



## The impact of matrix microstructure and reinforcement size (micron vs. nano-size) on the compressibility of Al-SiC powder mixtures and hardness of Al/SiC composites

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### ABSTRACT

In the present study, four series of Al-6061/SiC composites synthesized via powder metallurgy technique were used to investigate the impact of the matrix grain size and/or the size of reinforcing particles on enhancing the compressibility of powder mixtures and hardness of the composites. In two series, the as-received Al powders have micron-sized grains mixed with either nano-sized or micron-sized SiC particles. For the other two series, the Al powders were initially milled to convert their grain size to nano-scale before mixing with either nano-sized or micron-sized SiC particles. The powder mixtures containing 1, 2, and 3 vol.% of SiC particles were cold pressed and hot extruded. The decreased compressibility in all of the four series of Al/SiC powder mixtures with increased SiC content attributed to the enhanced portion of the hard and non-deformable SiC particles in the powder mixtures resulting in a reduced degree of plastic deformation. Increment the SiC<sub>n</sub> content from 0% to 3% resulted in a significant increase in the microhardness of 20h planetary ball milled powders accompanied with decrease compressibility of powders. The composites were subjected to Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), and X-ray diffraction (XRD) studies as well as density and hardness measurements. Metallographic studies and density measurements confirmed significant densification with no indication of voids in the samples' microstructures after hot extrusion. The results revealed that matrix microstructure, as compared with the size of the reinforcing particles, was more influential in enhancing the powder compressibility and composite hardness.

**Keywords:** Aluminum matrix composite, SiC, Matrix microstructure, Particle size, Compressibility, Hardness

### 1. Introduction

Aluminum matrix composites (AMCs) reinforced with ceramic particles offer several advantages over conventional alloys, including high modulus, superior creep and wear resistance as well as high strength to weight ratio [1]. The characteristics of reinforcement particles significantly affect the mechanical properties of AMCs. For instance, the increased volume fraction and/or decreased reinforcement particle size results in increased yield and tensile strength of such

composites at the expense of deteriorated toughness and ductility. It has been reported that by reducing the reinforcement particle size to nanoscale range, the strength of the composites can be enhanced by 3 times together with 10 times enhancement in ductility [2]. Such properties are mainly important for structural applications requiring high ductility and strength. In fact, smaller particles are less liable to internal defects, and hence they are less disposed to fracture. In addition, because of the increment number of reinforcing particles at a constant

particle content, the stress concentration level on each particle is decreased that in turn lowers the possibility of particle fracture. Nevertheless, for synthesizing nanocomposites with acceptable mechanical properties, the large surface area of nano-sized reinforcing particles imposes a great tendency for clustering. Therefore, the uniform dispersion of such particles throughout a metallic matrix is a challenging task [3-5]. Actually, the reinforcement clusters have less ability to transfer tensile and shear stresses. The increased volume fraction of reinforcing particles, particularly beyond a percolation threshold, results in severe clustering accompanied by impeding the achievement of a uniform distribution of particles throughout the matrix. For the production of metal matrix composites (MMCs), powder metallurgy (P/M) methods do not have the typical drawbacks of casting routes [1, 6]. However, the clustering of nanoparticles in conventionally P/M processed nanocomposites is inevitable [7]. Kang and Chan [8] reported the improved mechanical properties of Al-based nanocomposites containing up to 4vol% of alumina nanoparticles synthesized via the conventional P/M method. However, the higher nanoparticle loadings deteriorated the composite properties due to agglomeration. Mechanical milling (MM) is a useful technique for the fabrication of nanocomposites with a uniform distribution of the reinforcing nanoparticles [9-11]. This method is also capable of generating nano-crystalline structured materials with high thermal stability [12]. MM is a suitable technique for improving the reinforcing particle distribution in both micro and nanocomposites [1, 13, 14]. High-energy ball milling processes utilizing planetary ball mills, SPEX shaker mills, and attrition mills have been used as non-equilibrium mechanical processing techniques for inducing solid-state reactions in various alloy systems and obtaining nano-crystalline materials. The contribution of such ultrafine matrix structures, together with the nano-sized reinforcing particles, can enhance the mechanical properties of particle reinforced MMCs [15].

The effect of refined grain structure and/or nano-sized reinforcements on enhancing the materials' mechanical properties was reported by some researchers [16-18]. Jia [16] indicated that the fracture strength of Al-6Ti6Nb/SiC composites enhanced by almost 32% via declining the size of the reinforcement particles from 300 to 30 nm.

Fu et al. [17] reported that the tensile strength of Al6061 improved from 110 to 340 MPa as the alloy grain size declined from 66 to 0.86  $\mu\text{m}$  through severe plastic deformation (SPD). Significant improvement of the tensile strength and hardness of Al6061-5 wt.%  $\text{Si}_3\text{N}_4$  composite was achieved via grain refinement utilizing a combination of mechanical milling and hot powder extrusion [18].

It is well known that for synthesizing AMCs, during MM of a mixture of Al powders and nano-sized particles, both the de-agglomeration of reinforcing particles and refining the alloy structure occur simultaneously. Therefore, the uniform dispersion of particles throughout a grain refined matrix alloy enhances composite properties [9]. However, any report on the degree of contribution of (i): the size of the reinforcing particles and (ii): the grain size of the matrix alloy to the improved composite properties seems to be lacking. The purpose of the present paper is to cover this issue.

