Mechanical and Electrical Properties of Nano Al-Matrix Composites Reinforced with SiC and Prepared by Powder Metallurgy

Mahmoud F. Zawrah 1, Wafaa M. El-Meligy 2, Heba H. A. Saudi 3, Safae Ramadan 4, Mohammed A. Taha 5

1 Refractories; Ceramics and Building Materials Department; National Research Centre; 2622-Dokki; Cairo; Egypt
2 Department of Physics; Faculty of Science; Al-Azhar University (Girls’ Branch); Nasr City, Egypt; wafaa.mohamed@yahoo.com (W.M.E.M.); heba_saudi@hotmail.com (H.A.S.);
3 Solid State Physics Department; National Research Centre; 12622-Dokki; Cairo; Egypt; ramadan@gmail.com (S.R.); mahmoudrc@gmail.com (M.A.T.);
4 Scopus Author ID 6604055746

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Abstract: Nano Al-matrix composites reinforced with SiC were prepared by powder metallurgy process. The percentages of added SiC were varied between 0, 2, 4, 6, and 8 wt. %. The starting powders were milled in high-energy ball mill for 10hrs to convert into nanopowders; then compacted and sintered for 1h in an argon atmosphere at 400, 500, and 570°C. X-ray technique and transmission electron-microscope were utilized to examine the prepared powders, while scanning electron-microscope was utilized to test the sintered composites. The relative density, apparent porosity, electrical conductivity, and mechanical properties (microhardness, elastic moduli, and compressive strength) of sintered composites were studied. The results showed no sign for phase changes after milling, and the SiC reinforcement was uniformly distributed in the matrix. The relative density and electrical conductivity were decreased with increasing SiC content, while the apparent porosity was increased. It is also found that the mechanical properties were improved with increasing SiC content. Also, all properties were improved with increasing sintering temperature. The hardness, compressive strength, bulk modulus of Al-8wt.% SiC composite sintered at 570°C were 885.4 MPa, 276.2 MPa, and 135.9 GPa, respectively.

Keywords: Al-SiC composites; sintering; physical properties; mechanical-properties; electrical conductivity.

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1. Introduction

No doubt, the metal-matrix nanocomposites reinforced with ceramic particles have excellent properties like high thermal conductivity, high young's modulus, high ultimate strength, low density, high-temperature resistance. So, they can be applied for many engineering applications in the aerospace energy, defense, and automobile industry. The most interesting metal matrix composites are the Al base nanocomposites. They can be reinforced by different types of reinforcements in the form of particles, whiskers, or short-fibers [1-4]. The addition of ceramic particles to Al can improve the mechanical properties, especially the hardness and strength [5]. Many types of ceramics have been utilized to improve the properties of Al base nanocomposites; including Al2O3 [6,7], ZrO2 [8], ZrC [9], TiB2 [10], B4C [2], and SiC [5,11]. Among these ceramics, SiC ceramic can be used as an excellent reinforcement for
Al base nanocomposites due to its high microhardness (about 25 GPa), good wear resistance, and appropriate electrical and thermal conductivity. The addition of SiC leads to a significant improvement of the mechanical properties of the composite, i.e., microhardness, strength, elastic modulus, and wear resistance without a severe decrease in the electrical and thermal characteristics of the nanocomposites [1,12,13]. The ideal microstructure of Al-SiC composite should include homogeneously dispersed SiC nanoparticles in fine-grains Al matrix. This leads to improving the physico-mechanical properties of the nanocomposite; their values increase with increasing the percentages of added SiC [12,14,15]. Friction stir [7], stir casting [16], and mechanical alloying (MA) [17-19] are the most common processes for the production of Al-SiC nanocomposites. Mechanical alloying is one of the most interesting tools used to produce Al-SiC nanocomposites with significant mechanical properties. This is due to it can produce composites with uniform distribution of SiC particles between Al matrix grains. Throughout the milling process, the SiC particles can well disperse in the matrix, a repeated cold-welding has occurred, and finally, the fracturing is happened [1,20]. This process depends on many parameters, including the particle size of starting powders, types of matrix and reinforcement powders, milling time, the weight of ball-to-powder ratio, milling speed, and atmosphere of milling [21,22]. In this study, the effect of SiC contents on phase composition, sinterability, physical, mechanical, and electrical properties of Al-matrix nanocomposites is the main goal. In detail, throughout this work, the influence of the milling process on the powder particle size, morphology, crystal size, and dislocation density are also investigated. For the sintered composites, the microstructure, density, porosity, microhardness, compressive, yield, elastic moduli, fracture strain, and electrical conductivity are studied in relationship to SiC contents and sintering temperatures.

