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# Fabrication and mechanical properties of nano-/micro-sized Al<sub>2</sub>O<sub>3</sub>/SiC composites

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#### ABSTRACT

The processing and mechanical behavior of  $Al_2O_3$  composites with 1–20 wt.% nano-/micro-sized SiC particles was investigated. The composites were densified by hot-pressing. The mechanical properties of nano-/micro-sized SiC/Al $_2O_3$  composites including hardness, fracture toughness and flexural strength were investigated. It was found that the fracture strength and fracture toughness of the nano-/micro-sized SiC/Al $_2O_3$  composites were significantly improved in comparison with the monolithic  $Al_2O_3$ . 7.6 MPa m $^{1/2}$  was the highest fracture toughness and was found in the composite with 5% SiC, while the 20% SiC composite exhibited the highest flexural strength. The toughening and strengthening mechanisms of the ceramic composites were discussed.

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#### 1. Introduction

Structural ceramics including Al<sub>2</sub>O<sub>3</sub> exhibit excellent properties such as high thermal resistance, good chemical stability and moderate to high mechanical strength. However, the fracture toughness of the materials is low because dislocation movement is extremely limited by their ionic and/or covalent bonds. The brittleness and poor damage tolerance have so far limited their application as advanced engineering materials [1]. During the past two decades, much effort has been made to improve the strength and toughness of ceramic materials. Several useful methods have been proposed some of which are crack-surface bridging [2], particle dispersion of different phases in a matrix [3], fiber reinforced composites [4], macroscopic crack deflection [5], and phase transformation induced toughening as revealed by zirconia [6]. The addition of one or more components into the base material to form ceramic matrix nano-composites (CMNCs) has been found to be effective to enhance the fracture toughness and to improve strength [7–15]. Usually in so doing an intragranular nano-structure is formed in nano-sized or nano-/micro-sized ceramic composite materials [7,15]. While some earlier studies emphasized that micro-sized particles still have priority to the toughening of ceramic composites [7,16], in this case intragranular nano-structure could also be formed [14].

Recently, the grain size effects on the properties of  $Al_2O_3/SiC$  composites have been thoroughly studied [17–19]. In order to promote the application of these advanced materials, comprehensive study should cover material compositions, processing techniques, reinforcing and toughening mechanisms, and the subsequent properties. In the present work, the formation of intragranular nano-structures is investigated in the  $Al_2O_3$  ceramic composites reinforced with SiC particles with widely varied sizes. The morphology and formation mechanism of the intragranular nano-structures are investigated along with its mechanical properties.

#### 2. Material and experimental methods

The starting materials used were high purity  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> ( $\geq$ 99.99%) and SiC powders. The average size of the as-received high purity  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> powders is 1  $\mu$ m. The SiC powders used in this study has an average particle size of 1  $\mu$ m, however, the particle size scatters from 100 nm to 1.9  $\mu$ m. In order to disperse the agglomerated particles, the powder mixtures were first milled for 30 min with ethanol as a dispersant agent. Subsequently, they are dried in a drying oven and sieved through a 60-mesh screen. The as-prepared Al<sub>2</sub>O<sub>3</sub> powders containing 1 wt.%, 2 wt.%, 5 wt.%, 10 wt.%, 15 wt.% and 20 wt.% SiC (thereafter denoted as AS1, AS2, AS5, AS10, AS15, AS20, respectively) were ball-milled with Al<sub>2</sub>O<sub>3</sub> grinding media in de-ionized water for 4 h. The homogeneous slurry was then dried and degassed in a drying oven. The resulting powder cake was then put into a plastic bag and de-agglomerated with a rolling pin. Next, the powder was passed through a 60-mesh screen sieve. The as-prepared pow-

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der was poured into a graphite die and care was taken to ensure that the powder was distributed evenly within the die cavity. Hotpressing was carried out at 1635 °C and 25 MPa in a vacuum for 1 h to fabricate thick diskettes with dimensions of 30 mm in diameter and 5 mm in thickness. The BN powder was chosen as the high temperature lubricant. For comparison, a monolithic  $Al_2O_3$  compact (denoted as ASO) was also consolidated using the same hot-pressing conditions.

The density of ceramic materials was measured using the Archimedes method, with de-ionized water as the immersion medium. The relative density was calculated based on the theoretical density of single phase alumina and the ROM (rule of mixture) density of ceramic composite. An average density was obtained from 6 to 10 measurements for each material. Specimens used for hardness and fracture toughness measurements were polished by diamond grinding powder down to 1.5  $\mu m$ . Vickers hardness was measured on the polished surface with a load of 98 N for 15 s using an Hv-50A Hardness Tester. Ten points with intervals of 2.5 mm were made along the diameter for each sample. The fracture toughness was measured using the indentation method.

