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Original Article

Microstructure and improved mechanical properties of Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites with YSZ addition

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ABSTRACT

Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites were fabricated by solid-state reaction sintering of Al_2O_3, $BaZrO_3$, and yttria stabilized zirconia (YSZ) powders. The effects of YSZ addition on microstructure and mechanical properties have been investigated. The incorporation of YSZ promoted the densification of the composites and formation of tetragonal ZrO_2 phase. The microstructure of the composites was characterized by elongated Ba-β-Al_2O_3 phase and equiaxed ZrO_2 particles including added YSZ and reaction-formed ZrO_2. The Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites with YSZ addition exhibited improved fracture toughness, as a result of multiple toughening effects including crack deflection, crack bridging, crack branching, and martensitic transformation of ZrO_2 formed by the reactions between Al_2O_3 and $BaZrO_3$. Moreover, owing to the grain refinement of Al_2O_3 matrix, dispersion strengthening of the added YSZ particles, and an increase in density of the composites, the Vickers hardness and flexural strength of Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites were dramatically enhanced in comparison with the composites without YSZ addition.

1. Introduction

Al_2O_3 is one of the most widely used ceramic materials because of its high strength and hardness, excellent heat and wear resistance. Nevertheless, the large-scale applications of monolithic Al_2O_3 ceramic are very limited due to its relatively low fracture toughness. Many studies have been carried out to improve the fracture toughness of Al_2O_3 by incorporating reinforcements with high aspect ratios (such as fibers, whiskers, and platelets) to form ceramic matrix composites (CMCs) [1–4]. Compared to direct additions of such reinforcements, the incorporation of elongated reinforcements with high aspect ratios through in-situ reactions has advantages of reducing processing costs, as well as obtaining denser and more homogeneous microstructure.

Self-reinforced Al_2O_3 matrix composites with improved fracture toughness have been developed by incorporating various hexaaluminate compounds such as $CaAl_{12}O_{19}$ [5–7], $SrAl_{12}O_{19}$ [8,9], $Ba_{0.75}Al_{11}O_{17.25}$ (one of Ba-β-Al_2O_3 compounds) [10,11], $LaAl_{11}O_{18}$ [12,13], and $LaMgAl_{11}O_{19}$ [14,15]. The formation of these hexaaluminate compounds with elongated morphologies resulted in enhancement of fracture toughness of Al_2O_3 ceramic due to crack deflection and crack bridging. Meanwhile, the phase transformation of ZrO_2 from tetragonal (*t*) to monoclinic (*m*) has been widely used to improve the fracture toughness of Al_2O_3 [16–19]. Based on above-mentioned toughening mechanisms, some researchers [20–22] have introduced multiple reinforcing phases into Al_2O_3 matrix. The in-situ formed $SrAl_{12}O_{19}$, $LaAl_{11}O_{18}$, and $CaAl_{12}O_{19}$ enhanced the fracture toughness of zirconia toughened alumina (ZTA) composites. Nevertheless, almost all studies dealt with the influences of elongated reinforcements on microstructure and mechanical properties of ZTA composites. So far, little research work has been focused on the effects of YSZ addition on microstructure and mechanical properties of Al_2O_3/hexaaluminate composites. Burger et al. [23] prepared an Al_2O_3 matrix composites containing homogeneously distributed metastable ZrO_2 particles and in-situ formed hexagonal $SrAl_{12}O_{19}$ platelets and investigated the effects of ZrO_2 addition on strength, fracture toughness, and wear resistance of Al_2O_3/$SrAl_{12}O_{19}$ composites.

Chen et al. have examined the in-situ synthesis of Al_2O_3 matrix composites by using reactive sintering of Al_2O_3 and $BaZrO_3$ powders [10,11]. The elongated Ba-β-Al_2O_3 phase with a layered β-Al_2O_3 structure and equiaxed ZrO_2 particles were formed during sintering period. Since the reaction-formed ZrO_2 is completely monoclinic phase (*m*-ZrO_2), the fracture toughness enhancement of the obtained Al_2O_3 matrix composites is mainly attributed to the contribution of Ba-β-Al_2O_3 phase through crack deflection and crack bridging.

In the current work, an attempt has been made to introduce yttria-stabilized zirconia (YSZ) powder to Al_2O_3 and $BaZrO_3$ powder mixtures

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Table 1
Nominal compositions of Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites (vol%).

Samples	Al_2O_3	Ba-β-Al_2O_3	*m*-ZrO_2	3YSZ
AB-2.5Z	70	27.5	2.5	0
AB-8Z	72	20	1.8	6.2
AB-16Z	64	20	1.8	14.2

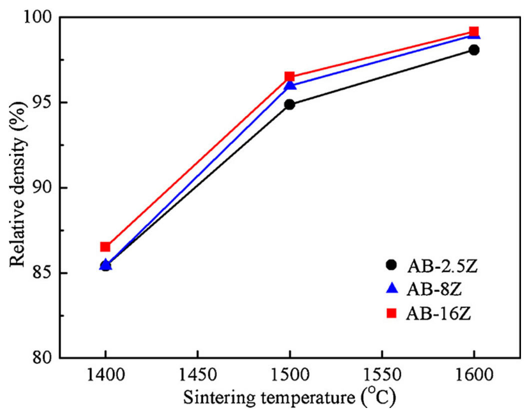

Fig. 1. Relative density of in-situ synthesized samples sintered at different temperatures.

