Microstructure and Mechanical Properties of Al$_2$O$_3$-SiC Nanocomposites with 0.05% MgO and Different SiC Volume Fraction

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Abstract: In this study, Al$_2$O$_3$–SiC nanocomposites have been fabricated by mixing of alumina powder containing 0.05% weight magnesium oxide and silicon carbide nano powders, followed by hot pressing at 1650^{0}C. The mechanical properties of Al$_2$O$_3$-SiC nanocomposites containing different volume fraction of nano scale SiC particles were investigated and compared with those of alumina. The fracture mode and microstructure of specimens were investigated by means of scanning electron microscopy. The nanocomposites were tougher compared to alumina when they were hot pressed at the same temperature. The young's modulus is decreased by increasing the volume percent of SiC. The values hardness and fracture toughness of the nanocomposites is increased by increasing the volume percent of SiC up to 7.5% and then decreased slightly. The ballistic energy dissipation ability is decreased by increasing the volume percent of SiC up to 5% and then increased slightly. The Scanning electron microscopy observations showed that fracture mode is changed from intergranular for alumina to transgranular for nanocomposites. It also proves that growth of grain is decreased by increasing the volume fraction of SiC particles.

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1   INTRODUCTION

Al_2O_3 ceramics are widely used in many industrial fields. However, the abnormal grain growth of Al_2O_3 during sintering can decrease its mechanical properties. The applications of Al_2O_3 can be limited because of its brittle nature [1]. There are several methods to change its mechanical properties. The first method is by adding small amounts of MgO that can effectively inhibit the abnormal growth of Al_2O_3 grains. The second method is by adding second phase particles. It has been reported that SiC nano particles as second phase particles could change the mechanical properties of Al_2O_3 significantly [2]. Ceramic nanocomposites represent a new class of materials with significantly improved mechanical properties even at high temperatures compared to monolithic ceramics.

One notable system is Al_2O_3-SiC nanocomposite with distinguishably improved mechanical properties. Generally, these nanocomposites are fabricated at high densities through a hot-pressing process because of the difficulty in densifying the composites. However, this process can only manufacture ceramic particles with simple geometrical shapes, and would be expensive and unsuitable for mass production. For many potential applications of these materials, pressureless sintering process would be preferable if full density is desirable [3]. To explain the improvement in the mechanical properties of nanocomposites, several strengthening/ toughening mechanisms including the reduction of flaw size in the composites, stronger Al_2O_3-SiC interfaces relative to those of Al_2O_3-Al_2O_3, and residual stresses due to the thermal expansion mismatch between Al_2O_3 and SiC grains have been suggested [2-4].

There were some studies on fabricating the Al_2O_3-SiC nanocomposites by pressureless sintering process [3, 5-6]. Joeng and Nihara [3] fabricated Al_2O_3-5%SiC nanocomposites with high fracture strength by pressureless sintering and hot-isostatic pressing (HIP) technique which can subsequently break through the disadvantage of hot-pressing process. Boras et al [5] fabricated Al_2O_3-5%SiC (mass fraction) nanocomposite by a pressureless sintering route to a maximum relative density of about 95%. Anya and Roberts [6] promote Al_2O_3-%SiC nanocomposites with high relative density (≥99.6%) and with up to 15% SiC (volume fraction) by pressureless sintering. However, strength improvement of Al_2O_3-%SiC nanocomposites through pressure-less sintering method has not been reported.

In this research, 500 ppm of MgO nano powders were added to all components and Al_2O_3–SiC nanocomposites were sintered by hot-press method.

Fracture mode, microstructure, Young's modulus, hardness, fracture toughness, and ballistic energy dissipation ability of nanocomposites with different volume fraction of SiC nano particles were investigated. The produced densities of nanocomposites are very close to theoretical density compared to the composites produced by pressure-less method [7].

2   EXPERIMENTAL PROCEDURES

In this research, consumed raw materials were highly pure γ-Al_2O_3 nanopowders (99.9% purity and <100 nm size), β-SiC nano particles (98% purity and <100 nm size) and MgO nano particles that were produced in MUT institute. After weighing the Al_2O_3 and SiC powders and adding 500ppm MgO nano powders, raw materials were mixed by planetary mill in isopropanol for 3 hours. After milling, isopropanol was vaporized by means of heater-magnet followed by heating in oven at 100 ºC. Dry powders were milled again for another one hour to crush soft agglomerates. Mixed powders were sintered at 1650 ºC and 20 MPa through hot-press method in the graphite furnace in argon for 2 hours. In addition, the planetary milling media and the container are highly pure tungsten carbide (WC). Seventeen balls were used in this process.

