

1 Article

2 Effects of SiC wt% Content on Microstructure and 3 Mechanical Properties of Al/SiC Nano Composite 4 produced by Mechanical Alloying, Sintering and 5 Milling

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12 **Abstract:** Nano Silicon carbide reinforced aluminum (Al/nanoSiC) metal matrix composites are
13 attractive because of their superior properties such as high strength and stiffness, Application of
14 aluminum in technological and structural application is growing steadily. The major limitation for
15 metal matrix nano composites, however, is their propensity to brittle fracture. The new
16 technologies and new materials are two basic aims for companies. In this research, the effect of
17 addition Al/SiC nano particles on microstructure and mechanical properties of pure aluminum has
18 been investigated. Pure aluminum powder and various fractions of SiC particles with an average
19 diameter of 50 nm were milled by a high-energy planetary ball mill to produce nanocrystalline Al-
20 SiC nanocomposite powders. Pressing and sintering applied to consolidate powders to tablet shape.
21 Then the samples were rolled to cylindrical shape. The nano SiC Percentage were 0%, 2.5%, 5%,
22 7.5%, 10% , 12.5% and 15%. Mechanical tests such as tensile, hardness, fracture toughness and
23 young's modules measurement carried out to study the mechanical behavior of each alloy.
24 Scanning electron microscopy was used to study the morphology and microstructure of
25 nanocomposite powders and bulk samples. The role of wt% fraction of SiC nanoparticles was
26 investigated. The results shows that the addition of SiC nano particles has significant influence on
27 the microstructure and mechanical properties of composites and usually the optimum properties
28 depends on wt% SiC.

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

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32 1. Introduction

33 Metal matrix composites (MMCs) are engineering materials which contain a hard ceramic
34 particles or fibers in metallic matrix to obtain specific properties which make them superior to
35 conventional metals and alloys. Aluminum matrix composites are important categories in these
36 fields. It consist of a non-metallic reinforcement (SiC, B₄C, Si₃N₄, AlN, TiC, TiB₂, TiO₂)
37 incorporated into aluminum matrix which provides advantageous properties over base metal (Al)
38 alloys. These include improved abrasion resistance, creep resistance, dimensional stability,
39 exceptionally good stiffness-to-weight and strength-to-weight ratios and better high temperature
40 performance, and have extended application in aerospace, automotive and high duty structures,
41 biotechnology, electronic and sporting goods industries. The type, size and uniform distribution of
42 the reinforcing particles in metal matrix, have critical effects on physical and mechanical properties
43 of the composite. [1- 7], Al/SiC nano composites are important group of aluminum MMCs which

