Mechanics of Advanced Composite Structures 2 (2015) 73-78

Mechanics of Advanced Composite Structures

journal homepage: http://macs.journals.semnan.ac.ir

Semnan University

# Effect of Particle Size on the Structural and Mechanical Properties of Al–AlN Nanocomposites Fabricated by Mechanical Alloying

**H. Ghods[a], S.A. Manafi[a], E. Borhani[b*]**

[a]*Department of Materials Engineering, Islamic Azad University of Shahrood Branch, Shahrood, Iran*

[b]*Department of Nano-Technology, Nano-Materials Science and Engineering Group, Semnan University, Semnan, Iran*

**PAPER INFO**

***Paper history:***
Received 14 Febrruary 2015
Received in revised form 26 September 2015
Accepted 25 October 2015

***Keywords:***
Mechanical properties
Powder characteristics
Al/$B_4C$ nanocomposite
Mechanical alloying

**ABSTRACT**

Nanostructured Al composites with 2.5 wt.% aluminum nitride (AlN) were fabricated by powder metallurgy using mechanically milled aluminum powder mixed in a planetary ball mill with different particle sizes of AlN (50 nm and 1 μm) as reinforcement. After 20 h milling, the powders were die-pressed uniaxially in a steel die and then sintered at 670 °C for 2 h. The morphologies and properties of the obtained powders were determined by scanning electron microscopy and X-ray diffraction analysis. The results have indicated that the crystallite sizes of the composites decreased by increasing the milling time, resulting in sizes of 46 nm and 55 nm for the composites containing large (1 μm) and small (50 nm) AlN particles, respectively. After 20 h of milling, the microhardness of the nanocomposites with AlN particle sizes of 1 μm and 50 nm were 101 and 122, respectively. The flexural strength of the composite containing smaller AlN particles (50 nm) was higher than that of the composite containing larger AlN particles (1 μm). The testing results have indicated that the strength and hardness properties of the composite containing smaller AlN particles are better than those of the composite with larger AlN particles.



## 1. Introduction

Aluminum matrix composites (AMCs) have attracted much attention in automotive, aerospace, and military industries due to their advantageous mechanical and physical behavior [1, 2]. The reinforcement of metal matrix composites with ceramic particles results in a combination of the metallic properties (ductility and toughness) and the ceramic properties (strength and high modulus) [3-5]. The reinforcing particulates used for Al-matrix composites are always SiC, Al2O3, AlN and TiC etc., and have the particle size within decades micrometers. The tensile strengthand elastic modulus of AlN/Al composite materials increases obviously with the increase in the volume fraction of AlN particles, but the related plasticity reduces sharply. The coarse particles will induce the stress concentration and increase the residual stress in the fabricated products. The residual stress makes the size of product unstable. The investigations on the Al-matrix composites reinforced by submicron (0.1–1 lm) and nanometer (<0.1 lm) particulates are widely studied [6–9]. Aluminum nitride (AlN) is a good example of a ceramic reinforcement material; its appropriate physico-chemical, mechanical, and thermal properties make it a suitable candidate as a reinforcement material for aluminum [10].

Mechanical alloying (MA) has been used to achieve the homogeneity of particle distribution throughout a matrix [11, 12]. MA involves two opposing processes, namely, cold welding and fracturing. Stearic acid is used as a process control agent (PCA) to nullify the forces of the cold welding during MA.

*Corresponding author, Tel.: +98-33-654119; Fax: +98-33-654119

*E-mail address:* e.borhani@semnan.ac.ir

This technique, first developed by Benjamin [13] to produce nickel super alloys hardened by oxide dispersion, consists of the repeated welding–fracturing–welding of a mixture of powder particles in a high-energy ball mill. The process is used to produce a variety of materials and alloys: supersaturated solid solutions, amorphous and nanostructured materials, intermetallic compounds and metal matrix composites (MMCs) [14]. To obtain bulk composites from mechanically alloyed powders, several consolidation techniques have been employed, such as sintering [15], hot-pressing [16], severe plastic deformation [17], and hot extrusion of loose or prepressed powders [18]. The purpose of this study is to investigate the effects of the powder characteristics on the microstructures and mechanical properties of composites prepared by MA–cold pressing technique.