Milling process was performed with 150 rpm and $550 cm^3$ net volumes for 3 hours. The densities of sintered specimens were measured using the Archimedes method and ASTM B311 [8]. For mechanical testing, the hot pressed specimens were cut and ground into rectangular specimens (4×3×50mm). Young's modulus was measured based on ASTM C769 [9]. Vickers indentation method was used to determine the hardness and the fracture toughness of specimens. Hardness was measured according to ASTM C1327 [10] and the fracture toughness values were determined for the specimens. The formula used for median/radial cracks is the equation proposed by Moradkhani et al [11].

$$K_{IC} = 0.003693 \, (\frac{E}{H})^{\frac{1}{2}} \, t_{Ave}^{\frac{3}{2}} \, \frac{P}{A^{\frac{3}{2}}} \tag{1}$$

In Eq. (1), K_{IC} is the fracture toughness (MPa.√m), E is the young's modulus (GPa), H is the Vickers hardness (GPa), P is the indicator of load (N), t_{Ave} is the micro cracks thickness average of circumferential indentation (mm), and A is the area of micro cracks in circumferential indentation (mm^2).

The ballistic energy dissipation ability was determined by the equation proposed by Medvedovski [12]:

$$D = \frac{0.36(H.E.v)}{K_{IC}^2} \qquad (2)$$

where D is the ballistic energy dissipation ability (×10⁻¹²1/s), H is the Vickers hardness (MPa), E is the young's modulus (GPa), v is voice speed in the specimen (m/s), and K_{IC} is the fracture toughness (MPa.√m).

The top and bottom surfaces of the hot-pressed samples were ground with a diamond wheel. Before indentation, the top surfaces were polished using diamond paste to a 1μm finish. The loaded surfaces and the fracture across sections were examined using a scanning electron microscope. XRD analysis was used to investigate probable reactions between components of composites after sintering process.

3   RESULTS AND DISCUSSION

3.1. Relative density and the MgO effects

Figure 1 shows changes of relative density versus volume fraction of SiC particles. It can be seen that by increasing the volume fraction of SiC particles, relative density is decreased. In addition, it is obviously evident that all the components could reach very high densities, near theoretical densities. The reason for producing these highly densified composites could be the efficient sintering temperature, hot-press method and existence of small amounts of MgO nano powders [7]. On the other hand, since nanoSiC particles do not have the sufficient motion and do not react with alumina at sintering temperature, they decrease the grain domain of mobility and hinder the agglomeration of alumina. Therefore, adding nanoSiC causes alumina density to be decreased; hence, the relative density to be reduced. Figure 2 demonstrates the relative density as a function of MgO content in Al_2O_3-5% nanoSiC nanocomposite, when it is sintered at 1650°C for 2 hours in argon. Adding a few hundred ppm MgO into an Al_2O_3-5% nanoSiC nanocomposite causes a dramatic increase of relative nanocomposite density. In this figure, the relative density of Al_2O_3-5% nanoSiC for 0, 50 and 300 ppm of MgO are marked [3].

3.2. Young's modulus

Figure 3 shows changes of young's modulus versus volume fraction of SiC particles. It can be seen that the young's modulus of the specimens is decreased by