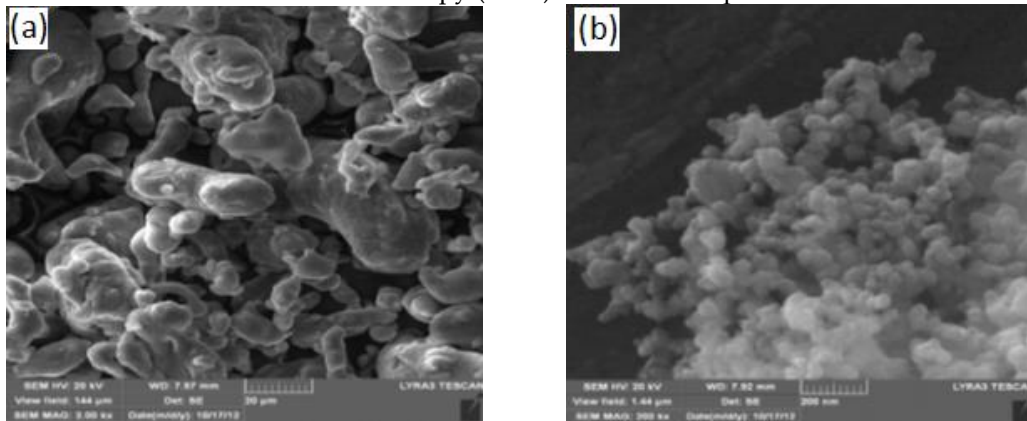
44 have higher mechanical and physical properties [8-18]. There are many researches and published
45 papers about Al/nano SiC composites. These researches can be categorized into 6 major fields. The
46 studies of the first focused on manufacturing methods. The results consist of different techniques for
47 fabrication of Al/SiC composites. The major methods of fabrications are squeeze casting, metal spray,
48 metal infiltration, laser deposition technology and mechanical alloying, powder metallurgy, and so
49 [19–27]. Among them, mechanical alloying have more advantages [25], although there are a lot of
50 problems about uniform distribution of the reinforcement particles in the metal matrix. The second
51 field, concern the role of SiC particles on formability of aluminum matrix. The results showed that
52 nano SiC particles play like a barrier against aluminum formability [28]. The third field of researches
53 concern corrosion behavior of Al/SiC composites [19–32]. They demonstrated that the weight loss of
54 the composites in corrosive media depends strongly on both volume fraction and particle size of
55 nano SiC [26–32]. The studies of 4th group focused on the role of SiC size and distribution on
56 mechanical properties and machinability of Al/SiC nano composites [33, 34]. The 5th group
57 researches concern mechanical, optical, thermal, and electrical properties of Al/SiC composite [35–
58 38]. The searching results shows variation of physical and mechanical properties by variation in
59 volume fraction and particle size of SiC Particles. The latest group investigated dimensional stability
60 and coefficient thermal expansion (CTE) by variation in size and distribution of SiC nano particles.
61 The CTE values of Al/nano SiC composites are important in electronic industry as electronic chips
62 [39–40]. Particle reinforced metal matrix composites have already found commercial use on account
63 of the fact that conventional processing techniques, such as powder metallurgy, vacuum hot
64 pressing, co-spray deposition process, squeeze casting, and stir casting methods can be readily
65 adopted for the processing of such materials [41–42]. The introduction should briefly place the study
66 in a broad context and highlight why it is important. It should define the purpose of the work and its
67 significance. The current state of the research field should be reviewed carefully and key
68 publications cited. Please highlight controversial and diverging hypotheses when necessary. Finally,
69 briefly mention the main aim of the work and highlight the principal conclusions. As far as possible,
70 please keep the introduction comprehensible to scientists outside your particular field of research.
71 References should be numbered in order of appearance and indicated by a numeral or numerals in
72 square brackets, e.g., [1] or [2,3], or [4–6]. See the end of the document for further details on
73 references.

74 2. Materials and Methods

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

83 Planetary ball milling is carried out for fabrication of engineering materials via a mechanical
84 alloying process. In planetary ball milling, the milling media contains considerably high energy, as
85 the milling stock and balls come off the inner wall of the vial (milling bowl) and the effective
86 centrifugal force can reach up to twenty times of gravitational acceleration. The centrifugal forces
87 caused by the rotation of the supporting disc and autonomous turning of the vial act on the milling
88 charge (balls and powders). Since the turning directions of the supporting disc and the vial are
89 opposite, the centrifugal forces alternately are synchronized and opposite. Therefore, the milling
90 media and the charged powders alternatively roll on the inner wall of the vial, and are lifted and
91 thrown off across the bowl at high speed. One advantage of this type of mill is the ease of handling
92 the vials inside a glove box. The aluminum powder used in this research has a purity of 99%. The
93 particle size of 90% of powder is about 54 nm. In this study stearic acid (C₁₈H₃₆O₂ or CH₃
94 (CH₂)₁₆COOH) and methanol (CH₃OH) with purity of 99.99% are used as process control agent to

95 avoid cold welding. First Suitable amount of pure powder aluminum with 5% wt% of SiC with 99%
 96 purity and particle size of 90% of powder about 76 nanometer was ball milled mixing planetary ball
 97 mill. The Transmission Electron Microscopy (TEM) of Al and SiC powders are illustrate in figure 1.