2. Materials and Methods

2.1. Materials.

Al powder having 99.9% purity and average particle size of 45 μm, as well as SiC having 99.8 purity and average particle size of 60 nm, were used as starting powders to produce Al-SiC nanocomposites.

2.2. Experimental methods.

Various weight percentages of SiC, i.e., 0.0, 2, 4, 6, and 8 wt%, were utilized to produce designed Al-SiC composites (AS0, AS2, AS4, AS6, and AS8). The mixed powders have been milled for 10 hrs using a planetary ball-mill type MTI/SFM-1(QM-3SP2) in the presence of 1wt.-% stearic acid as a processing-controlling agent to avoid agglomeration of the powders through milling. The powder mixtures were milled using Al2O3-balls with a diameter of 20mm, with 500 rpm rotating speed and ball to powder ratio of 20:1. To recognize the phase composition, crystal size, lattice strain, dislocation density, and particle size of milled powders, X-ray instrument type “Philips PW 1373 and transmission electron-microscopy (TEM) kind “JEOL JEM-1230” were employed. The crystal size (D) of the milled powders was calculated using Scherer eqn. (1) [23].

$$D = \frac{0.9\lambda}{B\cos\theta}$$  

(1)

since \(\lambda = 1.54059 \text{ A}°\), B is the full width at half maximum and \(\theta\) is angle (in radians).

The dislocation density (\(\delta\)) was estimated by eqn. (2) [24].
\[ \delta = \frac{1}{D^2} \quad (2) \]

The lattice strain (\( e \)) was also estimated by the following eqn. (3) [25].

\[ \varepsilon = \frac{B}{4 \tan \theta} \quad (3) \]

The prepared powders were cold-compacted at 20 MPa into small pellets with the desired size having 16mm in diameter and 5mm in height. The pressed specimens were sintered for 1 h at 400, 500, and 570°C in an inert atmosphere with heat-rating 7°C/min. The physical properties in terms of relative density and apparent-porosity of sintered composites were assessed using Archimedes’ process permitting to ASTM-B962-13. The theoretical density of Al-SiC composites was considered according to the rule of mixtures, considering the full dense values of Al and Al₂O₃ are 2.7 and 3.95g/cm³, respectively. Then, the relative density of sintered pellets was estimated using the bulk- and theoretical-density. Vickers microhardness of sintered composites was determined following ASTM-B933-09 using load 1.961N for 10 seconds. Vickers hardness was estimated by the mathematical expression no. 4, where P is the applied load and D is the diagonal of obtained shape after indentation [26,27].

\[ H_v = 1.854 \times \frac{P}{D^2}\ldots \quad (4) \]

The compressive strength of sintered composites was determined according to ASTM E9. Also, the yield strength, compressive strength, strengthening efficiency (R), and fracture strain were calculated from the stress-strain curve. The longitudinal (\( V_L \)) and shear (\( V_S \)) ultrasonic wave-velocities spread in the sintered composites were estimated by pulse-echo technique MATEC-Model-MBS8000-DSP (ultrasonic digital-signal process) having 5MHz resonance. According to the following equations, the quantities of \( \lambda \) and \( \mu \) (Lame’s constants) were calculated from VL and VS ultrasonic velocities [28].

\[ \lambda = \rho(V_L^2 - 2V_S^2) \quad (5) \]
\[ \mu = \rho V_S^2 \quad (6) \]

since \( \rho \) is the bulk density of matter.