## 2. Materials and Methods

The base material used in the present study is nitrogen gas atomized aluminum 6061 (Al-6061) with a particulate size range of 38-63  $\mu\text{m}$  having the nominal chemical composition (wt.%) as shown in Table 1. Figure 1 displays the typical SEM and TEM micrographs of these powder particles in the as-received condition. Nanoparticles of silicon carbide ( $\text{SiC}_n$ ) in 25-50 nm size range provided by Plasma-Chem. Co., Germany and micron-sized SiC particles ( $\text{SiC}_m$ ) with the average size of 35  $\mu\text{m}$  were used as the reinforcing materials.

Four series of composites were prepared by using (a) un-milled Al powders and  $\text{SiC}_m$  (CG-Al/ $\text{SiC}_m$ ), (b) planetary ball milled Al powders and  $\text{SiC}_m$  (NS-Al/ $\text{SiC}_m$ ), (c) attrition-milled Al powders and  $\text{SiC}_n$  (CG-Al/ $\text{SiC}_n$ ), and (d) planetary ball milled Al powders and  $\text{SiC}_n$  (NS-Al/ $\text{SiC}_n$ ) where CG-Al and NS-Al denote coarse-grained aluminum and nanostructured aluminum respectively. For all the composite series, the reinforcement contents were in the range of 1-3 vol.%.

For the preparation of CG-Al/ $\text{SiC}_m$  samples, the weighted amounts of as-received Al powders and micron-sized SiC particles were blended in ethanol media for 5min in a laboratory blender to gain a

Table 1- Chemical composition (wt. %) of 6061 Aluminum alloy

Mg	Si	Fe	Cu	Cr	Al
1.12	0.64	0.48	0.33	0.04	Balance

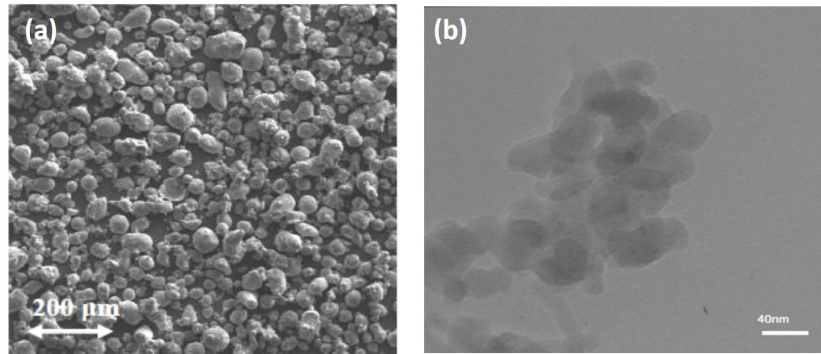


Fig. 1- (a) SEM micrograph of Al-6061, and (b) TEM micrograph of the used nano SiC powder particles.

uniform distribution of  $\text{SiC}_m$  within the powder mixture.

In order to prepare NS-Al/ $\text{SiC}_m$  samples, initially, the Al powders were milled for 20h [9] under argon media in a laboratory planetary ball mill (PM-2400) using hardened steel balls with diameter of 10mm and a hardened stainless steel vial. The rotational speed and the ball-to-powder weight ratio (BPR) were 300 rpm and 15:1, respectively, and 1.5 wt.% of stearic acid (S.A.) was used as the Process Control Agent (PCA). Then the milled Al powders mixed with  $\text{SiC}_m$  particles using the same procedures as mentioned above for the CG-Al/ $\text{SiC}_m$  samples. For the preparation of NS-Al/ $\text{SiC}_n$ , Al powders and the required amount of  $\text{SiC}_n$  commilled in a laboratory planetary ball mill for 20h under the abovementioned conditions.

For synthesizing CG-Al/ $\text{SiC}_n$  samples, at first, the nano-sized SiC powders de-agglomerated by ultrasonic agitation in ethanol for 22min. Then, the required amount of the as-received Al powders was added to the suspension and the resultant slurry milled in a low energy attrition mill for 4h using 5mm diameter hardened steel balls and 320 rpm rotational speed. The aim of this procedure was to gain a uniform distribution of SiC nanoparticles in the powder mixture.

The powder mixtures were cold pressed at a constant pressure of 750 MPa in a steel die using a 45t hydraulic press. Furthermore, the dimensions of the compacts were 15 and 25 mm in height and diameter, respectively. Green compacts held at 500 °C for 45min and hot extruded at this temperature using the above mentioned hydraulic press at a ram speed of 36 mm/min.

The morphology of powders at different milling time steps were determined using a scanning electron microscope (CamScan MV2300, UK).

X-ray diffraction (XRD) analysis was used to evaluate the crystalline size and the internal stress within the powders by a Philips X'Pert MPD diffractometer with Cu K $\alpha$  radiation utilizing the Williamson–Hall method [9] by means of the following equation:

$$B_s \cos\theta = \frac{K\lambda}{d} + 2\epsilon \sin\theta \quad (1)$$

where  $\lambda$  is the wavelength of the X-ray radiation,  $d$  and  $\epsilon$  are crystalline size and internal strain, respectively,  $\theta$  is the Bragg angle.  $K$  is a constant equal to 0.9 and  $B_s$  is the sample broadening related to FWHM (the width of the diffraction peak, in radians, at a height half-way between the background and the peak maximum) and could be measured by removing the instrument broadening ( $B_i = 0.01$ ) by the following equation:

$$B_s^2 = B_e^2 - B_i^2 \quad (2)$$

The microhardness values of powders measured on the polished surfaces utilizing a micro-hardness tester (Wolpert D-6700 Vickers, Germany) at a load of 1 Kg for specified time of 10 s. The density values of the samples were measured using Archimedes' principle according to ASTM B325 standard. The samples were precision weighed in distilled water and air using an electronic balance to an accuracy of 0.1 mg. then the density values obtained as follows [19]:

$$\rho = \frac{m_{\text{(in air)}}}{m_{\text{(in air)}} - m_{\text{(in water)}}} \quad (3)$$

where  $m_{\text{in air}}$  and  $m_{\text{in water}}$  are the mass of the sample in air and in water, respectively. Furthermore, the theoretical densities calculated according to the rule of the mixture using the volume fractions of