Wang et al. fabricated AlN/Al composite by means of a combined method of wet mixing, cold isostatic pressing and hot extrusion [19]. They observed that in compare with pure aluminum, the composite has better mechanical properties, lower coefficient of thermal expansion and higher dimensional stability.

Abdoli et al. made Al–AlN-nanostructured composites via 25 h high energy ball-milling of powder mixture with the different content of AlN, followed by cold press and sintering. The sinterability, tribilogical and corrosion behavior of composites were investigated at predefined conditions [20]. They observed that sinterability of composites is degraded by increasing the reinforcement content and wear resistance is improved by increasing the volume fraction of AlN and this improvement was more pronounced at the higher fraction of reinforcement.

In the present study, aluminum matrix reinforced with 2.5%wt. of AlN particles produced via mechanical alloying (MA) and cold pressing. Mechanical and structural properties of bulk nanocomposites such as crystal size, lattice strain of matrix alloy and microhardness in different periods have been studied.

## 2. Materials and experimental procedure

High-purity spherical shaped aluminum powders (Al) with an average particle size of 5 μm (Fig. 1a) and AlN particles were used as starting materials. The size of the AlN particles are 1 μm (composite 1; Fig. 1b) and 50 nm (composite 2; Fig. 1c).

The AlN particles were mixed with the aluminum powder and mechanically were milled to produce Al–AlN composites containing 2.5 wt. % of AlN as reinforcement. To minimize the extreme cold welding of the aluminum powders, 2 wt. % of stearic acid was used as a process control agent. Ball milling was executed at a rotation speed of 270 rpm, and the ball–powder weight ratio was 7:1. In this order, a hardened chromium steel vial containing ten steel balls (high chromium–carbon steel) was used. The vial was evacuated and then filled with the pure argon gas to prevent oxidation during the milling process. The composite powders were compacted by cold pressing at a pressure of 200 MPa. The as-milled powders were then heated at 670 °C under argon atmospher for 2 h. The samples were withdrawn at time intervals of 5, 10, 15, and 20 h for morphological and structural studies. The X-ray diffraction (XRD) patterns of the powders in air were collected using a Philips X-ray diffractometer with CuK$\alpha$ radiation. The average crystal sizes and lattice strains were obtained using the Williamson–Hall equation;

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

where $\beta$, $\lambda$, $\theta$, $D$, and $\eta$ are the full width at half maximum (FWHM), wavelength, peak position, crystal size, and lattice strain, respectively [21, 22]. Morphological characterization of the specimens was carried out using scanning electron microscopy (SEM; MIRA\\TESCAN). A schematic illustration of the preparation process of Al–AlN composites is shown in Fig. 2.

## 3. Discussions

### *3.1 Morphological evolution of mixed powders during milling process*

Fig. 3 shows the SEM micrograph of composite powders containing micro sized AlN particles (1 μm) (composite 1) after several milling times. Despite the ductile nature of the Al powder [23], the presence of AlN particles prevents the formation of large flake-like particles [23]. As shown in Fig 3(a), the resultant particles after 5 h milling are not as flat as the unreinforced Al particles. As the milling time increases to 10 h, the aspect ratios of the irregular particles gradually decrease (Fig. 3(b)). After increasing the milling time to 15 h, equiaxial particles with stable shapes and a more normal size distribution are almost formed (Fig. 3(c)).

