Effects of SiC wt% Content on Microstructure and Mechanical Properties of Al/SiC Nano Composite produced by Mechanical Alloying, Sintering and Milling

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Abstract: Nano Silicon carbide reinforced aluminum (Al/nanoSiC) metal matrix composites are attractive because of their superior properties such as high strength and stiffness, Application of aluminum in technological and structural application is growing steadily. The major limitation for metal matrix nano composites, however, is their propensity to brittle fracture. The new technologies and new materials are two basic aims for companies. In this research, the effect of addition Al/SiC nano particles on microstructure and mechanical properties of pure aluminum has been investigated. Pure aluminum powder and various fractions of SiC particles with an average diameter of 50 nm were milled by a high-energy planetary ball mill to produce nanocrystalline Al–SiC nanocomposite powders. Pressing and sintering applied to consolidate powders to tablet shape. Then the samples were rolled to cylindrical shape. The nano SiC Percentage were 0%, 2.5%, 5%, 7.5%, 10%, 12.5% and 15%. Mechanical tests such as tensile, hardness, fracture toughness and young’s modules measurement carried out to study the mechanical behavior of each alloy. Scanning electron microscopy was used to study the morphology and microstructure of nanocomposite powders and bulk samples. The role of wt% fraction of SiC nanoparticles was investigated. The results shows that the addition of SiC nano particles has significant influence on the microstructure and mechanical properties of composites and usually the optimum properties depends on wt% SiC.

Keywords: Al/nanoSiC, Mechanical Alloying Powder Metallurgy, Mechanical Properties, Microstructure.

1. Introduction

Metal matrix composites (MMCs) are engineering materials which contain a hard ceramic particles or fibers in metallic matrix to obtain specific properties which make them superior to conventional metals and alloys. Aluminum matrix composites are important categories in these fields. It consist of a non-metallic reinforcement (SiC, B4C, Si3N4, AlN, TiC, TiB2, TiO2) incorporated into aluminum matrix which provides advantageous properties over base metal (Al) alloys. These include improved abrasion resistance, creep resistance, dimensional stability, exceptionally good stiffness-to-weight and strength-to-weight ratios and better high temperature performance, and have extended application in aerospace, automotive and high duty structures, biotechnology, electronic and sporting goods industries. The type, size and uniform distribution of the reinforcing particles in metal matrix, have critical effects on physical and mechanical properties of the composite. [1-7], Al/SiC nano composites are important group of aluminum MMCs which
have higher mechanical and physical properties [8-18]. There are many researches and published papers about Al/nano SiC composites. These researches can be categorized into 6 major fields. The studies of the first focused on manufacturing methods. The results consist of different techniques for fabrication of Al/SiC composites. The major methods of fabrications are squeeze casting, metal spray, metal infiltration, laser deposition technology and mechanical alloying, powder metallurgy, and so on [19–27]. Among them, mechanical alloying have more advantages [25], although there are a lot of problems about uniform distribution of the reinforcement particles in the metal matrix. The second field, concern the role of SiC particles on formability of aluminum matrix. The results showed that nano SiC particles play like a barrier against aluminum formability [28]. The third field of researches concern corrosion behavior of Al/SiC composites [19–32]. They demonstrated that the weight loss of the composites in corrosive media depends strongly on both volume fraction and particle size of nano SiC [26–32]. The studies of 4th group focused on the role of SiC size and distribution on mechanical properties and machinability of Al/SiC nano composites [33, 34]. The 5th group researches concern mechanical, optical, thermal, and electrical properties of Al/SiC composite [35–38]. The searching results shows variation of physical and mechanical properties by variation in volume fraction and particle size of SiC Particles. The latest group investigated dimensional stability and coefficient thermal expansion (CTE) by variation in size and distribution of SiC nano particles. The CTE values of Al/nano SiC composites are important in electronic industry as electronic chips [39–40]. Particle reinforced metal matrix composites have already found commercial use on account of the fact that conventional processing techniques, such as powder metallurgy, vacuum hot pressing, co-spray deposition process, squeeze casting, and stir casting methods can be readily adopted for the processing of such materials [41-42].The introduction should briefly place the study in a broad context and highlight why it is important. It should define the purpose of the work and its significance. The current state of the research field should be reviewed carefully and key publications cited. Please highlight controversial and diverging hypotheses when necessary. Finally, briefly mention the main aim of the work and highlight the principal conclusions. As far as possible, please keep the introduction comprehensible to scientists outside your particular field of research. References should be numbered in order of appearance and indicated by a numeral or numerals in square brackets, e.g., [1] or [2,3], or [4–6]. See the end of the document for further details on references.