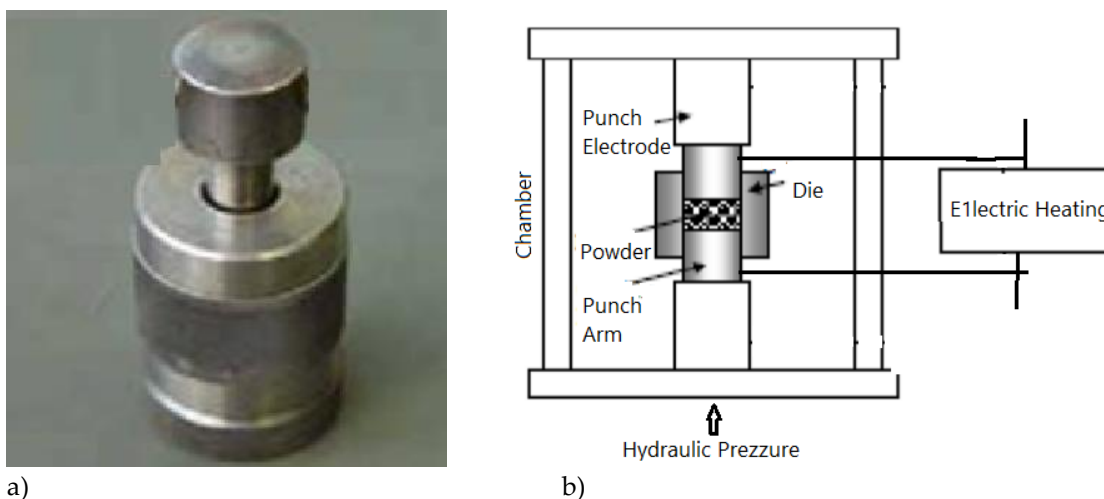


100 **Figure 1.** SEM micrograph from Nano- Powders (a) Pure Al, (b) Sic

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102 For mixing the powder, a mixer device with effective sun wheel diameter of 300mm, speed ratio
 103 of 1: 2.5, interval operation with direction reversal and 1 min pause time after each 15 min running
 104 time. The jars were ran under argon gas and then were agitated with 3 different stainless steel
 105 milling balls size of 10, 15 and 20 mm at rotation speeds of 200 rpm. The ball numbers of 100 were
 106 used for milling times 20 hr. Stearic acid [CH₃ (CH₂)₁₆COOH] of 2 wt% and 5 ml methanol
 107 [CH₃OH] were added as a process control agent separately and giving a ball to powder weight (B/P)
 108 ratio of 10:1 to reach a fine powder particle size regarding to powder morphology and to minimize
 109 cold welding of the aluminum particles and to prevent powders from sticking to the balls and the jar
 110 wall. Mixing was carefully controlled under blowing argon inert gas with 5 liter per minute and
 111 afterward was transferred to the glove box to avoid impurities. Impurities in the sample influence
 112 the physical, interface reaction and mechanical properties of milled aluminum. For pressing of
 113 prepared powders, the mold illustrate in figure 2-a, which made from heat treated tool steel type
 114 SPK, and pressing machine in figure 2-b with loading force 120 tons was used. The samples are
 115 shown in figure 3. The sintering operation was carried out in 0.05 bar vacuum furnace at 495°C.
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Figure2. The mold (a) and (b) tools used in powder pressing