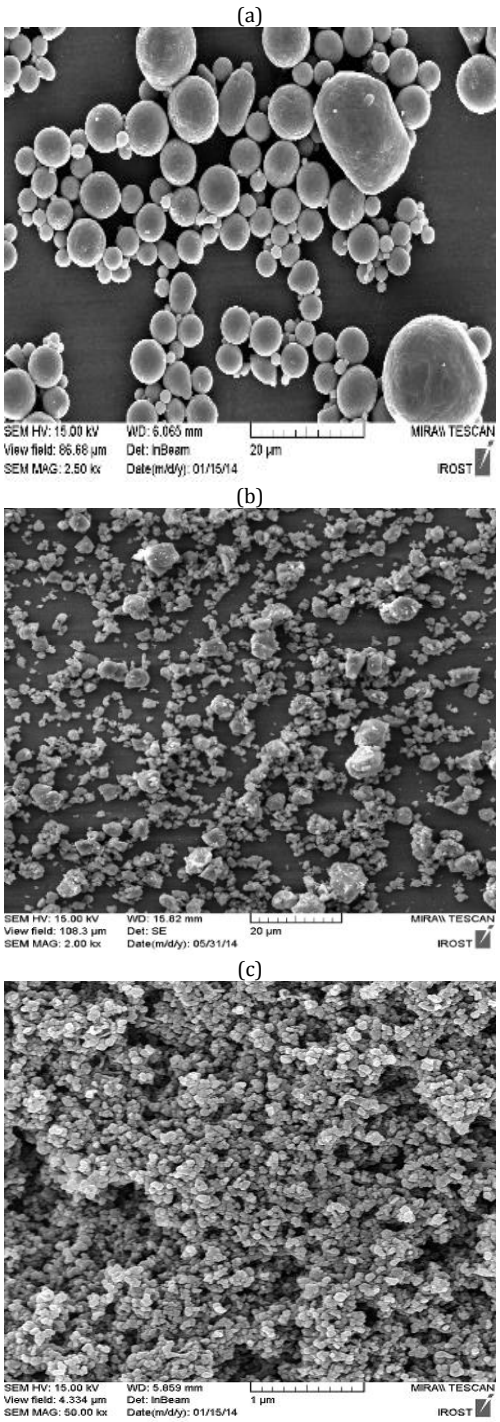


**Figure 1.** Morphology of as-received powder particles: (a) SEM micrograph of Al, and (b) AlN (1µm), (c) AlN (50 nm) powders.

Milling for longer than 20 h had no further effect on the morphology (Fig. 3(d)). It is clear that the presence of AlN particles decreases the necessary milling time and also decreases the time needed to reach steady-state which confirms the result of previous study [24].

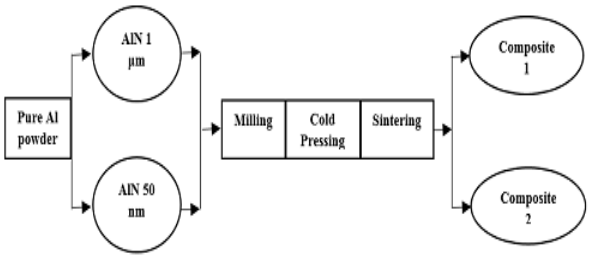


**Figure 2.** The illustration of the preparing process of Al/AlN composite

### *3.2 Structural characterization*

Fig. 4 shows the XRD patterns of composite powders containing micro sized AlN particles (1 µm) (composite 1) milled for different times, revealing the structural evolution of the powder mixtures as the milling process progresses. As shown in this figure, by increasing the milling time, the peaks gradually broaden, and their intensities decrease.

This noteworthy phenomenon (peak broadening) is probably due to the decrease in the crystallite size and the increase in the lattice microstrain, which has been previously reported by Alizadeh et al. and Sharifi et al. [23, 25]. The XRD pattern of composite 1 and composite 2 containing micro sized (1 µm) and nano sized (50 nm) AlN particles after 20 h milling is shown in Fig. 5, indicating the effect of particle size on intensity of XRD peaks. The peak intensities of composite 2, which contains smaller particles is higher than that of composite 1. The effect of sintering on composite structure is shown in Fig. 6. The $Al_2O_3$ peaks appear in the composites after sintering, probably due to the high reactivity of the aluminum powders, which are easily oxidized during sintering. The intensity of $Al_2O_3$ peaks in composite 2 is higher than that of composite 1, which is probably due to the high surface area of composite 2 compared to composite 1.