means of increasing the volume fraction of SiC. Young's modulus of Al_2O_3-15% SiC is less than alumina. This could be the result of increasing porosity due to nanoSiC volume fraction increase of up to 15%. As SiC is increased, the young's modulus nanocomposite is expected to increase. While increasing SiC leads to reduction of density, the formation of porosity causes decrease of voice speed in the specimen, resulting decrease of young's modulus [11].
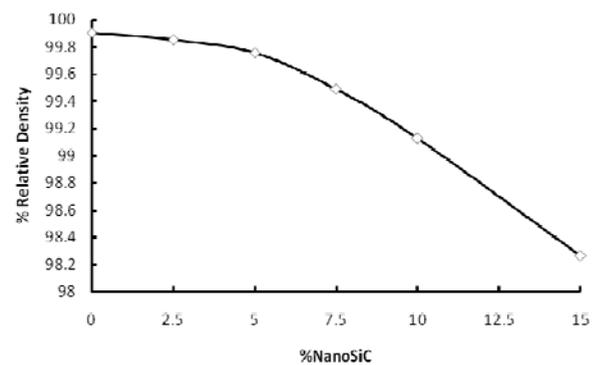
Fig. 1   Relative density of nanocomposites versus volume fraction of SiC.
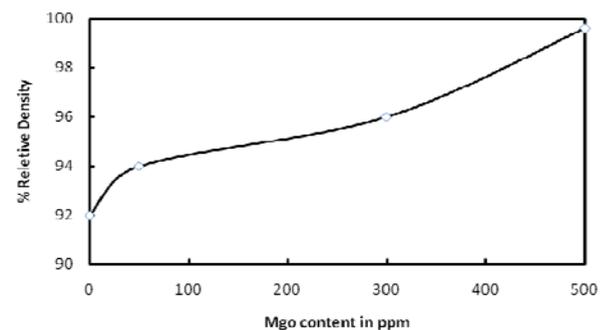
Fig. 2   The relative density versus MgO content in Al_2O_3-5% nanoSiC nanocomposite, when sintered at 1650°C for 2 h in argon.

3.3. Hardness

The hardness of nanocomposites was significantly improved by adding nanoSiC particles. This could be the effects of nanoSiC particles on refining matrix grains. Also, the hardness of SiC is higher than Al_2O_3. Thus, the presence of SiC particles in the matrix itself could improve the hardness of nanocomposites. On the other hand, this could be related to the granularity of composite microstructure. Changes of hardness versus volume fraction of SiC particles are shown in figure 4. It can be seen that the hardness of specimens was increased due to increase in the volume fraction of SiC

particles up to 7.5% and then afterwards it is decreased due to adding more SiC particles. Since the porosity is increased, the density decreases when the volume fraction of second phase is more than 7.5%. As a result the overall hardness of specimens decreases.
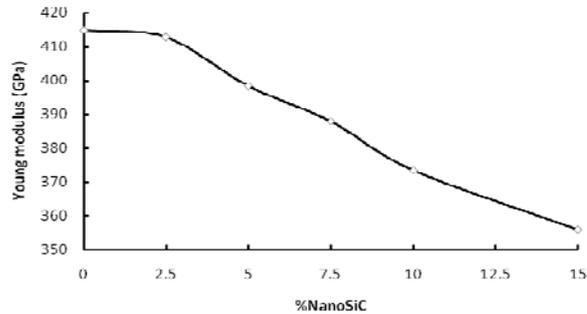
Fig. 3 Young's modulus of nanocomposites versus volume fraction of SiC.

On the other hand, agglomeration of nanoSiC particles is increased and their distribution uniformity in the matrix (alumina) also decreases. Therefore, the hardness of specimens is decreased. Adding SiC particles to the composite, the residual stress between Al_2O_3 and nanoSiC increases. This is caused by unequal thermal expansion. This increase releases the residual stress and develops micro-crack in the nanoSiC. Hence, the hardness of specimens reduces. As it is shown in figure 4, the hardness of the nanocomposite containing 7.5% SiC nanoparticles reduces as more nanoparticle is added to it reaching 15% its volume fraction.
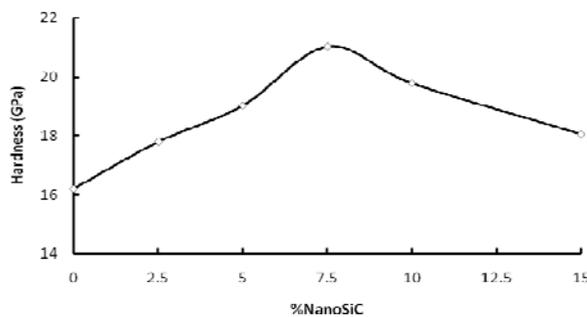
Fig. 4 Vickers hardness of nanocomposites versus volume fraction of SiC.