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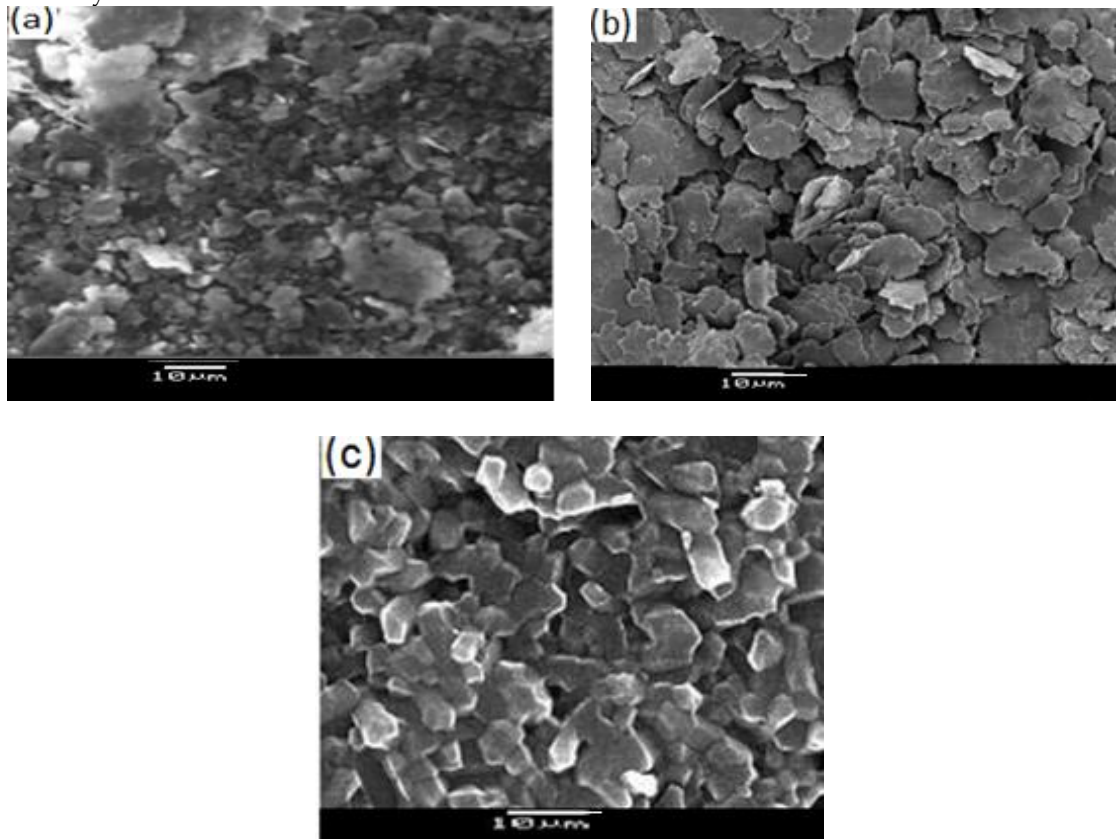
Then the rolling of sintered samples was carried out by a laboratory two mill with 6 Tons load capacity and 25/130 mm diameter/length mills and 20-30 RPM speed. The wire cut machine used for

124 preparation of tensile test specimens according to ASTM A370. Then the mechanical test was carried
125 out on specimens.

126 3. Results

127 3.1. Morphology of milled powder

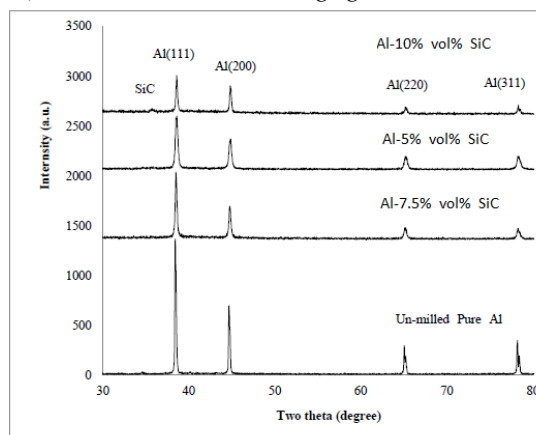
128 The Scanning Electron Microscopy ((SEM) images of mixed Al/SiC wt 5% , Al/SiC wt7.5%
129 and Al/SiC wt 10% after 20 hours milling is illustrate in figure 3a, 3b, 3c respectively. Figure 4
130 illustrate the XRD spectra of the as-received aluminum and Al-nano SiC composite powders
131 mechanically milled for 20 hour.