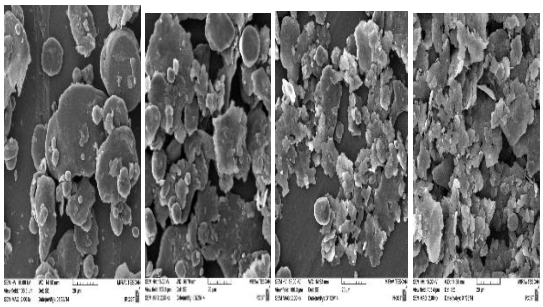

**Figure 3.** Morphology of composite powder containing micro sized AlN particles (composite 1) after (a) 5 h, (b) 10 h, (c) 15 h and (d) 20 h milling.

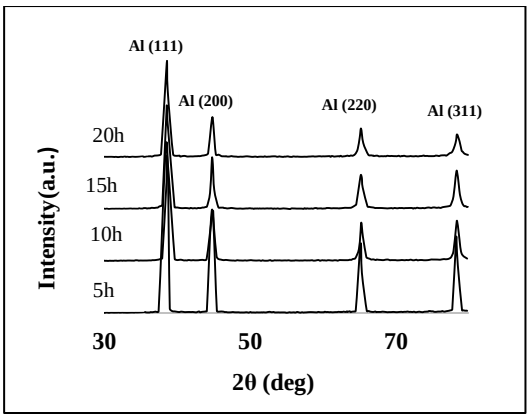


**Figure 4.** X-ray diffraction patterns of Al-2.5%$B_4C$ nanocomposite powders after (a) 5 h, (b) 10 h, (c) 15 h, and (d) 20 h of milling time

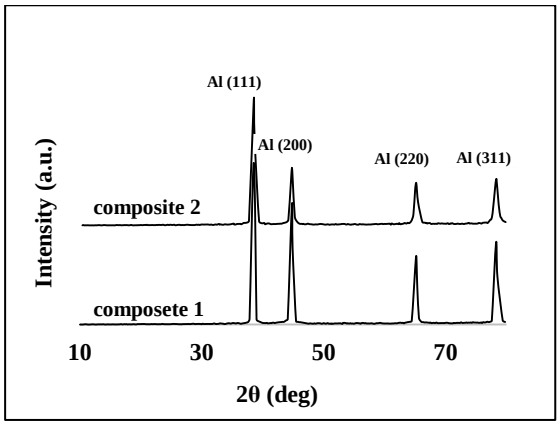


**Figure 5.** Comparison of X-ray diffraction pattern of composite 1 and composite 2 after 20 h milling

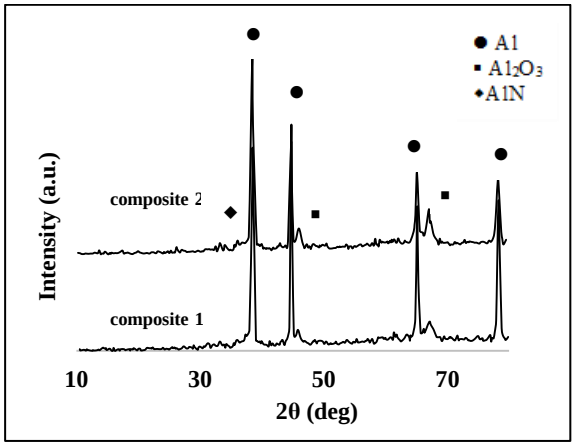


**Figure 6.** Comparison of X-ray diffraction patterns of composite 1 and composite 2 after Sintering

### *3.3. Crystallite size and lattice strain*

Crystallite size and lattice strain are important parameters for milled powders since they have a significant effect on compacting of the powders during sintering and the properties of obtained Al matrix strengthened by fine dispersions [26]. The average crystallite size of the Al matrix in the composites was estimated using XRD result. The crystallite size and the lattice strain were calculated based on Williamson–Hall plots; the results are shown in Table 1. Williamson-Hall (W-H) analysis is a simplified integra l breadth method where both size-induced and strain-induced broadening is decon-voluted by considering the peak width as a function of $2\theta$ [27].