3.4. Fracture toughness

In Figure 5 changes of fracture toughness versus volume fraction of SiC particles is illustrated. By increasing the volume fraction of SiC particles up to 7.5%, the fracture toughness is increased from 3.3 MPa.√m for Al_2O_3 to 4.4 MPa.√m for Al_2O_3-7.5% SiC.

However, adding more SiC nanoparticles until the volume fraction of 15%, the measured fracture toughness is decreased to 3.1 MPa.√m. This reduction could be caused by uneven distribution of SiC particles and the formation of agglomerates in composites with 15% SiC.

Residual stresses generated by difference in the coefficients of thermal expansion between Al_2O_3 matrix and SiC reinforcement is known as one of the most important factors in increasing the mechanical properties of Al_2O_3-SiC nanocomposites. The residual stresses generate micro cracks in the matrix. These micro cracks can divide the strain energy of a primary crack by branching and deflecting it [6, 13-14].
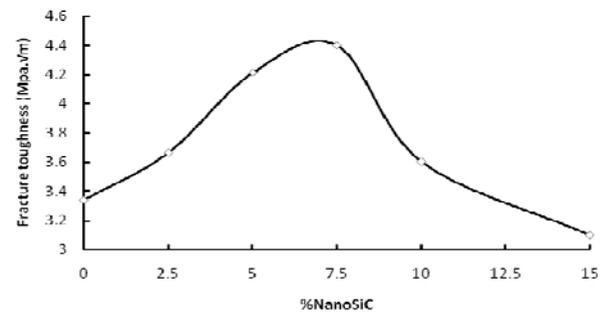
Fig. 5 Fracture toughness of nanocomposites versus volume fraction of SiC.

3.5. Ballistic energy dissipation ability

Figure 6 illustrates changes on ballistic energy dissipation ability versus volume fraction of SiC particles. By increasing the volume fraction of SiC particles up to 5%, D is decreased to 1.62×10^{-12} 1/s. But from that volume fraction onwards, adding more nanoparticles, increments measured D. Ref. [12] has reported that the ballistic energy dissipation ability of the alumina ceramics is between 1.5×10^{-12} 1/s and 2.4×10^{-12} 1/s which is close to our results.
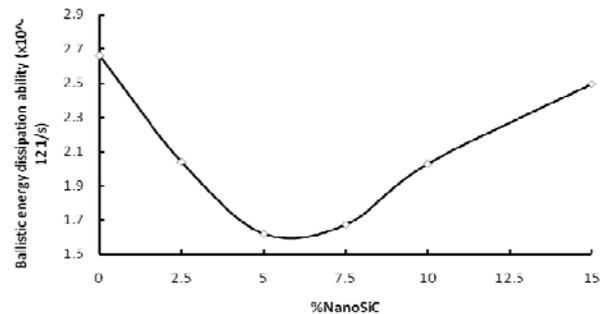
Fig. 6 Ballistic energy dissipation ability of nanocomposites versus volume fraction of SiC.

3.6. Fracture mode of nanocomposites

Micrographs from fracture surfaces of Al_2O_3 and Al_2O_3-SiC nanocomposites containing 5 and 15 percent of SiC particles are shown in Fig. 7. (a-c). The fracture mode in Al_2O_3 was mainly intergranular and it can be seen in Fig. 7. (a). That grains are pulled out from the boundaries while it was transgranular in nanocomposites. This change in the fracture mode is attributed to the SiC particles located in the grain boundaries [7].

a: Al_2O_3
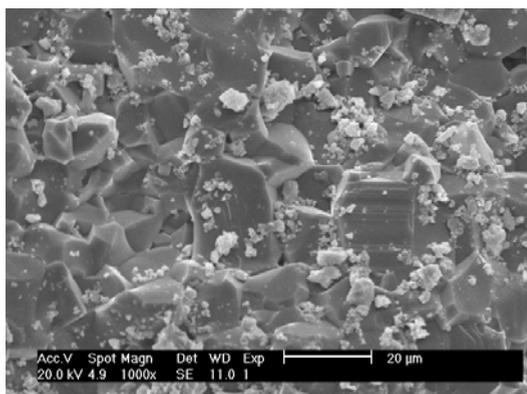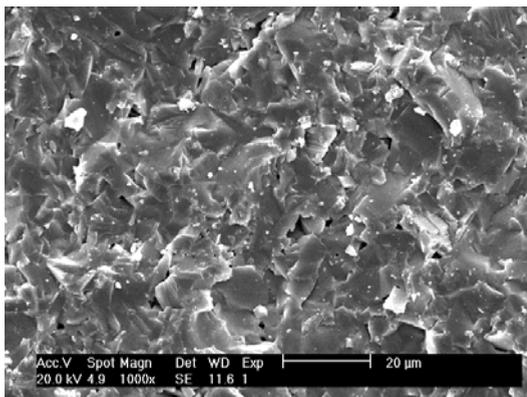
a: Al_2O_3