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Figure 3. SEM image of : a) Al/SiC wt 5% with average grain size 35,9 nm , b) Al/SiC wt7.5% with average grain size 39.8 nm c) Al/SiC wt 10% with average grain size 46,2 nm , after 20 hours milling.



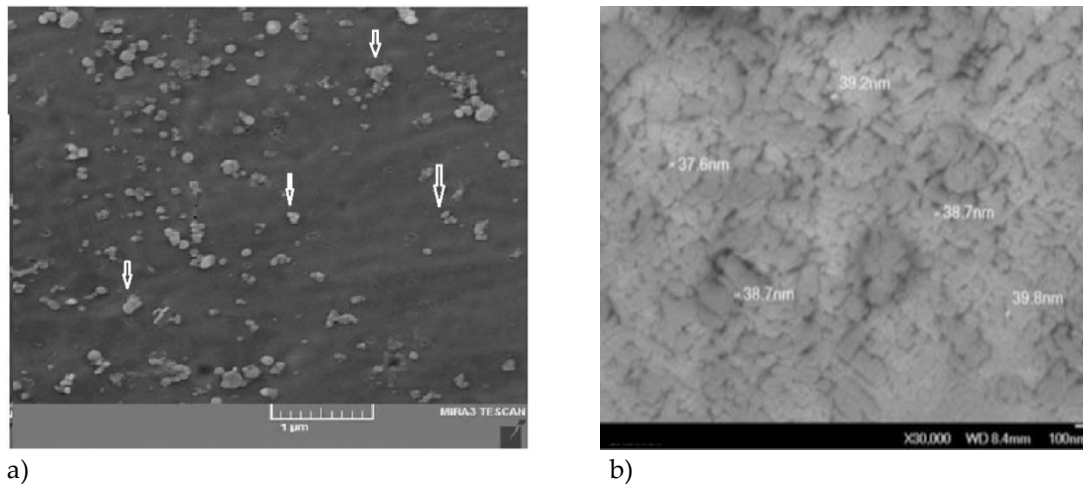
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Figure4. XRD spectra of the as-received aluminum and Al-SiC nanocomposite powders mechanically milled for 20 h.

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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.

142 The particle size was reduced due to high impact forces and temperature inside the jar and rolling
 143 load that forced particle towards higher density, lower porosity and closer to a spherical shape.
 144 During the milling process the temperature is going up so after a certain temperature will control the
 145 cold welding and it seems that warm welding occurred. At this stage, the impact energy is enough to
 146 break down the particles. Generally, the particle size and shape were considered as the most
 147 important factors which effect aluminum matrix and mechanical properties of samples.
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 151 **Figure 5.** SEM image of rolled Al/nano SiC a) 5% nano SiC b) 7.5% nano SiC
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153 3.2. Tensile Test

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

159 **Table 1-** Results for true tensile tests for various Al/ nano SiC wt%.

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

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

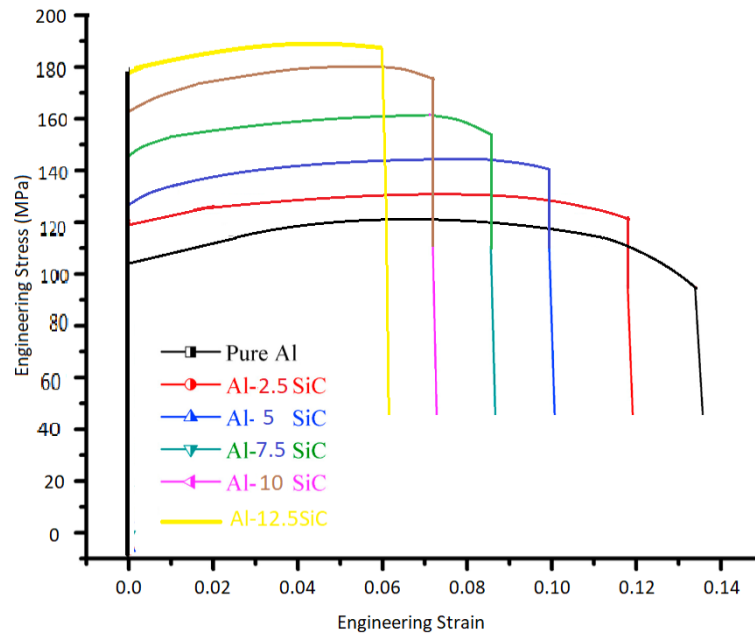


Figure 6. Engineering Stress - Strain curves for various Al/ nano SiC wt%.