Al and SiC. Metallographic studies conducted on samples using a Transmission Electron Microscope (TEM) (Philips CM30, Poland) and a Hitachi S-2700 scanning electron microscope (SEM). The hardness of extruded samples was determined on a Brinell hardness testing machine, using 300 N load, and the average value of 5 hardness measurements conducted on each specimen was considered.

### 3. Results and Discussions

#### 3.1. Powder characteristics

It is well known that the mechanical milling of ductile metallic powders introduces plastic deformation, micro-welding, and fracture of the particles [10]. Our previous research showed the uniform distribution of SiC<sub>n</sub> in nano-crystalline Al powders after 20h milling [9]. A typical TEM image of a 20h milled NS-Al/SiC<sub>n</sub> nanocomposite powder containing 2vol.% of SiC<sub>n</sub> particles, as shown in Figure 2, confirms the homogeneously distributed SiC<sub>n</sub> in the Al matrix.

Figures 3a and b show the morphology of 20h mechanically milled NS-Al/SiC<sub>n</sub> powder mixtures containing respectively 1 and 3vol.% of SiC<sub>n</sub>

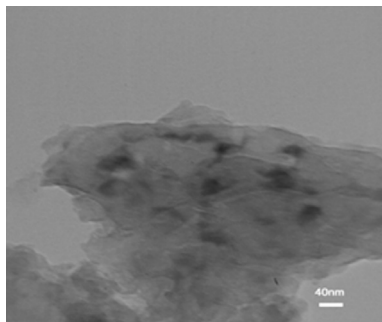


Fig. 2- TEM micrograph of the Al/2SiC nanocomposite powder.

particles. The results confirm that the increased SiC<sub>n</sub> content resulted in the decreased average size of the Al powders.

The presence of hard particles induces more local deformation of the matrix around the reinforcement particles that in turn enhances the work-hardening rate of the metallic matrix. The composite powders' fracture toughness is lower than the matrix material due to the altered slip

characteristics. Therefore, mechanical milling of Al and SiC powder mixtures is associated with accelerated fracture progression. Based on these speculations, increasing the amount of the reinforcement particles results in more frequent interactions between hard particles and dislocations, accelerating the onset of the fracture stage and contributing to the grain-refinement process [20].

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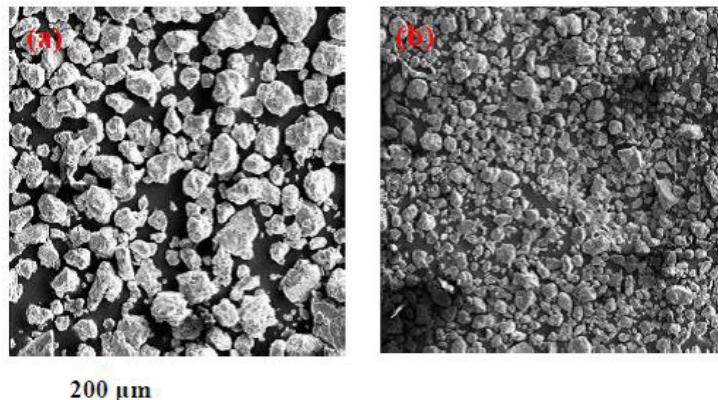


Fig. 3- Morphology of the powders after 20 h of milling: (a) NS-Al/1 vol.% SiC and (b) NS-Al/3 vol.% SiC.



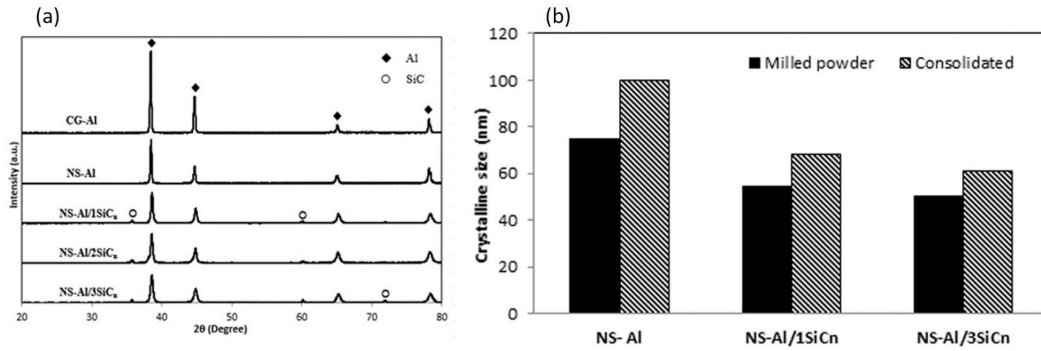


Fig. 4- (a) XRD pattern and (b) Crystallite size of Al matrix as a function of nano SiC content.