The quantities of elastic-moduli, i.e., longitudinal-modulus (\( L \)), shear-modulus (\( G \)), Young’s-modulus (\( E \)), bulk modulus (\( B \)) and Poisson’s ratio (\( \nu \)) were estimated according to the following equations [29,30].

\[ L = \lambda + 2\mu \quad (7) \]
\[ G = \mu \quad (8) \]
\[ E = \mu \frac{3\lambda + 2\mu}{\lambda + \mu} \quad (9) \]
\[ B = \lambda + \frac{2}{3}\mu \quad (10) \]
\[ \nu = \frac{\lambda}{2(\lambda + \mu)} \quad (11) \]

The electrical conductivity of sintered composites was determined at ambient temperature (25°C) and 40V using Hioki 3532 system.

3. Results and Discussion

3.1. Phase composition of prepared powders.

Figure 1 displays XRD diffraction patterns of Al-SiC mixed powders with various SiC weight percent, i.e., 0, 2, 4, 6, and 8wt.%, after milling for 10h. The patterns indicate the
existence of two phases, Al and SiC phases, according to the XRD-card numbers 89-4037 and 89-2225, respectively. The characteristic peaks of cubic crystal-structure Al phase appear at 2θ is equal to 38.47, 44.72, 65.09, and 78.01°, while characteristic peaks of rhombohedral crystal-structure SiC phase are exhibited at 2θ being equal to 35.6 and 60.13°. Its peak intensity increases with increasing its added amount. For the Al-SiC composite, which contains only 2 wt.% SiC, only one phase (Al phase) is detected because the quantity of SiC is small enough to be not detected by the XRD device. These results agree with those reported in Ref. [31]. On the other side, with increasing the amount of added SiC particles, its peaks start to appear in the patterns. All Al peaks are broad with low intensities indicating the higher lattice-strain and dislocation density while lower crystallite sizes [1,8,23]. Figure 2 exhibits the crystallite size, lattice strain, and dislocation density for Al-SiC composite powders having different SiC contents. It is observed from the figure that the crystallite size of Al reduces with increasing the weight percentages of SiC, while the lattice strain and dislocation density are increased. These results are related to the presence of hard SiC particles, which increase the refinement of Al grains and their diffusion activation energy [1,32,33]. The values of crystallite sizes of Al-SiC powders that contain 0, 2, 4, 6 and 8 wt.% SiC are 33.25, 31.15, 26.22, 21.48 and 18.60 nm, respectively, while the dislocation density are 9.05 x 10^{-4}, 1.03 x 10^{-3}, 1.45 x 10^{-3}, 2.17 x 10^{-3} and 2.98 x 10^{-3} %, respectively.

3.2. Particle features of prepared powders.

Figure 3 displays TEM images of Al-SiC nanopowders that contain different percentages of SiC, i.e., 0.0, 2, 4, 6, and 8 wt.%, after milling for 10 h. While the particle size of milled powers as calculated from TEM image analysis is shown in Figure 4. It is exhibited from the figure that the particles are agglomerated in the case of lower amounts of added SiC and well dispersed after adding higher amounts of SiC. All particles have undefined and
deformed shapes. Their sizes decrease with the increase of SiC added amounts. This is due to the higher hardness of SiC particles in comparison with that of Al particles. Throughout the mechanical-alloying process, the Al particles suffer flating and welding while SiC particles tend to fragment. The pure Al sample (un-reinforced with SiC) undergoes welding with a higher agglomeration of particles due to their strong plastic deformation [8,34]. This phenomenon appears in TEM images of pure Al or those having lower amounts of SiC. After increasing the percentages of added SiC particles with their uniform distribution, Al particles’ flating process is weakened, while the fracture of particles is increased. Subsequently, a reduction in particle size is occurred [1]. This means that the particle size decreases with increasing SiC contents until rich to 39.5 nm for the sample that contains 8 wt. % SiC.