The results indicate that the crystallite size decreases by increasing the milling time. This variation can be explained by the sever deformation of the powder particles that occurs with increasing the milling time, lead to an increase in the crystalline defects including point defects and dislocations [26]. The effect of increasing dislocation density on strengthening mechanism has been previously reported [28].

The presence of defects indeed results in an increase in the system energy and the lattice strain. On the other hand, after high energy milling (20 h), the crystallite sizes are 55 nm and 46 nm for composite 1 and composite 2, respectively. During the MA process, additional deformation of the matrix alloy is imposed by the hard ceramic particles, as reported by Abdoli et al. [28]. As milling time increases, severe plastic deformation brings about a deformed lattice with a high density of dislocations [30, 31].

### *3.4. Mechanical properties of the composites*

The hardness value of the alloy as a function of milling time is shown in Fig. 7. As shown in this figure, the hardness improves by increasing the milling time. The average hardness values of composite 1 and composite 2 after 20 h are 101 HV and 122 HV, respectively.

Table 2 shows the flexural strength of the two composites after 5 and 20 h of milling. The same tendency is observed in flexural strength of composites. The specimens containing smaller AlN particles (composite 2) exhibit improved powder density and compressibility, resulting in higher hardness value and flexural strength than that of composite 1. It has been reported that [32] the smaller particles normally has an advantage in a higher density. Therfore, the prior particle boundary (PPB), which comes from the packing of various sizes of powders, usually affects the mechanical property of the composite and increases the hardness and strength in the composite containing smaller particles.

**Table 1.** The crystallite size of powder and lattice micro-strain for composite 1 and composite 2

| Lattice strain (%) | Crystallite size (nm) | Milling time (h) | Composition |
|---|---|---|---|
| 0.00110 | 72 | 5 | |
| 0.00115 | 55 | 10 | Composite 2 |
| 0.00125 | 51 | 15 | |
| 0.00140 | 46 | 20 | |
| 0.00125 | 63 | 5 | Composite 1 |
| 0.00165 | 55 | 20 | |

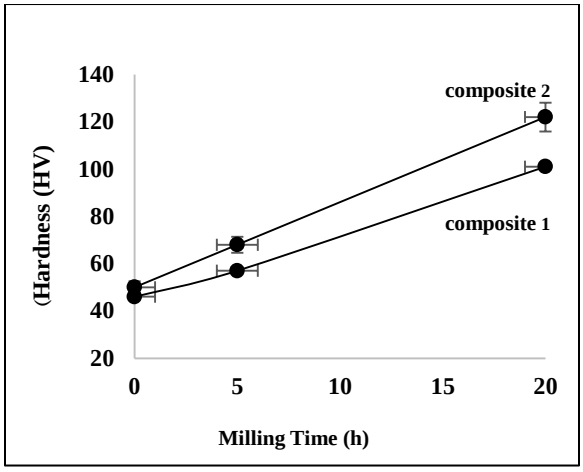


**Figure 7.** Averaged hardness values of composite 1 and composite 2

**Table 2.** Flexural strength values of composites

| Flexural strenght (MPa) | Milling time (h) | Composition |
|---|---|---|
| 5 | 145 | Composite 2 |
| 20 | 204 | |
| 5 | 99 | Composite 1 |
| 20 | 153 | |

## 4. Conclusions

Al–AlN nanostructured composites were made by the high energy ball milling (20 h) of a powder mixture with 2.5% wt. content of AlN followed by cold pressing and sintering. The properties of the specimens were evaluated by XRD and SEM, and the flexural strength and hardness of the composites were also determined. During the mechanical alloying, the crystallite size decreases by increasing the milling time. After 20 h of milling, the crystallite sizes of composites 1 and 2 were 55 nm and 46 nm, respectively. The mechanical properties of the specimens were studied using hardness and flexural strength tests. The microhardness of composite 1 and composite 2 after 20 h of milling were 101 and 122, respectively. The flexural strength of the composite containing smaller AlN particles (composite 2) was higher than that of the composite containing larger AlN particles (composite 1).