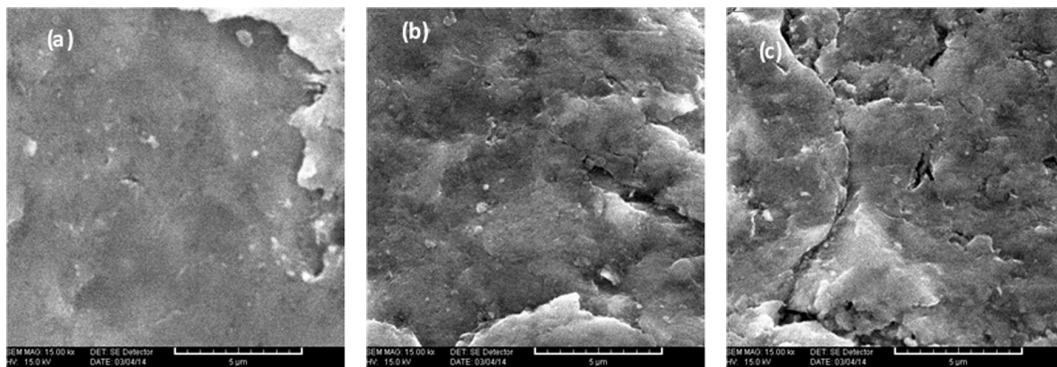


Fig. 5- SEM micrographs of Al-6061 powders co-attribution milled for 4h with (a)1, (b)2, and (c)3 vol.% of nano SiC particles.

Figure 4a reveals that the presence and increased content of hard SiC<sub>n</sub> particles during milling resulted in increased FWHM of Al peaks in the XRD pattern of powders confirming the increased amount of induced plastic deformation during milling. Consequently, the crystalline size of powders calculated by means of equations 1 and 2, as shown in Figure 4b, decreased leading to decreased size of generated nano-crystallines. This trend retained in the consolidated samples. However, hot extrusion of powder compacts resulted in grain growth of the matrix alloy, as revealed in this Figure.

Figure 5 displays the typical SEM micrographs of Al powders co-milled with different amounts of SiC<sub>n</sub> for 4h revealing the uniform dispersion of SiC nanoparticles on the surface of flattened Al powders. Therefore, the uniform distribution of SiC<sub>n</sub> in the consolidated CG-Al/SiC<sub>n</sub> samples anticipated.

Figure 6 shows the microhardness values of different powder samples co-milled with different amounts of SiC<sub>n</sub> particles. Increasing the SiC<sub>n</sub> content from 0% to 3% resulted in a significant increase in the microhardness of 20h planetary ball

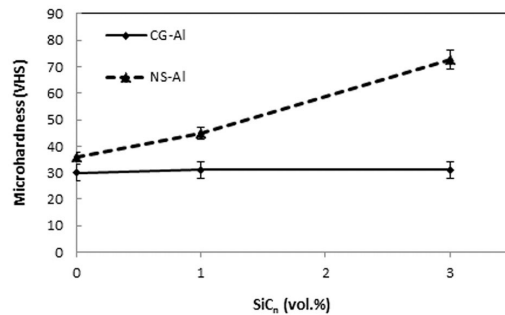


Fig. 6- The variation of the microhardness of CG-Al and NS-Al powder samples as a function of the percentage of the nanometric SiC particles.

milled Al (NS-Al) powders. However, for CG-Al powders co-attribution milled for 4h, identical microhardness obtained in all percentage range of added SiC nanoparticles. Furthermore, the results confirm our previous study [9], revealed that the SiC nanoparticles are embedded in the planetary ball milled Al powders (Figure 2) and contributed to their enhanced microhardness values. In the CG-Al/SiC powder mixtures, the presence of any amount of free SiC nanoparticles on the surface of flattened powders (Figure 5) did not change the hardness values.

### 3.2. Compressibility of powders

The characteristics of powders such as microstructure, size, size distribution, size, morphology, flowability, apparent density, and hardness affect the compaction behavior of P/M products [21, 22]. The materials and processing parameters of the powder production technique influence most of these characteristics. Generally, the powders' compressibility is characterized by enhancing green density after applying a special compaction pressure. Poquillon et al. indicated the declined compressibility of spherical particles compared to irregularly shaped [23]. Fogagnolo et al. [21] correlated the lower compressibility of spherical powders to the creation of symmetrical opposite forces at the contact points during plastic deformation, promoting only compressive deformation. Nevertheless, irregular particles bear asymmetrically opposing forces at contact points encouraging sufficient shear deformation and thus declined compacting pressure through pressing. Particle rearrangement and plastic deformation are two main stages of the cold compaction process. In the powder compaction's early stages, while the applied pressure is low, particle rearrangement and sliding are the principal compaction mechanisms. As the uniaxial compaction pressure increments, the particle movement is constrained, and the densification's second stage (i.e. plastic deformation) manages the consolidation mechanism [22]. The brittle and hard reinforcing ceramic particles distributed in the metallic matrix do not generally deform plastically. Thus, declined densification with increment reinforcement content is predicted. Densification is mostly controlled by the metallic powders' plastic deformation at relatively high pressures for these materials [22, 23].

In this study, the size and morphology of powders, i.e. semi-spherical vs. flake shaped (Figures 1a, 3, and 5), their different hardness values (Figure 6), the content and type of incorporation of SiC particles in the powder mixture (i.e. embedded within the matrix powders or blended with them) influenced the compressibility of powders. As shown in Figure 7, the compressibility of compacts in the CG-Al/SiC<sub>m</sub> series, due to their un-milled Al content, are higher than that of their counterparts in the other series. Attrition ball-milling as well as the presence of SiC<sub>n</sub> on the surface of powders (Figure 5), induced a significant decrease in the relative density of the compacts in the CG-Al/SiC<sub>n</sub> samples. Also, high-energy ball milling of powders for producing NS-Al/SiC<sub>m</sub> and NS-Al/SiC<sub>n</sub> samples resulted in further decrease in the compressibility of powders. These results are attributed to work hardening effects during powder milling.