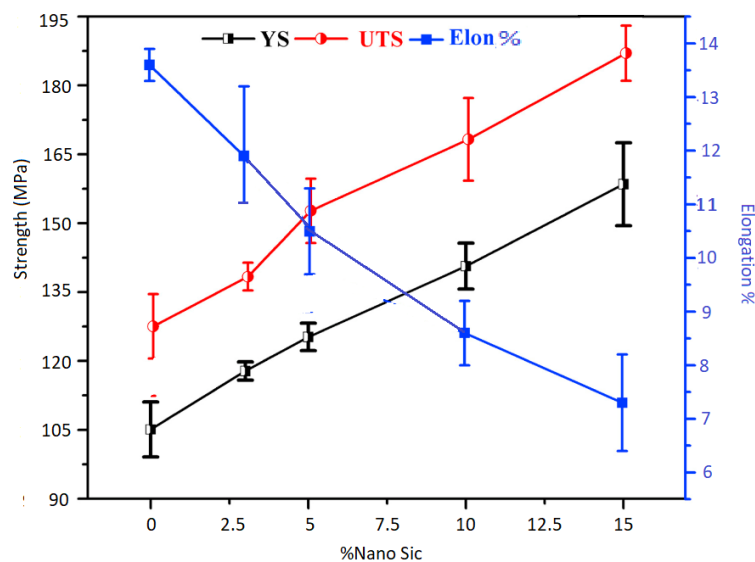


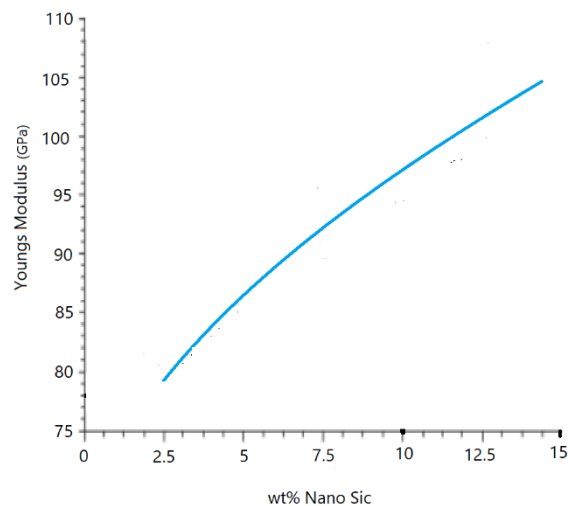
Figure 7. Yield Stress, Ultimate Tensile Strength and Elongation percentage for various versus wt% nano SiC.

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

175 Reinforcement particles which are smaller than the matrix particles situate themselves between
176 the matrix particles and fill the pores. This caused an increase in the density of the composites when
177 compared with the Al matrix. This behavior is as a result of high mechanical properties of SiC which
178 include relatively high yield strength, high Young's modulus, etc except its ductility that is very
179 poor. It is then expected that these features will contribute positively on the mechanical properties of
180 Al/SiC composites when SiC particles are uniformly dispersed as a reinforcement material. However,
181 there is an actual SiC particles percentage addition, through which the maximum values



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183 **Figure 9.** Young's modulus variations versus wt% nano SiC.
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185 of these mechanical properties decrease. To find this value, it is necessary to do more tests with
186 higher nano particles addition. Beyond this value, SiC clusters form in the composites and these
187 clusters deteriorate the mechanical properties of the composites by lowering its values.