## References

[1] Abdoli H, Asgharzadeh H, Salahi E. Sintering behavior of Al–AlN-nanostructured composite powder synthesized by high-energy ball milling. *J Alloy Compd* 2009; 473: 116-122.

[2] Smagorinski ME, Tsantrizos PG, Grenier S, Cavasin A, Brzezinski T, Kim G. The properties and microstructure of Al-based composites reinforced with ceramic particles. *Mater Sci Eng A* 1998; 244: 86-90.

[3] Sajjadi A, Ezatpour HR, Beygi H. Microstructure and mechanical properties of Al–$Al_2O_3$ micro and nano composites fabricated by stir casting. *Mater Sci Eng A* 2011; 528: 8765-8771.

[4] Ezatpour HR, Beygi H, Sajjadi SA, Torabi M. Microstructure and mechanical properties of Al–$Al_2O_3$ micro and nano composites fabricated by a novel stir casting rout. 2nd Conferences on Application of nanotechnology in Science, *Engineering and Medicine, Mashhad- Iran* 2001.

[5] Comoro J, Salvador MD, Cambronero LEG. High-temperature mechanical properties of aluminium alloys reinforced with boron carbide particles. *Mater Sci Eng* 2009; 499: 421-426.

[6] Han BQ, Agnew SR, Dunand DC. High-strain-rate deformation of pure aluminum reinforced with 25% alumina submicron particles near the solidus temperature. *Scripta Mater* 1999; 40: 801–8.

[7] Kim SS, Haynes MJ, Gangloff RP. Localized deformation and elevatedtemperature fracture of submicron-grain aluminum with dispersoids. *Mater Sci En A* 1995; 203: 256–71.

[8] Xiao YL, Li YL, Liang Y, Lu K, Zhou BL. Nanometre sized SiC particulates reinforced Al base composite material. *Acta Metall Sin* 1996; 6: 658–62. In Chinese.

[9] Wu GH, Ma SL, Zhao YC, Yang DZ. Microyield deformation characteristic of Particle with Submicron scale reinforced 6061 Al matrix composite. *Chinees J Mate Res* 1998; 3: 307–10. in Chinese.

[10] Troadec C, Goeuriot P, Verdieq P, Laurent Y, J. Vicenqc, Boitier G, Chermantc JL. AlN dispersed reinforced aluminum composite. *Eur Ceram Soc* 1997; 17: 1867-1875.

[11] Fogagnolo JB, Ruiz-Navas EM, Robert MH, Torralba JM. 6061 Al reinforced with silicon nitride particles processed by mechanical milling. *Scripta Mater* 2002; 47: 243-248.

[12] Fogagnolo JB, Robert MH, Ruiz-Navas EM, Torralba JM. 6061 Al reinforced with zirconium diboride particles processed by conventional powder metallurgy and mechanical alloying. *Mater Sci* 2004; 39: 127-132.

