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Mechanical properties and toughening mechanisms of silicon carbide nano-particulate reinforced Alon composites

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ABSTRACT

Aluminum oxynitride (Alon) has been considered as a potential ceramic material due to excellent stability, chemical and mechanical properties such as high rigidity and good chemical stability, but it has relatively low strength and poor fracture toughness. The aim of this study was to investigate a type of silicon carbide (SiC) nano-particulate reinforced Alon composites with improved mechanical properties and fracture resistance via hot-press sintering. The addition of SiC nano-particles resulted in a reduction of both porosity and grain size, and a change of fracture mode from intergranular cracking in the monolithic Alon to intragranular cracking in the composites due to the pinning effect of SiC nano-particles positioned at grain boundaries or triple junctions of micro-sized Alon particles. With 8 wt% SiC nanoparticles addition, the relative density, microhardness, Young's modulus, flexural strength, and fracture toughness all increased. Different toughening mechanisms including crack bridging, crack branching and crack deflection were observed, thus effectively increasing the crack propagation resistance and leading to a considerable improvement in the flexural strength and fracture toughness of the composites.

1. Introduction

Spinel-type aluminum oxynitride (Alon) is a solid solution of Al_2O_3 and AlN [1]. Since Yamaguchi and Yanagida [2] first reported the Alon synthesis method by carbothermic reduction and nitridation of Al_2O_3, Alon ceramic materials have been investigated by many scholars [3–5]. Alon ceramic is a good potential candidate ceramic material for a variety of applications such as high-performance structural and advanced refractory due to its excellent mechanical and chemical properties such as high rigidity, good chemical stability and good wear resistance against steel [6,7]. Additionally, it also can be processed into a fully dense transparent material which shows promising mechanical and optical properties suitable for use in infrared and visible window applications [8].

Silicon carbide (SiC) is becoming an attractive material because of its excellent chemical and physical properties such as high chemical and thermal stability with a melting point of 2730 °C, high hardness, wear and creep resistance, low density and low coefficient of thermal expansion [9,10]. Silicon carbide particles were normally added into other ceramics such as ZrB_2 [11,12], Al_2O_3 [13,14], mullite (-ZrO_2) [15] and Sialon [16] to improve their mechanical properties and thermal properties allowing toughening effects by mechanisms such as microcracking [17]. Furthermore, when the temperature was above 1100 °C, the SiC addition could heal the cracks and toughen ceramic materials such as ZrB_2 [18], Al_2O_3 [19] and AlN [20].

During the past two decades, much effort has been made to improve the mechanical properties such as flexural strength and fracture toughness of Alon ceramic and its composites. It could be improved by adding other particles, e.g. Al_2O_3 [21], ZrO_2 [22], TiN [23] and Sialon [24]. Djenkal et al. [17] added the SiC particles into the Al_2O_3–Alon ceramic in the form of powders or platelets. Mandal et al. [25] investigated SiC–Alon composites with the range of 10–55 wt% of Alon as additive sintered in both a N_2 and an Ar atmosphere. Furthermore, it was proven that the nano-sized SiC particles could effectively improve the oxidation resistance of Alon ceramic [26]. However, to the authors' knowledge, there is rare report on the mechanical properties of SiC-added Alon composites in the open literature. The questions remain if and to what extent the SiC addition can improve the mechanical properties of Alon ceramic and what the toughening and strengthening mechanisms are. The aim of this

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work was, therefore, to develop SiC–Alon particulate composites, evaluate the microstructure and mechanical properties, and identify the underlying toughening and strengthening mechanisms.

2. Materials and methods

2.1. Starting materials and processing

The powder materials used for preparation of SiC–Alon composites were commercial α-Al_2O_3 (40–100 nm, Nanjing Emperor Nano Material Co., Ltd., China, 99.5%), AlN (30–100 nm, Shenzhen Honesty Nano-Tech Co., Ltd., China, 99%), β-SiC (40–90 nm, Nanjing Emperor Nano Material Co., Ltd., China, 99.5%) and Al powders (4–8 μm, Gaizhou City Metal Powder Factory, China, 99.63%). Appropriate amounts of Al_2O_3, AlN and Al powders in the weight ratio of 81:18:1 were intimately mixed by attrition milling using ethanol as the liquid medium for 24 h. The obtained mixtures were dried and then placed in a graphite mold to synthesize Alon powders at 1750 °C for 2 h in nitrogen atmosphere using solid synthesis method. Then, amount of 8 wt% SiC was introduced into Alon powders and mixed by ball milling using ethanol as the liquid medium for 24 h. The mixtures were dried at 80 °C for 24 h and sieved through a 180 μm mesh. The thoroughly mixed powders were placed in a graphite die to sinter SiC–Alon composites at 1850 °C with an applied uniaxial load of 25 MPa for 40 min in nitrogen atmosphere and then furnace-cooled down to room temperature. For comparison, Alon powders were also hot pressed to fabricate Alon ceramic in the same manner without adding SiC powders in present study.