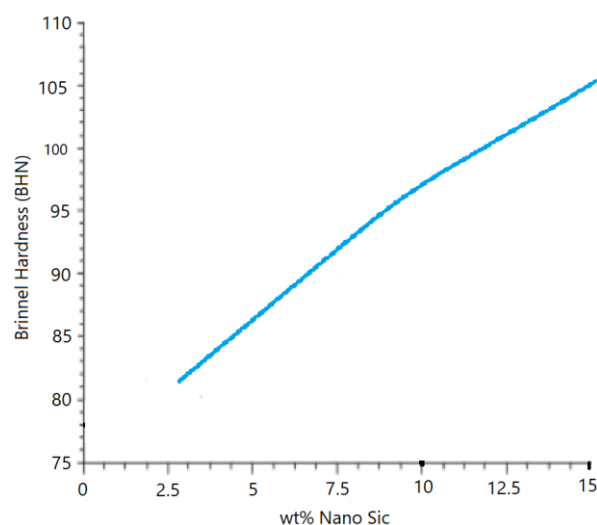
188 3.3. Hardness Test

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

193 **Table 2.** Brinell hardness of samples

Alloy	Aluminum	Al-2.5% SiC	Al-5% SiC	Al-7.5% SiC	Al-10% SiC	Al-12/5% SiC	Al-15% SiC
Brinell Hardness	23	79	86	92	96.8	101.3	106

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Figure9. Hardness variations versus wt% nano SiC.

197 3.4. Fracture Toughness Test

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

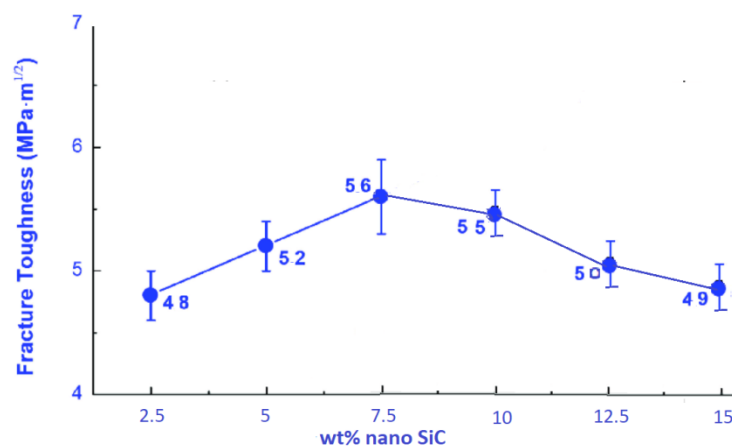
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Table 3. Fracture Toughness test Results

Alloy	Aluminum	Al- 2.5% SiC	Al-5% SiC	Al-7.5% SiC	Al-10% SiC	Al-12/5% SiC	Al-15% SiC
Fracture Toughness MPa.m ^{0.5}	35.6	48	52	56	55	50	49

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

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

217

218 4. Conclusion

219 The reinforcement of Al alloys with nano SiC particulates has led to the development of a
 220 group of composite materials with very interesting properties. This research considered aluminum
 221 metal matrix reinforced by nano silicon carbide particles by mechanical alloying process. In
 222 particular, Al/nano SiC composites exhibit better stiffness and strength than the corresponding
 223 unreinforced alloys. The main limitation of Al/SiC nano composites to be used as structural

224 elements arises from their reduced ductility and fracture toughness specially at high amount of
225 nano SiC induced by the presence of brittle, ceramic reinforcements, so the study of the fracture
226 behavior has received increasing attention from the research community in recent years.
227 The prepared samples provide higher mechanical properties than the commercial composite
228 materials. Mechanical tests show increase in reinforcement particles can increase the yield and
229 tensile strength, hardness, and also modify the fracture toughness at certain percentage of wt% nano
230 SiC. Fracture toughness increased significantly by addition up to 7.5%. Higher contents, reduced the
231 fracture toughness of the composites. This was mainly attributed to premature yielding caused by
232 the high ductility/low strength of the Al matrix.
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