b: Al_2O_3-2.5%SiC

b: Al_2O_3-5%SiC

c: Al_2O_3-5%SiC
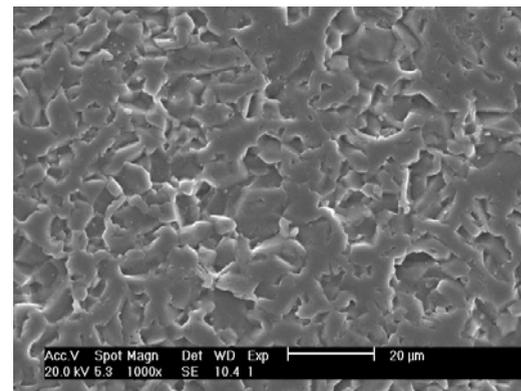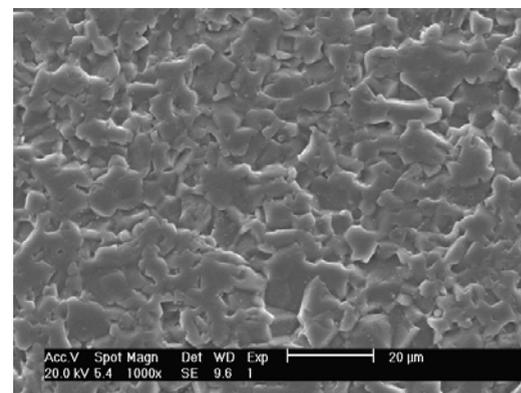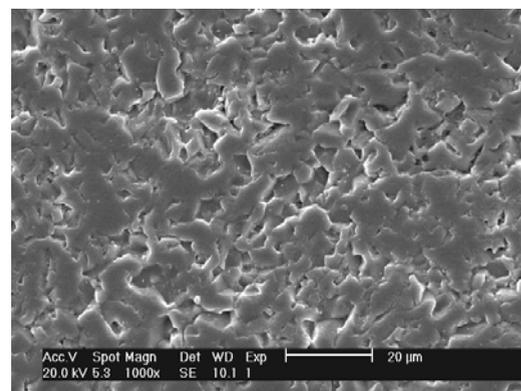
c: Al_2O_3-15%SiC

Fig. 7 SEM micrographs from fractured surfaces for Al_2O_3 and Al_2O_3-SiC nanocomposites.

d: Al_2O_3-7.5%SiC
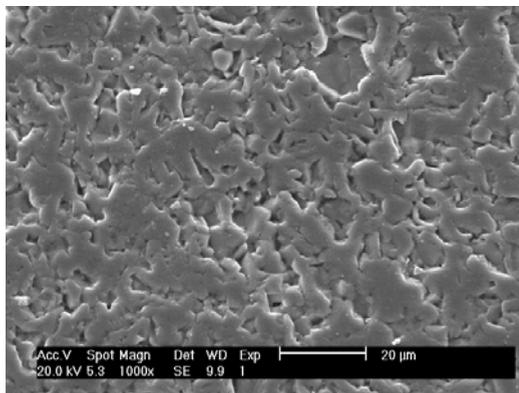
e: Al_2O_3-10%SiC
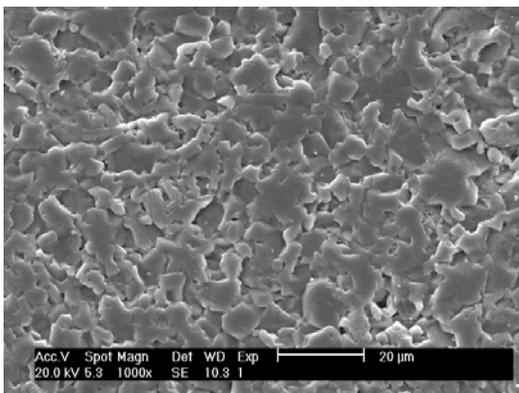
f: Al_2O_3-15%SiC