The decreased compressibility in all of the four series of Al/SiC powder mixtures with increased SiC content attributed to the enhanced portion of the non-deformable SiC particles in the powder mixtures resulting in a reduced plastic deformation's degree. Furthermore, soft Al particles should undergo extra deformation to fill the voids between the hard inclusions with increment SiC content in the powder mixtures. The continuous network's formation made of the hard particles supports the applied pressure to some extent and declines the load transmission to the soft deformable particles. The percolating particle network formation is intensified with enhanced nanosized SiC particles contents in the powder mixtures.

The declined compressibility of NS-Al/SiC<sub>n</sub> nanocomposite powders with increment content of SiC<sub>n</sub> attributed to improved hardness

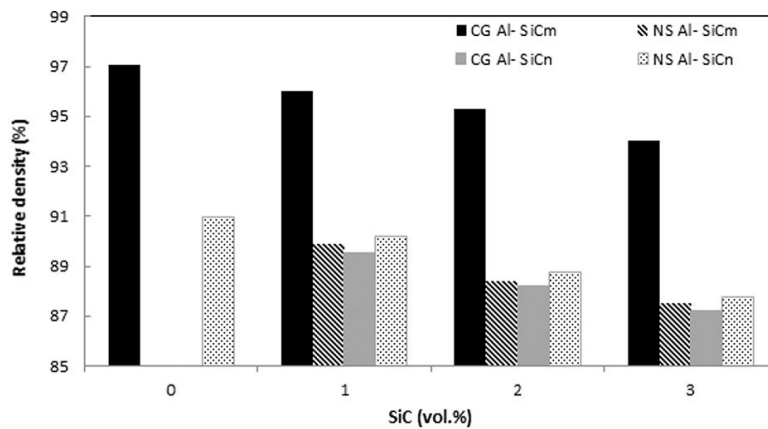


Fig. 7- Compressibility values for four different series of Al-SiC powder mixtures.

(Figure 6) and thus declined plastic deformation. According to Figure 7, monolithic CG-Al powders consolidated better than NS-Al particles. Nevertheless, the densification of NS-Al/SiC<sub>n</sub> nanocomposite powders compared to the CG-Al/SiC<sub>n</sub> powder mixture with the same percentage of SiC<sub>n</sub> particles was easier. For instance, NS-Al and CG-Al powder mixtures both containing 1 vol.% of SiC nanoparticles gained 91% and 88% of their theoretical green densities, respectively. These results indicate the more pronounced deleterious effect of SiC<sub>n</sub> particles on the powders' densification while these particles are not embedded within the metallic matrix powders.

### 3.3. Properties of the Extruded Composites

#### 3.3.1. Microstructure Observations

Density measurements confirmed significant densification of samples after hot extrusion. The porosity of hot extruded samples was mostly less than 0.3%, and full densification achieved for most of these samples. Our immense metallographic studies indicated no evidence of the presence of any cracks or pores in the composites. Figure 8 illustrates a typical SEM micrograph of the hot-extruded nanocomposite, indicates SiC<sub>n</sub> (white spots) uniformly distributed in the matrix. The uniform distribution of the reinforcing particles contributes to enhanced tribological and mechanical properties of the composites [3]. Moreover, repeated welding and fracture of aluminum particles encourage the uniform distribution of SiC particles during mechanical milling. Mechanical milling through repeated cold welding and fracture procedures results in the penetration of reinforcement particles into aluminum matrix particles (Figure 2). It intercepts their agglomeration and provides their separation in Al matrix grain boundaries, as confirmed by Abdollahi et al. [24] in Al-B<sub>4</sub>C nanocomposites. Our previous study [25] revealed the absence of any detrimental phases in the composite prepared via the same route, confirming the clean interface between Al and SiC particles. It is well known that the homogeneous distribution of the reinforcing phase led to the remarkable performance of composite materials. Any reinforcing particle agglomeration deteriorates the mechanical properties in particulate-reinforced composites. Development of any alteration in particle geometry, size, density, electrical charge, or flow during mixing can lead to particle agglomeration in the composites processed via

the P/M route. Reduction in the size of reinforcing particles contributed to enhancing the composite mechanical strength at the expense of particle clustering. The reinforcement and matrix mixing process is a critical step for achieving homogeneity of particle distribution throughout the matrix in powder metallurgy. When mechanical milling employed for mixing, the size of particles is also reduced (Figures 3a and b) [9]. Hot extrusion reduces the reinforcement particles' clustering and provides a better distribution through the metal matrix. In the present study, both the mechanical milling and hot extrusion processes contributed to the uniform distribution of SiC nanoparticles in the matrix alloy (Figure 8). Figure 4b reveals a slight increase in the crystalline size of the samples after hot extrusion attributed to grain growth. These results attributed to the presence of oxides and other dispersoids (i.e. some SiC nano-particles) at the boundaries retarding excessive grain growth via grain boundary pinning. This phenomenon can contribute to the thermal stability of such composites at elevated temperatures, as reported by Razavi Hesabi et al. [26]. Shafei-Zarghani [27] reported that smaller Al<sub>2</sub>O<sub>3</sub> nanoparticles (20 and 80nm) are more influential in inhibiting matrix grain growth at elevated temperatures in Ti-Al<sub>2</sub>O<sub>3</sub> nanocomposites.