The flexural strength was measured by three-point bending. Specimens had dimensions of  $30\,\mathrm{mm} \times 4\,\mathrm{mm} \times 3\,\mathrm{mm}$  (length  $\times$  width  $\times$  thickness). The span of the three-point bending fixture was  $18\,\mathrm{mm}$ . A Shimadzu servo-hydraulic testing machine was used with a crosshead speed of  $0.5\,\mathrm{mm/min}$ . At least three specimens were tested for each composite all of which were at room temperature. Fracture surfaces of specimens were observed by a scanning electron microscope (SEM, model JEOL JSM-5600LV, operating at  $20\,\mathrm{kV}$ ). A thin layer of gold was coated on specimen surfaces to eliminate charging effects under electron irradiation.

#### 3. Results and discussions

#### 3.1. Density of the ceramic composites

The relationship between relative density and SiC content (wt.%) in Al $_2$ O $_3$ /SiC composites is plotted in Fig. 1. It can be seen that ASO, AS1 and AS2 all approached full density. AS1 had the highest relative density of 99.8%. The density of AS2 was slightly higher than monolithic Al $_2$ O $_3$  but lower than AS1. For samples AS5 through AS20, the relative density decreased with increasing SiC content, all having lower relative densities than that of monolithic Al $_2$ O $_3$ . This is mainly attributed to the incorporation of SiC particles that blocked

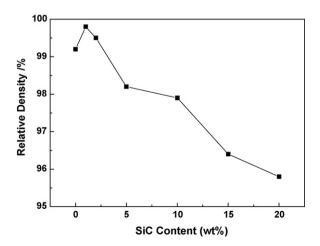


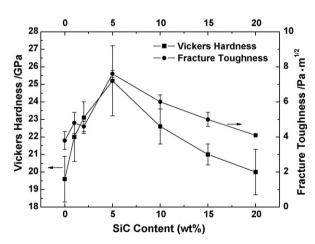
Fig. 1. Relative density versus SiC contents in Al<sub>2</sub>O<sub>3</sub>/SiC ceramic composites.

grain boundary movement and hindered the densification of  $Al_2O_3$  [20]. In fact, silica and other impurities are present in the raw SiC powder, which could serve as sintering additives. Furthermore, SiC particles can fill pores within  $Al_2O_3$  particles. While inclusions of second phase particles may suppress grain growth, the particles will also obstruct densification [7,21]. As for AS1 and AS2, the former factor appears to be dominant, and hence the density is higher than that of monolithic  $Al_2O_3$ . In order to achieve a sufficiently high, higher sintering temperatures and/or higher external pressures are required.

#### 3.2. Mechanical properties

The variations of Vickers hardness and fracture toughness versus SiC content in Al<sub>2</sub>O<sub>3</sub>/SiC composites are shown in Fig. 2. It can be observed that the Vickers hardness increased with increasing SiC addition from 0 wt.% to 5 wt.%. Further increases in SiC (up to 20 wt.%) however, resulted in decreasing the Vickers hardness. The results shown in Fig. 2 also revealed that a large scattering of hardness was observed for each individual composite, which may be due to the presence of residual internal stresses resulting from the thermal expansion mismatch between Al<sub>2</sub>O<sub>3</sub> and SiC. The Vickers hardness is known to be very sensitive to the residual internal stresses [22]. The average Vickers hardness of all samples is higher than that of unreinforced monolithic Al<sub>2</sub>O<sub>3</sub>, which is consistent with the results obtained by Ko et al. [23]. The improved hardness can be attributed to the addition of hard secondary SiC particles. For AS10-AS20, the lower relative density may have a negative effect on the properties.

Because of the simplicity of specimen preparation and the acceptance of a small specimen, the measured fracture toughness from Vickers indentations has been widely accepted, even though the results show discrepancies comparing to that obtained with standard methods, for instance the precracked beam method [24]. The Vickers indentation method was employed to measure the fracture toughness of the composites. It is noted that the variation of fracture toughness in the samples generally follows the trend identified above for hardness. The highest fracture toughness was found to be 7.6 MPa m $^{1/2}$  in composite containing 5 wt.% SiC particles. Flexural strengths obtained by three-point bending test are listed in Table 1. Strength values of all samples are higher than that of the monolithic Al $_2$ O $_3$ . The strengths of AS5 and AS20 ceramic composites are 30% and 64% higher in comparison to monolithic Al $_2$ O $_3$ , respectively.



**Fig. 2.** Vicker's hardness and fracture toughness versus SiC content in Al<sub>2</sub>O<sub>3</sub>/SiC ceramic composites.

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