[13] Benjamin JS. Mechanical Properties of Metallic Composites. *Metall Transaction*, 1970; 1: 2943-2951.
[14] Suryanarayana C. Mechanical Alloying and Milling. *Prog Mater Sci* 2001; 46: 1-184.
[15] Lu L, Lai MO, Ng CW. Enhanced mechanical properties of an Al based metal matrix composite prepared using mechanical alloying. *Mater Sci Eng A* 1998; 252: 203-211.
[16] Bhaduri A, Gopinathan V, Ramakrishnan P, Miodownik AP. Processing and properties of SiC particulate reinforced A1-6.2Zn-2.5Mg-I.7Cu alloy (7010) matrix composites prepared by mechanical alloying. *Mater Sci En A* 1996; 221: 94-101.
[17] Fogagnolo JB, Kiminami CS, Bolfarini C, Botta Filho WJ. Consolidation of Mechanically Alloyed Aluminium Matrix Composite Powders by Severe Plastic Deformation. *JMNM* 2003; 307: 15-16.
[18] Fogagnolo JB, Robert MH, Torralba JM. "Mechanically alloyed AlN particle-reinforced Al-6061 matrix composites: Powder processing, consolidation and mechanical strength and hardness of the as-extruded materials." *Mater Sci Eng A* 2006; 426: 85-94.
[19] Wang J, Yi D, Su X, Yin F, Li H. Properties of submicron AlN particulate reinforced aluminum matrix composite. *Mater Design* 2009; 30: 78–81.
[20] Abdoli H, Saebnouri E, Sadrnezhaad SK, Ghanbari M, Shahrabi T. Processing and surface properties of Al–AlN composites produced from nanostructured milled powders. *J Alloy Compo* 2010; 490: 624–630.
[21] Razavi Hesabi Z, Simchi A, Seyed Reihani SM. Structural evolution during mechanical milling of nanometric and micrometric $Al_2O_3$ reinforced Al matrix composites. *Mater Sci Eng A* 2006; 428: 159-168.
[22] Simchi H, Kaflou A, Simchi A. Synergetic effect of Ni and $Nb_2O_5$ on dehydrogenation properties of nanostructured $MgH_2$ synthesized by high-energy mechanical alloying. *Int J Hydrogen Energ* 2009; 34: 7724-7730.
[23] Alizadeh A, Taheri-Nassaj E, Baharvandi HR. Preparation and investigation of Al–4 wt% $B_4C$ nanocomposite powders using mechanical milling. *Mater Sci* 2011; 34: 1039-1048.
[24] Abdoli H, Salahi E, Farnoush H, Pourazrang K. Evolutions during synthesis of Al–AlN-nanostructured composite powder by mechanical alloying. *J Alloy Compo* 2008; 461: 166-172.
[25] Mohammad Sharifi E, Karimzadeh F, Enayati M.H. Fabrication and evaluation of mechanical and tribological properties of boron carbide reinforced aluminum matrix nanocomposites. *Mater Design* 2011; 32: 3263-3271.
[26] Fathy A, Wagih A, M. Abd El-Hamid, Hassan A. Effect of Mechanical Milling on the Morphology and Structural Evaluation of Al-$Al_2O_3$ Nanocomposite Powders. *Int J Eng* 2014; 27: 625-632.
[27] VD Mote VD, Purushotham Y, Dole BN. Williamson-Hall analysis in estimation of lattice strain in nanometer-sized ZnO particles. *J theor appl phys* 2012; 1: 5-11.
[28] Borhani E. Microstructure and Mechanical Property of Heavily Deformed Al-Sc Alloy Having Different Starting Microstructures. PhD thesis, 2012, Kyoto University.
[29] Abdoli H, Farnoush HR, Asgharzadeh H, Sadrnezhaad SK. Effect of high energy ball milling on compressibility of nanostructured composite powder. *Powder Metall* 2011; 54: 24-29.
[30] Razavi Tousi SS, Yazdani Rad R, Salahi E, Mobasherpour I, Razavi M. Production of Al-20 wt.% $Al_2O_3$ composite powder using high energy milling. *Powder Tech* 2009; 192: 46-51.
[31] Nalwa HS. Nanoclusters and nanocrystals. *Am Sci Pub* 2003, los Angeles, California.
[32] Sanghoon N, Byoung-Kwon C, Sukhoon K, Taekyu K. Influence of mechanical alloying atmosphere on the microstructures and mechanical properties of 15Cr ODS steels. *Nucl Eng Tech* 2014; 46: 857-862.