In the present study, despite the presence of nanometer reinforcing particles that limited the movement of the grain boundaries (Zener pinning), grain growth occurred during hot extrusion. Grain growth occurs to reduce the stored energy in the material that was subjected to mechanical milling [28]. As the grains grow, the amount of high-energy

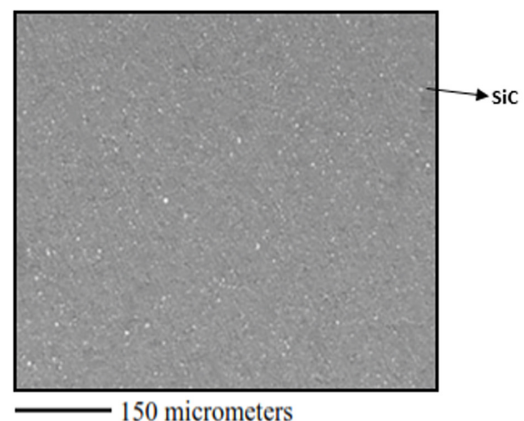


Fig. 8- Typical SEM micrograph of the polished surface of NS-Al/2SiC<sub>n</sub> nanocomposite. White spots indicate the SiC nanoparticles.

boundaries decreases resulting in decreased energy of the system. On the other hand, as the reinforcing nanoparticles are dispersed in the nanostructured Al matrix, the locking of the grain boundaries can be considered as an important factor in maintaining the grain size in the sub-micron range [29].

According to the X-ray diffraction results (Figure 4), the average grain size in both the milled powders and consolidated composites decreased with the increased volume fraction of SiC nanoparticles. Reinforcing particles accumulate on the grain boundaries, reduce their movement, and at the same time suppress the movement of dislocations. Therefore, they can prevent the growth of nanometer grains to a great extent [30]. With increased amount of reinforcing nanoparticles, the grain boundaries locking is encouraged. As a result, the size of Al grains reinforced with a high volume fraction of SiC particles becomes smaller.

Due to the small size of the initial grains in the extruded sample, a high driving force for growth exists at high temperatures to reduce the surface energy. In fact, while dynamic recrystallization leads to a decrease in the size of grains accompanied with increased amount of grain boundaries, the growth phenomenon at high temperatures acts as an opposite factor. According to Figure 4b, the composite samples experienced less growth as

compared with the matrix. However, due to the low volume fraction of the reinforcements and thereby decreased effectiveness of these particles, the growth of grains at high temperatures was relatively small, i.e. from 50 to 60.8 nm for NS-Al/3SiC<sub>n</sub>, before and after hot extrusion, respectively. However, in the NS Al sample, due to the absence of nanoparticles, grain growth occurred more effectively resulting in increased grain size from 74.9 to 99.8 nm.

The microstructure of the extruded composites with different amounts of SiC<sub>m</sub> particles, as shown in Figure 9, reveal that the micron-sized SiC particles are also distributed uniformly within the matrix alloy. In addition, there is no indication of voids in the microstructures, confirming the material full-densification upon extrusion.

### 3.3.2. Hardness

Figure 10 presents the variation in the hardness of four series of composites with their SiC content. Figure 11 shows the percentage of the enhanced hardness of the matrix alloy when its microstructure converted from coarse-grained to nano-structured. In addition, the percentages of the altered (increased or decreased) hardness of extruded composites as compared to the hardness of unreinforced samples prepared by un-milled Al powders (CG-Al) and milled Al powders (NS-Al)

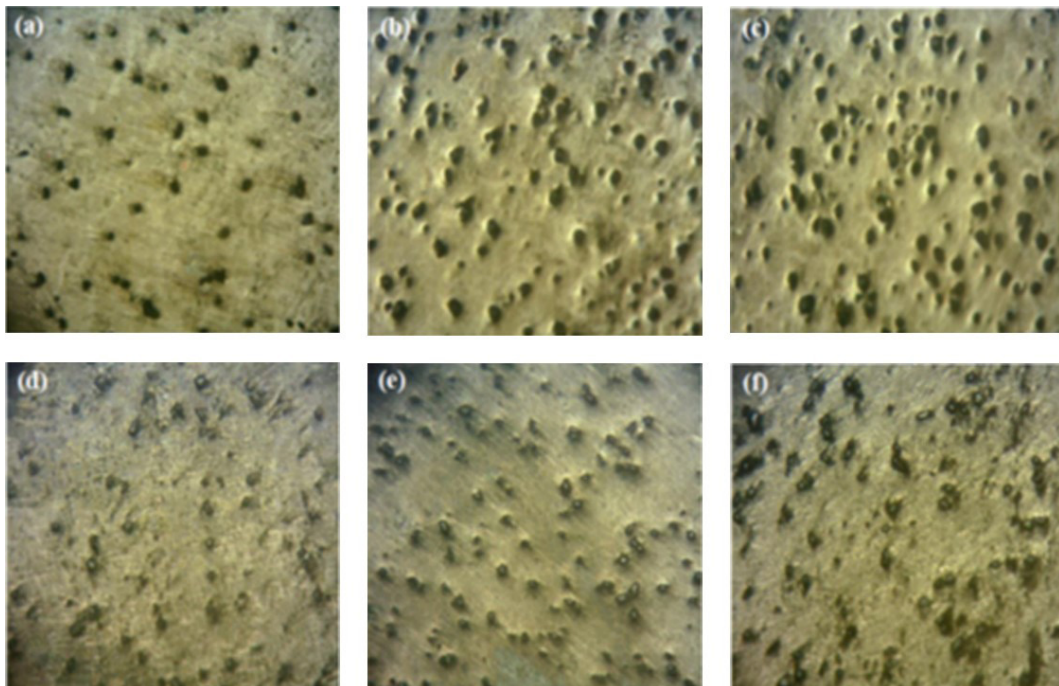


Fig. 9- Compressibility values for four different series of Al-SiC powder mixtures.



are shown in this Figure.

