. 142, pp. 738-743.

[17] K. D. Woo and H. B. Lee, "Fabrication of Al alloy matrix composite reinforced with subsive-sized Al_2O_3 particles by the in situ displacement reaction using high-energy ball-milled powder," Material Science and Engineering A, 2007, Vol. 449-45, pp. 829–832.

[18] D. L. Zhang, J. Liang and J. Wu, "Processing Ti_3Al–SiC nanocomposites using high energy mechanical milling," Material Science and Engineering A, 2004, Vol. 375, pp. 911-916.

[19] G. O'Donnell and L. Looney, "Production of aluminium matrix composite compon-ents using conventional PM technology," Material Science and Engineering A, 2001, Vol. 303, pp. 292–301.

[20] A. Alizadeh, E. Taheri-Nassaj and H. R. Braharvandi, "Preparation and Inve-stigation of Al-4 wt % B_4C Nano-composite Powder using Mechanical Milling", Bull. Material Science, 2011, Vol. 34, No. 5, pp. 1039-1048.