2.2. Characterization

The bulk density and apparent porosity of sintered samples were measured by the Archimedes principle method in distilled water, and the theoretical density was calculated by a volumetric rule of mixtures. The phase and crystallinity were analyzed via X-ray diffraction (XRD) using CuK_α radiation at 45 kV and 40 mA, with a step size of 0.05° and duration of 1 s in each step. Thin disks (3 mm Ø) of the sintered composites were subsequently prepared by ultrasonic cutting, dimpling, polishing, and ion milling. Microcharacterization was performed using transmission electron microscopy (TEM) and electron diffraction (ED). The microstructures of polished and fractured surfaces were analyzed by scanning electron microscopy (SEM). The elemental concentration profile of the microstructure was determined using energy dispersive X-ray spectroscopy (EDS). The Vickers microhardness was evaluated on the polished surface using a computerized microhardness tester at a load of 4.9 N for a dwell time of 15 s in accordance with ASTM C1327. Eleven indentations were made on each sample and the average values were reported.

The three-point bending (TPB) samples, with a dimension of 25 mm × 2 mm × 2 mm, were ground and polished to a surface finish using 1 μm diamond paste. The edges of all the specimens employed in flexural strength testing were chamfered to minimize the effect of stress concentration due to machining flaws. The TPB tests were performed with 1 kN load cell and using a TPB stage with a span of 20 mm attached to a computerized Instron 8801 fatigue testing system. In the TPB tests the cross-head speed was set at a rate of 0.2 mm/min in accordance with ASTM C1161. The values for bending strength σ, and Young's modulus E were calculated according to the following equations [22]:

$$\sigma = \frac{3FL}{2bh^2}, \quad (1)$$

$$E = \frac{kL^3}{4bh^3}, \quad (2)$$

Table 1
The properties of fully dense Alon and sintered pure Alon ceramic and 8 wt% SiC–Alon composites.

Properties	Fully dense Alon [1,29]	Pure Alon	8 wt% SiC–Alon
Relative density (%)	100	97.6	99.6
Apparent porosity (%)	0	0.57	0.31
Microhardness (GPa)	14.6	13.78	14.07
Young's modulus (GPa)	307	237.2	261.6
Flexural strength (MPa)	306	295.6	398.5
Fracture toughness (MPa m$^{1/2}$)	2.0	1.54	1.90

where F is the load at failure (N), L is the span (mm), b is the sample width (mm) and h is the thickness (mm), k is the slope ($dF/d\nu$) in the load-deflection curve. To evaluate the slope k more accurately, a load range from 5 N to the maximum load was used in all TPB tests. Also, five samples were prepared and tested in each group, and the average values were reported.

Fracture toughness was evaluated using a Vickers indentation technique [27]:

$$K_C = \chi\left(\frac{E}{H}\right)^{1/2}\frac{F}{c^{3/2}}, \quad (3)$$

where F is the applied load (N), E is the Young's modulus (GPa), H is the Vickers hardness (GPa), a is the radial crack length (m) measured from the center of indent and χ is an empirically determined "calibration" constant taken to be 0.016 ± 0.004 [28]. The fracture surfaces of the sintered SiC–Alon composites after the TPB tests were examined via SEM to identify the fracture mechanisms. To achieve a better resolution the SEM observations were done after the fracture surfaces were sputter-coated with a thin film of gold.

3. Results

The SEM micrographs of Alon and SiC powders as well as the XRD patterns of pure Alon and 8 wt% SiC–Alon composites were reported in [26]. It can be seen clearly that the SiC powders were nano-sized. And no other new phase or impurity could be identified in the both patterns of sintered Alon and SiC–Alon composites. Thus, it can be suggested that the structure of Alon and SiC–Alon composites obtained in the present study was thermally stable enough after hot-press sintered at 1850 °C in a nitrogen atmosphere.

3.1. Density and mechanical properties

The relative density, apparent porosity, microhardness and mechanical properties such as flexural strength and fracture toughness of fully dense Alon [1,29] and sintered pure Alon ceramic and 8 wt% SiC–Alon composites are given in Table 1. Djenkal et al. [17] reported the added SiC would bring about a sintering inhibiting effect on the Alon matrix. However, in present study, while a relative density of 97.6% for pure Alon was obtained, a relative density of 99.6% for the composites with the addition of SiC particles was achieved using the hot-press sintering process. It is clear that the nano-sized SiC particles played a key role in the densification of SiC–Alon composites. A similar result was also reported in ZrB_2-based ceramic [11]. Furthermore, it can be seen that the added nano-sized SiC particles played an important role in enhancing the mechanical properties of Alon ceramic. The flexural strength and fracture toughness of 8 wt% SiC–Alon were 34.8% and 23.4% higher in comparison to pure Alon, respectively. A detailed explanation on underlying toughening mechanisms as well as the properties enhanced for nano-sized SiC addition will be given in the section of discussion.