According to Figure 11, nanostructured Al (NS-Al) hardness was more than 120% larger than that of the coarse-grained Al (CG-Al). The increment of the hardness of nanostructured Al is principally due to the grain refinement following the Hall-Petch equation [31]:

$$H = H_0 + K D^{-1/2} \quad (4)$$

Where  $D$  is grain size,  $H_0$  is the hardness of annealed coarse-grained sample, and  $K$  is a constant. As mentioned before, the mechanical milling process brings about grain refining to the nanometer scale. This equation explains the larger hardness of NS-Al sample as compared to that of the CG-Al sample. In fact, the huge amount of grain boundaries in nanostructured materials act as barriers to the dislocations movement leading to increased pile up of dislocations. The present investigation results are consistent with those of the other reports [8, 24, 32]. Abdollahi et al. [24] indicated that the hardness of nanostructured Al-2wt% Cu was roughly twice the same alloy with a coarse grain structure. According to some reports, the ultrafine grain structure retained after hot consolidation of the nanostructured aluminum alloy powders [15, 32]. Zhang and Chen [33] reported that both the decreased size and increased nanoparticle volume fraction enhanced the dislocation density strengthening consequence induced by the Orowan strengthening mechanism.

According to Figure 10, in the CG-Al/ $\text{SiC}_m$  series, up to 3vol.%  $\text{SiC}_m$  particles addition did not increase the hardness of the coarse-grained Al matrix significantly. Al-Rubaie et al. [34] showed that by adding 10 vol.% of  $43\mu\text{m}$  SiC particles to

the aluminum matrix, the hardness increased from 29.6 to 37.2 Hv. In another research study, for Al /10 wt.%  $\text{SiC}_m$  composite only 29% increase in hardness reported as compared to the matrix alloy [3]. Therefore, the negligible increase in the hardness of CG-Al/ $\text{SiC}_m$  by 3vol.%  $\text{SiC}_m$  addition over that of the CG-Al, as shown in Figure 11 is acceptable and consistent with other reports. This Figure also confirms that the hardness of CG-Al/3 $\text{SiC}_m$  is about 50% lower than that of the unreinforced nanostructured alloy.

However, according to Figure 10, the hardness of nanostructured Al improved by  $\text{SiC}_m$  particles addition with a slight slope. In fact, in NS-Al/ $\text{SiC}_m$  series, particle boundary pinning by even such a relatively low number of  $\text{SiC}_m$  particles enhanced hardness. The pronounced higher hardness of NS-Al/ $\text{SiC}_m$  composites as compared to their GC-Al/ $\text{SiC}_m$  counterparts (Figure 10) is attributed to the considerably higher hardness of the matrix of the former series. Figure 11 clearly shows that while the contribution of 3vol.% micron-sized SiC addition to the hardness enhancement of NS-Al/3 $\text{SiC}_m$  is less than 10%, the nano-structured matrix of this composite contributed for about 140% increased hardness.

Figure 10 shows that the hardness of coarse-grained matrix alloy composites also improved with increased content of  $\text{SiC}_n$  addition. As mentioned before, in these composites, the  $\text{SiC}_n$  nano-particles were not embedded within Al powders but accumulated on their boundaries. However, the relatively large number of  $\text{SiC}_n$  particles compensated for the larger grain size of the matrix alloy and enhanced hardness. Here, the increased hardness is attributable to the Orowan strengthening mechanism activated by the  $\text{SiC}_n$

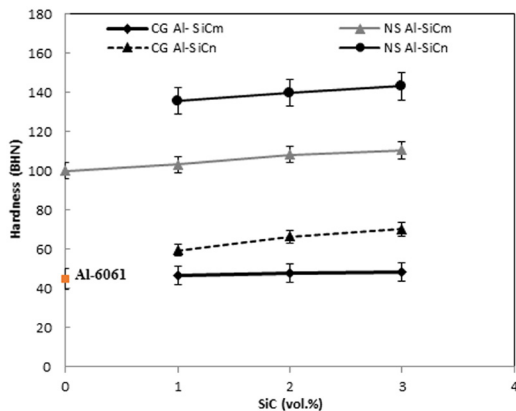


Fig. 10- Hardness of extruded samples.

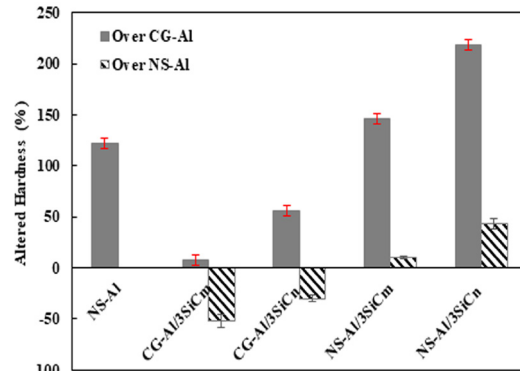


Fig. 11- The altered hardness of extruded samples as compared to CG-Al and NS-Al.

particulates. According to this mechanism, the addition of reinforcing particles to the matrix leads to increased barriers to the movement of dislocations, according to the following equation [33]:

$$\tau = \frac{Gb}{\lambda} \quad (5)$$

where  $\lambda$  is the distance between the reinforcing particles,  $b$  is the Burgers vector,  $G$  is the Shear modulus, and  $\tau$  is the required stress for passing the dislocations through secondary particles. The decreased reinforcing particle size accompanied by the decreased value of  $\lambda$  results in an increased value of  $\tau$ , causing the increased strength of the material. In other words, dislocations pinned behind the  $\text{SiC}_n$  particles impose the work-hardening effect resulting in increased strength.