2. Materials and Methods

Mechanical alloying (MA) is high energy ball milling process by which constituent powders repeatedly deformed, fractured and welded by grinding media to form a homogeneous alloyed microstructure or uniformly dispersed particulates in a matrix [42]. The main objectives of the milling process are to reduce the particle sizes (breaking down the material), mixing, blending and particle shaping. The major process in MA for producing quality powders of alloys and compounds with well-controlled microstructure and morphology is the repeated welding, fracture, and re-welding of the reactant mixed powders. It is critical to establish a balance between fracturing and cold welding in order to mechanically alloy successfully [43].

Planetary ball milling is carried out for fabrication of engineering materials via a mechanical alloying process. In planetary ball milling, the milling media contains considerably high energy, as the milling stock and balls come off the inner wall of the vial (milling bowl) and the effective centrifugal force can reach up to twenty times of gravitational acceleration. The centrifugal forces caused by the rotation of the supporting disc and autonomous turning of the vial act on the milling charge (balls and powders). Since the turning directions of the supporting disc and the vial are opposite, the centrifugal forces alternately are synchronized and opposite. Therefore, the milling media and the charged powders alternatively roll on the inner wall of the vial, and are lifted and thrown off across the bowl at high speed. One advantage of this type of mill is the ease of handling the vials inside a glove box. The aluminum powder used in this research has a purity of 99%. The particle size of 90% of powder is about 54 nm. In this study stearic acid (C18H36O2 or CH3(CH2)16COOH) and methanol (CH3OH) with purity of 99.99% are used as process control agent to
avoid cold welding. First Suitable amount of pure powder aluminum with 5% wt% of SiC with 99% purity and particle size of 90% of powder about 76 nanometer was ball milled mixing planetary ball mill. The Transmission Electron Microscopy (TEM) of Al and SiC powders are illustrate in figure 1.

![Figure 1. SEM micrograph from Nano-Powders](image)

For mixing the powder, a mixer device with effective sun wheel diameter of 300mm, speed ratio of 1:2.5, interval operation with direction reversal and 1 min pause time after each 15 min running time. The jars were ran under argon gas and then were agitated with 3 different stainless steel milling balls size of 10, 15 and 20 mm at rotation speeds of 200 rpm. The ball numbers of 100 were used for milling times 20 hr. Stearic acid [CH₃(CH₂)₁₆COOH] of 2 wt% and 5 ml methanol [CH₃OH] were added as a process control agent separately and giving a ball to powder weight (B/P) ratio of 10:1 to reach a fine powder particle size regarding to powder morphology and to minimize cold welding of the aluminum particles and to prevent powders from sticking to the balls and the jar wall. Mixing was carefully controlled under blowing argon inert gas with 5 liter per minute and afterward was transferred to the glove box to avoid impurities. Impurities in the sample influence the physical, interface reaction and mechanical properties of milled aluminum. For pressing of prepared powders, the mold illustrate in figure 2-a, which made from heat treated tool steel type SPK, and pressing machine in figure 2-b with loading force 120 tons was used. The samples are shown in figure 3. The sintering operation was carried out in 0.05 bar vacuum furnace at 495°C.

![Figure 2. The mold (a) and (b) tools used in powder pressing](image)

Then the rolling of sintered samples was carried out by a laboratory two mill with 6 Tons load capacity and 25/130 mm diameter/length mills and 20-30 RPM speed. The wire cut machine used for
preparation of tensile test specimens according to ASTM A370. Then the mechanical test was carried out on specimens.

3. Results

3.1. Morphology of milled powder

The Scanning Electron Microscopy (SEM) images of mixed Al/SiC wt 5%, Al/SiC wt 7.5% and Al/SiC wt 10% after 20 hours milling illustrate in figure 3a, 3b, 3c respectively. Figure 4 illustrate the XRD spectra of the as-received aluminum and Al-nano SiC composite powders mechanically milled for 20 hour.