3.3. Density and porosity of sintered composites.

The relative density and apparent porosity of Al-SiC composites in relationship with SiC contents and sintering temperatures (i.e., 400, 500, and 570°C) are shown in Figure 5.

Figure 3. TEM images of Al-SiC milled powders having various SiC contents; (a) 0.00; (b) 2.0; (c) 4.0; (d) 6.0; (e) 10wt.% SiC.

It is worth mentioning that the calculated theoretical density of Al-SiC composites that contain 0.0, 2, 4, 6, and 8 wt.-% SiC are 2.700, 2.709, 2.717, 2.726, and 2.735g/cm³,
The relative density (Figure 5a) of sintered composites drops with raising the SiC content, while it rises with the increase of firing temperature. On the other hand, the apparent-porosity (Figure 5b) rises with rising SiC content and reduces with rising firing temperature (i.e., opposite trend of relative density). The decreasing relative density with a higher added quantity of SiC is due to the reduction of densification rate since the SiC grains are considered a barrier throughout the diffusion stage of sintering and weaken the bonds between Al matrix particles interfacial interaction [9]. Moreover, due to the rising of SiC content, the boundaries between Al/SiC phases increase, improving pore-nucleation and inhibiting the wettability at the interfaces among the grains [35].

Similarly, some results have been published by researchers in the refs [8,21,23]. They found that with increasing the amounts of added ZrO₂ and SiC particles incorporated into Al-matrix composites, the relative density was decreased while the apparent porosity was decreased. It also indicated from the presented data that the relative density increases with higher firing temperature, while the apparent porosity reduces with increasing sintering temperature. The relative density of specimens sintered at 400 & 570°C decreased from 89.5 to 83.3% and 96.2 to 92.6 %, respectively, increasing SiC contents from 0 to 8 wt.%. The improving of relative density at higher firing temperature is related to the lower melting point of Al (it is about 660°C) and related to the increasing of diffusion rate with increasing the activation energy required for sintering mechanism to precede neck growth which causes grain-grain interaction [36-38].

![Figure 4. Particle size of milled powders as a function of SiC percentages.](https://doi.org/10.33263/BRIAC122.20682083)

![Figure 5. (a) Relative-density; (b) apparent-porosity of Al-SiC composites fired at 400, 500, and 570°C.](https://biointerfaceresearch.com/)
3.4. SEM features of sintered specimens.

Figure 6 shows SEM micrographs of Al-SiC specimens that contain 0.00, 2 & 8wt.-% SiC, sintered at 570°C for 1h. Due to the milling of starting powders for 10h, nanopowders are successfully formed, and SiC particles are well embedded in the Al-matrix and wetting the Al particles. Consequently, the homogenous and dense microstructure is obtained with well-dispersed SiC grains. Due to the small size of starting powders and low sintering temperature (570°C), the grain size of obtained microstructure is very small and seems to be like gel. This means that the grain growth rate is very low even after sintering at 570°C. As indicated from the microstructure, the number and size of pores rise with the rising of added SiC quantities. This good microstructure will be reflected in the mechanical properties.

Figure 6. SEM images of Al, Al-4wt.% SiC and Al-8wt.% SiC composites sintered at 570 °C.

3.5. Mechanical characteristics of sintered composites.

Figure 7 shows the effect of SiC content and sintering temperature on the microhardness of prepared composites. It can be seen that the microhardness of composites increases with
increasing both SiC percent and sintering temperature. After sintering at 400°C, the microhardness values for Al-SiC composites containing 0.0, 2.4, 6.0, and 8 wt. % SiC are 279.2, 338.8, 429.8, 611.2, and 701.5 MPa, respectively. With increasing the sintering temperature to 500 and 570 °C, the microhardness exhibits further increase. The microhardness values for composites reinforced with 8 wt.% SiC are 820.1 and 885.4 MPa, respectively.