Similarly, Kang and Chan [8] showed that the strength of Al/2vol.% $\text{Al}_2\text{O}_3$  nanocomposite is higher than that of Al/10 vol.% SiC microcomposite. Al-Rubaie et al. indicated the increased hardness of Al-SiC composites with decreased size of the reinforcing particles [34]. Mula et al. measured 92% hardness enhancement after 2wt.%  $\text{Al}_2\text{O}_3$  nanoparticles addition to aluminum [32]. According to Figure 11, the incorporation of 3vol.% of nano-sized SiC particles into the coarse grained Al resulted in 60% increase in the hardness of the material. However, the hardness of this composite is still about 25% lower than that of the unreinforced nanostructured material.

In NS-Al/ $\text{SiC}_n$  series, the hardness value increased significantly by the addition of only 1% of  $\text{SiC}_n$  particles (Figure 10). The nano-sized SiC particles embedded into Al powder particles probably located at the grain boundaries and enhanced grain boundary pinning. However, the increased amount of  $\text{SiC}_n$  particles addition up to 3% increased hardness with a considerably smaller slope due to the increased tendency of particles agglomeration. According to Figure 4, the grain size of the Al powders co-milled with  $\text{SiC}_n$  particles are smaller than that of the Al powders milled without  $\text{SiC}_n$  particles addition. Therefore, In NS-Al/ $\text{SiC}_n$  series the presence of nano-sized SiC particles in the powder mixture during milling intensified the grain refinement of Al matrix and contributed in enhanced hardness. According to Figure 11, the NS-Al/3 $\text{SiC}_n$  composite is respectively about 45% and 220% harder than the nano-structured and coarse-grained matrix alloy. In fact, the nano-sized grains

of these composites [15, 32] and embedding these particles within the powder particles [9, 22] both contributed in significant hardness achievement in these composites.

#### 4. Conclusions

In this study, four series of composites prepared via P/M route by utilizing either coarse-grained (CG) or nanostructured (NS) 6061 aluminum powder particles as the matrix. 1-3 vol.% of silicon carbide particles in two different size ranges of micron sized ( $\text{SiC}_m$ ) or nano-sized ( $\text{SiC}_n$ ) served as the reinforcing material. The effects of matrix grain size and/or the size of reinforcing particles on enhancing the compressibility of powder mixtures and hardness of the composites investigated. The main conclusions from the present study are as follows:

TEM studies confirmed that 12h planetary ball milling of a mixture of Al and  $\text{SiC}_n$  particles results in homogeneously distribution of  $\text{SiC}_n$  particles inside the Al matrix. SEM images showed that the increased  $\text{SiC}_n$  content resulted in the decreased average size of the milled Al powders. XRD studies revealed that the presence and increment content of hard  $\text{SiC}_n$  particles during milling resulted in decreased size of generated nano-crystallites. This trend retained in the consolidated samples. However, hot extrusion of powder compacts resulted in grain growth of the matrix alloy to some extent.

The decreased compressibility in all of the four series of Al/SiC powder mixtures with increased SiC content attributed to the enhanced portion of the hard and non-deformable SiC particles in the powder mixtures resulting in a reduced degree of plastic deformation. Increment the  $\text{SiC}_n$  content from 0% to 3% resulted in a significant increase in the microhardness of 20h planetary ball milled powders accompanied with decrease compressibility of powders. However, the densification of NS-Al/ $\text{SiC}_n$  nanocomposite powders was better than that of their CG-Al/ $\text{SiC}_n$  counterparts due to their embedded  $\text{SiC}_n$  particles. The CG-Al/ $\text{SiC}_m$  compacts, due to their un-milled Al content, exhibited the highest compressibility among their counterparts in other series

Metallographic studies and density measurements confirmed significant densification with no indication of voids in the samples' microstructures after hot extrusion. Both the micron-sized and nano-sized SiC particles distributed uniformly

within the matrix alloy. For nanocomposites, both the mechanical milling and hot extrusion processes contributed to the uniform distribution of SiC nanoparticles in the matrix alloy.

While up to 3vol.% SiC<sub>m</sub> particles addition did not impose any significant increment in the hardness of the coarse-grained Al matrix composites, this property improved with increased content of SiC<sub>n</sub> addition because of the Orowan strengthening mechanism. Similarly, the hardness of nanostructured Al improved by SiC<sub>m</sub> particles addition with a slight slope. While a significant hardness enhancement achieved by only 1% SiC<sub>n</sub> addition to NS-Al, the increased amount of these particles addition up to 3% did not increase the hardness considerably.

The hardness of unreinforced nanostructured Al was more than 120% larger than that of the coarse-grained Al. Incorporation of 3 vol.% of nano-sized SiC particles in a nanostructured instead of coarse-grained Al matrix resulted in a composite with 110% increased hardness. On the other hand, employing 3 vol.% of nano-sized instead of micron-sized SiC particles in a nanostructured Al matrix resulted in only about 30% increase in the hardness value. These results imply the dominant role of refined microstructure over the nano-sized SiC addition in enhancing the hardness of such composites

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