Figure 3. SEM image of: a) Al/SiC wt 5% with average grain size 35.9 nm, b) Al/SiC wt 7.5% with average grain size 39.8 nm, c) Al/SiC wt 10% with average grain size 46.2 nm, after 20 hours milling.

Figure 4. XRD spectra of the as-received aluminum and Al-SiC nanocomposite powders mechanically milled for 20 h.

Figure 5 show the SEM images of rolled Al/SiC 5 and Al/SiC 7.5 wt%. It shows the powder size is effectively reduced after milling and rolling, to average 35.9 nm and 38.7 nm with good uniformity.
The particle size was reduced due to high impact forces and load that forced particle towards higher density, lower porosity and closer to a spherical shape. During the milling process the temperature is going up so after a certain temperature will control the cold welding and it seems that warm welding occurred. At this stage, the impact energy is enough to break down the particles. Generally, the particle size and shape were considered as the most important factors which effect aluminum matrix and mechanical properties of samples.

![SEM image of rolled Al/nano Sic](image)

**Figure 5.** SEM image of rolled Al/nano Sic a) 5% nano Sic  b) 7.5% nano SiC

### 3.2. Tensile Test

Table 1 show the results of tensile test on specimens and figure 6 show engineering stress-strain curves. It show, increasing nano SiC percentage, increase engineering tensile strength, but decrease engineering strain. SiC nano particles have roles as barriers to prevent movement of dislocations, so the required stress for dislocations movement will increase, but regarding these barriers, the engineering strain will decrease.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Yield Strength (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>El%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Aluminum</td>
<td>54.3</td>
<td>85.4</td>
<td>27.8</td>
</tr>
<tr>
<td>Al-2.5% SiC</td>
<td>118.1</td>
<td>132.2</td>
<td>11.95</td>
</tr>
<tr>
<td>Al-5% SiC</td>
<td>125.5</td>
<td>151.9</td>
<td>10.5</td>
</tr>
<tr>
<td>Al-7.5% SiC</td>
<td>133.1</td>
<td>159.4</td>
<td>9.55</td>
</tr>
<tr>
<td>Al-10% SiC</td>
<td>139.6</td>
<td>168.7</td>
<td>8.6</td>
</tr>
<tr>
<td>Al-12/5% SiC</td>
<td>149.1</td>
<td>177.8</td>
<td>7.95</td>
</tr>
<tr>
<td>Al-15% SiC</td>
<td>157.5</td>
<td>187.5</td>
<td>7.3</td>
</tr>
</tbody>
</table>

Figure 7 illustrate the variation of true yield and tensile strength and strain and elongation with various nano Al/SiC wt%. As nano particles increase, the ultimate tensile strength (UTS) and yield strength (YS) increase and elongation percentage decrease. The same reason about dislocation displacement is reliable for this behavior.
Figure 6. Engineering Stress - Strain curves for various Al/nano SiC wt%.

Figure 7. Yield Stress, Ultimate Tensile Strength and Elongation percentage for various versus wt% nano SiC.

Figure 8 show Young’s modulus variations versus wt% nano SiC. From 0% to 2.5%, there is no visible change in results but from 2.5% to 15 wt% nano SiC, there are notable increase in young modules so it increase from 77 to 106 GPa.

Reinforcement particles which are smaller than the matrix particles situate themselves between the matrix particles and fill the pores. This caused an increase in the density of the composites when compared with the Al matrix. This behavior is as a result of high mechanical properties of SiC which include relatively high yield strength, high Young’s modulus, etc except its ductility that is very poor. It is then expected that these features will contribute positively on the mechanical properties of Al/SiC composites when SiC particles are uniformly dispersed as a reinforcement material. However, there is an actual SiC particles percentage addition, through which the maximum values
of these mechanical properties decrease. To find this value, it is necessary to do more tests with higher nano particles addition. Beyond this value, SiC clusters form in the composites and these clusters deteriorate the mechanical properties of the composites by lowering its values.