The increase of hardness with increasing of SiC content is due to the greater hardness of SiC in comparison with that of Al metal, while the increase of hardness with increasing the sintering temperature is due to the lower porosity of sintered composites at higher temperatures. Figure 8 shows the compressive stress-strain curves of Al-SiC composites-specimens having different percentages of SiC and sintered at 400, 500, and 570°C for 1h. It is indicated that the presence of SiC increases the load stress and decreases the deformation of the composite. The values of strain decrease with increasing SiC content; the lowest values were obtained for the composites that contain 8 wt.% SiC sintered at different temperatures. Also, the stress of composites increases with increasing sintering temperature, while the deformation of composites decreases with increasing sintering temperature. This means that the presence of hard SiC in the matrix of the composites improves their load-bearing. On the other hand, the effect of SiC contents and sintering temperatures on compressive strength, yield strength, and fracture strain are shown in Figure 9. It can be seen that compressive strength and yield strength are improved with the rising of SiC amounts and firing temperature, while the fracture strain is reduced with higher SiC content and increases with the rising of firing temperature. The values of compressive strength for pure Al sintered at 400, 500, and 570 °C, are 136.3, 181.3, 187.4 MPa, while for the composite that contains 8 wt.% SiC sintered at the same sintering temperatures are 240.2, 261.3, 276.2 MPa, respectively. This means that the existence of 8wt.% SiC improves compressive strength values by about 47.5% compared to their original values of pure Al. Figure 10 shows the strengthening factor (R) of the reinforcement for the sintered composites as a function of SiC contents and sintering temperatures. The R values of metal-matrix composites could be calculated by the following equation [39]:

$$R = \frac{\sigma_c - \sigma_m}{V\sigma_m}$$  \hspace{1cm} (12)

where $\sigma_m$ is the strength of the matrix, $\sigma_c$ is the strength of metal matrix composite, while $V$ is the volume-percent of SiC (reinforcement). The strengthening factor indicates the efficiency of strength enhancement due to reinforcement embedding in the matrix. The figure shows that the values of R increase with increasing the percentages of added SiC and sintering temperature. The values of R for the composites Al-2wt.% SiC and Al-8 wt.% SiC sintered at
570°C are 7.64 and 10.11, respectively. These results are comparable with all aforementioned mechanical properties.

Figure 8. Stress-strain curves of Al-SiC composites that contain 0, 2, 4, 6, and 8 wt. % SiC and sintered at (a) 400°; (b) 500°; (c) 870°C.

The elastic-moduli values (i.e., E, L, B, G & ν) of all sintered composites in relationship with SiC contents and sintering temperatures are shown in Figure 11. As seen in the figure, the values of elastic-moduli are enhanced with the rising of SiC percent and firing temperature (same trend of microhardness and strength). For pure Al samples fired at 400 °C, the bulk-modulus (B) and Poisson's-ratio (ν) are 41.6 GPa and 0.3033; they are increased into 84.8 GPa & 0.3199, respectively, when the SiC percent reaches 8 wt.%. When the sintering temperature increases to 500 and 570 °C, the composite containing 8 wt. % SiC exhibits a bulk modulus of
116.7 and 135.9 GPa, respectively, and Poisson’s ratio of 0.3254 and 0.3304, respectively. The obtained results are similar to those reported in many works of literature [32, 40-42].

**Figure 9.** Effect of SiC quantity on compressive-strength, yield-strength, and fracture-strain of composites fired at different sintering temperatures.

**Figure 10.** Strengthening factor of reinforcement for sintered composites as a function of SiC contents and sintering temperatures.
Figure 11. Effect of SiC content on: (a) Young's modulus; (b) longitudinal modulus; (c) bulk modulus; (d) shear modulus; (e) Poisson's ratio, of Al-SiC composites fired at various temperatures.