Fig. 8 SEM micrographs from microstructure for Al_2O_3 and Al_2O_3-SiC nanocomposites

3.7. Microstructure of nanocomposites

SEM micrographs of Al_2O_3 and Al_2O_3-SiC nanocomposites containing 2.5, 5, 7.5, 10 and 15 percent of SiC particles are shown in Fig. 8. (a-f). It can be seen that the grain growth is decreased by means of increasing the volume fraction of SiC particles.

3.8. XRD analysis

Figure 9 shows the XRD patterns for Al_2O_3- 10% SiC nanocomposite after being sintered at 1650ºC for 2 hours. It confirms that this phase neither reacted extensively with the liquid formed by the nanoMgO additives nor oxidized. This is an important observation, since the presence of the nanoSiC phase is responsible for improvements in the mechanical properties in Al_2O_3-SiC nanocomposite. However, considering MgO additives, formation of some secondary phases were predicted. These phases were

not observed in this work, since their amounts were below the detection limit of the equipment.
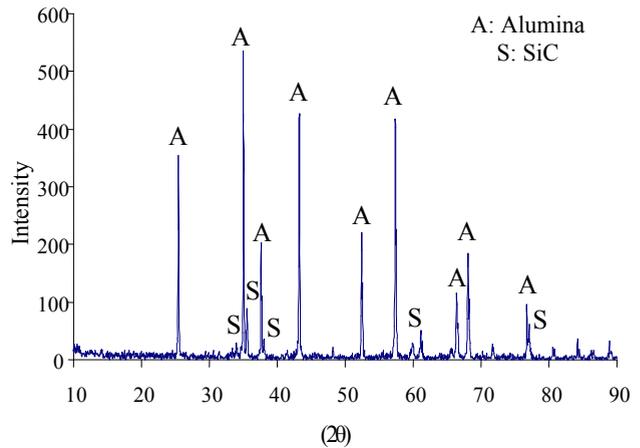
Fig. 9 XRD patterns for Al_2O_3- 10% SiC nanocomposite after sintering

4   CONCLUSION

In this paper, material properties of Al_2O_3-SiC nanocomposites containing 0.05% MgO weight and different volume fraction of nano scale added SiC particles are investigated and the following results are obtained.

- The MgO based compound with 500 ppm Al_2O_3-SiC nanocomposite can promote the nanocomposite density.

- NanoSiC particles can decrease the density of Al_2O_3-SiC nanocomposites. Hot-press sintering method heated to a sufficient temperature causes the nanocomposite density approximate to the theoretical density.

- By adding SiC nano particles to Al_2O_3, the young's modulus of this ceramic was decreased from 415GPa for Al_2O_3 to 356Gpa for Al_2O_3-15% SiC.

- The maximum Vickers hardness of nanocomposites was improved up to 21 GPa for Al_2O_3-7.5% SiC.

- By adding SiC nano particles to Al_2O_3, the fracture toughness of this ceramic was increased from 3.3 MPa.$\sqrt{m}$ for Al_2O_3 to 4.4 MPa.$\sqrt{m}$ for Al_2O_3-7.5% SiC.

- By adding SiC nano particles to Al_2O_3, the ballistic energy dissipation ability of this ceramic was decreased from 2.66 $\times 10^{-12}$1/s for

Al_2O_3 to 1.62×10^{-12} 1/s for Al_2O_3-5% SiC. But by adding more SiC nano particles to the Al_2O_3 matrix, the ballistic energy dissipation ability increased to 2.45×10^{-12} 1/s for Al_2O_3-15% SiC.

- The fracture mode of Al_2O_3 is intergranular but it changes to transgranular in nanocomposites.

- XRD analysis after sintering Al_2O_3-10% SiC nanocomposite couldn't detect any reaction between raw materials except for γ-$Al_2O_3 \rightarrow \alpha$-$Al_2O_3$.

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