3.3. Hardness Test

Table 2 and figure 9 show hardness variation versus wt% nano SiC. Silicon carbide particles are hard phase and distribution of these particles in aluminum matrix, increase hardness of alloy. Higher wt% nano SiC, increase hardness of matrix, so the hardness increase by 32% when wt% nano SiC increase from 2.5% to 15%.

Table 2. Brinnel hardness of samples

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Brinell Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>23</td>
</tr>
<tr>
<td>Al-2.5% SiC</td>
<td>79</td>
</tr>
<tr>
<td>Al-5% SiC</td>
<td>86</td>
</tr>
<tr>
<td>Al-7.5% SiC</td>
<td>92</td>
</tr>
<tr>
<td>Al-10% SiC</td>
<td>96.8</td>
</tr>
<tr>
<td>Al-12.5% SiC</td>
<td>101.3</td>
</tr>
<tr>
<td>Al-15% SiC</td>
<td>106</td>
</tr>
</tbody>
</table>

Figure 9. Young’s modulus variations versus wt% nano SiC.

Figure 9. Hardness variations versus wt% nano SiC.
3.4. Fracture Toughness Test

Six blanks of 12.7 x 63.5 x 63.5 mm were cut from the raw samples where the blanks were further machined to final dimensions of the Compact Specimens as specified in ASTM Standard E399-83™. The specimens were 12.7 mm thick and notched in the L-T orientation according. Table 3 and figure 10 shows fracture toughness test results for various Al wt% nano SiC. The Fracture Toughness increase from 48 in 2.5wt% nano Si to 55 MPa.m^{0.5} at 10%.

Table 3. Fracture Toughness test Results

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Fracture Toughness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Al-2.5% SiC</td>
</tr>
<tr>
<td></td>
<td>Al-5% SiC</td>
</tr>
<tr>
<td></td>
<td>Al-7.5% SiC</td>
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<tr>
<td></td>
<td>Al-10% SiC</td>
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<tr>
<td></td>
<td>Al-12.5% SiC</td>
</tr>
<tr>
<td></td>
<td>Al-15% SiC</td>
</tr>
<tr>
<td>Aluminum</td>
<td>35.6</td>
</tr>
<tr>
<td>Al</td>
<td>48</td>
</tr>
<tr>
<td>Al</td>
<td>52</td>
</tr>
<tr>
<td>Al</td>
<td>56</td>
</tr>
<tr>
<td>Al</td>
<td>55</td>
</tr>
<tr>
<td>Al</td>
<td>50</td>
</tr>
<tr>
<td>Al</td>
<td>49</td>
</tr>
</tbody>
</table>

Figure 10. Fracture toughness variation versus wt% nano SiC.

As can be see, that fracture toughness increased significantly by addition of wt% nano SiC reinforcement particles up to 7.5%, but in higher amount decrease. It can be describe by increasing the stress concentration sites by increasing particles amount. At 2.5-7.5% wt% nano SiC, There is 2 different behaviors. First one increasing the mechanical properties and second the stress concentration sites which decrease toughness behavior. But the mechanical properties have higher influence on fracture toughness than sites, and the total fracture toughness increase. When wt% nano SiC increased more than 7.5%, the stress concentration sites have higher influence and reduce final fracture toughness.

4. Conclusion

The reinforcement of Al alloys with nano SiC particulates has led to the development of a group of composite materials with very interesting properties. This research considered aluminum metal matrix reinforced by nano silicon carbide particles by mechanical alloying process. In particular, Al/nano SiC composites exhibit better stiffness and strength than the corresponding unreinforced alloys. The main limitation of Al/SiC nano composites to be used as structural
elements arises from their reduced ductility and fracture toughness specially at high amount of
nano SiC induced by the presence of brittle, ceramic reinforcements, so the study of the fracture
behavior has received increasing attention from the research community in recent years.

The prepared samples provide higher mechanical properties than the commercial composite
materials. Mechanical tests show increase in reinforcement particles can increase the yield and
tensile strength, hardness, and also modify the fracture toughness at certain percentage of wt% nano
SiC. Fracture toughness increased significantly by addition up to 7.5%. Higher contents, reduced the
fracture toughness of the composites. This was mainly attributed to premature yielding caused by
the high ductility/low strength of the Al matrix.

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