Generally, the improvement of mechanical properties (as microhardness, strength, and elastic-moduli) of Al-SiC specimens with the increase of SiC amount is attributed to several reasons. Firstly, the excellent interface and good dispersion between Al matrix and SiC reinforcement formed during mechanical milling are effective factors [43]. Secondary, increasing the percentage of hard SiC particles compared to Al raises the development of dislocations, which is considered a barrier for plastic deformation, thus increasing nanocomposites' mechanical properties [44,45]. Gautam et al. [46] reported that the improvement of the strength for AA5052 matrix reinforced with ZrB2 is attributed to refining grains that obstruct the cracks' transmission and growth. The elongation of Al-SiC composites is decreased owing to the presence of nano-SiC reinforcement. The applied load transfers from the Al-matrix into SiC particles which raise the resistance to plastic-deformation of specimens. Also, there is a thermal expansion mismatch between the Al matrix (higher thermal expansion coefficient) and the SiC grains (lower thermal expansion coefficient). This thermal expansion mismatch causes thermal stress in the specimens and dislocates at the interface [47,48]. Increasing the sintering temperature of the composites also has a positive effect on overall
mechanical properties due to increasing the interaction between the grains and decreasing the porosity. Similar results about the influence of firing temperature on mechanical characteristics of metal-matrix composites have been reported by several researchers using different reinforcements [1,2,23,49,50].

3.6. Electric properties of sintered composites.

Figure 12 shows the electrical conductivity of Al-SiC composites that include 0.0, 2, 4, 6, and 8 wt.% SiC and sintered at different temperatures. The results in Figure 12 exhibit that the conductivity of sintered composites decreases slightly with increasing SiC contents and increases remarkably with firing temperature. The conductivity of the composites fired at 400°C reduces from 3.41×10⁷ to 2.62×10⁷ S/m when the SiC content increases from 0.0 to 8 wt.%, while it increases with increasing sintering temperature to 500 and 570 °C.

![Figure 12. Electrical conductivity of Al-SiC composites sintered at different sintering temperatures.](image)

The conductivity decreases from 4.13×10⁷ to 7.15×10⁶ S/m and from 4.58×10⁷ to 9.9×10⁶ S/m, respectively. These results are related to the fact that the conductivity of ceramic SiC reinforcement is less than the conductivity of the Al matrix. Therefore, when the content of added SiC particles increases in the Al matrix, this leads to decreasing the conductivity of the composites [1,23,51]. Moreover, in Al-metal, the electrons-nucleus interaction is low, so the movement of electrons is easy and results in high conductivity. After adding SiC to the Al matrix, this causing stable bonds between the electrons and nucleus, leading to decreasing the conductivity [52-54]. The increasing electrical conductivity of fired composites with the increase of firing temperature might decrease the porosity and improve the density [23, 55,56].

4. Conclusions

Al-SiC nanopowders having different SiC percentages (i.e., 0.0, 2, 4, 6, 8 wt.%) have been successfully prepared by the mechanical alloying method and fired in an inert atmosphere at various firing temperatures, i.e., 400, 500, and 570°C. The crystallite and particle sizes of milled powders were decreased with increasing SiC contents; their values were 18 nm and 39.5nm, respectively, when the value of SiC reached 8wt.%. SEM analysis showed that the SiC particles were distributed homogeneously in the Al matrix, indicating a good bonding between the SiC and Al matrix. The relative density of fired composites was decreased with increasing SiC content, while it increased with the increase of firing temperature. The rising of SiC content and sintering temperature caused improving the mechanical properties, especially the fracture strain. The microhardness, compressive strength, strengthening factor, and elastic
modulus of the Al-8wt.%SiC composite sintered at 570 °C were 885.4 MPa, 276.2 MPa, 10.11, and 182 GPa, respectively. Furthermore, the electrical conductivity of fired composites was also decreased with increasing SiC percentages and improved with the increase of firing temperature.

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**Conflicts of Interest**

The authors declare no conflict of interest.

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