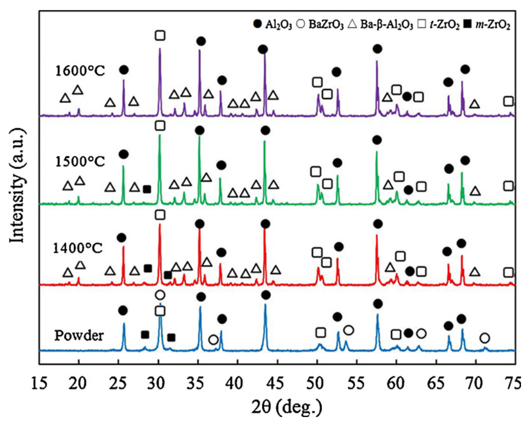

Fig. 2. XRD patterns of mixed powder and sintered AB-8Z samples at different sintering temperatures.

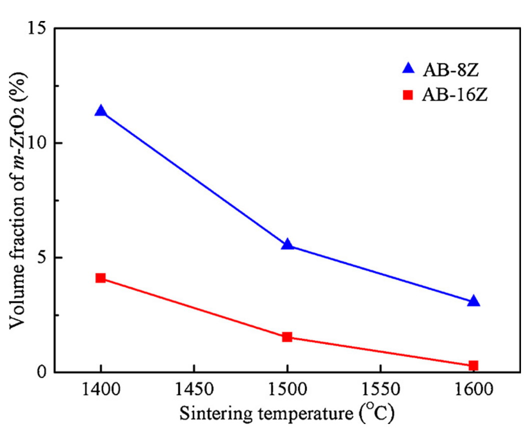

Fig. 3. Volume fractions of monoclinic ZrO_2 phase in AB-8Z and AB-16Z samples as a function of sintering temperature.

and to promote the formation of tetragonal zirconia (*t*-ZrO_2) during reactive sintering. In this way, the fracture toughness of the Al_2O_3 matrix composites containing both Ba-β-Al_2O_3 and *t*-ZrO_2 may be further improved as a result of multiple toughening effects including crack deflection, crack bridging, and martensitic transformation of ZrO_2 from tetragonal to monoclinic phase. The objective of this paper was to clarify the effects of YSZ addition on densification behavior, phase evolution, microstructure, and mechanical properties of Al_2O_3/Ba-β-Al_2O_3/ZrO_2 composites and to understand the mechanisms of improving the mechanical properties of these in-situ synthesized Al_2O_3 matrix composites.

2. Experimental procedure

The starting materials used in this study were Al_2O_3 (average particle size 0.16 µm, 99.99% purity; Taimei Chemicals, Tokyo, Japan), $BaZrO_3$ (1.5 µm, 99% purity; Kojundo Chemical Lab., Saitama, Japan), and 3YSZ (3 mol% Y_2O_3-ZrO_2 (TZ-3Y); 50 nm, 99.9% purity; Tosoh, Tokyo) powders. The raw powders with nominal compositions listed in Table 1 were mixed in ethanol for 24 h at a speed of 90 rpm with Al_2O_3 balls of 5 mm diameter (grade SSA-995, NIKKATO, Osaka, Japan) as milling media. For simplicity's sake, in the present paper, the composites containing both reaction-formed ZrO_2 and added YSZ are expressed as Al_2O_3/Ba-β-Al_2O_3/ZrO_2, whereas Al_2O_3/Ba-β-Al_2O_3/*m*-ZrO_2 implies the composites without YSZ addition. After milling, the slurries were dried at 120 °C for 24 h. The disk-shaped green compacts (*Φ*19 mm × 3 mm) were prepared by uniaxial pressing at 20 MPa for 2 min, followed by cold isostatic pressing at 200 MPa for 2 min. The obtained green compacts were sintered in an electric furnace at 1400, 1500, and 1600 °C in air with a heating rate of 400 °C/h and a dwelling time of 1 h at each temperature. The specimens were then cooled down to room temperature at a rate of 400 °C/h. In addition, some AB-8Z specimens were subjected to the following thermal shock test. The specimens were heated from room temperature to 1100 °C with a heating rate of 400 °C/h in a furnace. After heating at 1100 °C for 15 min, the specimens were taken out of the furnace and cooled in air to room temperature.

Density values of the sintered samples were measured in distilled water using the Archimedes principle. The phase identification of mixed powder, sintered samples, and fractured surfaces was performed by X-ray diffraction (XRD; RINT-TTR III, Rigaku, Japan) using Cu Kα radiation with a step of 0.02° (2*θ*) and a scanning rate of 2°/min ranging from 15° to 75°. The volume fractions of monoclinic and tetragonal ZrO_2 phases (V_m and V_t) were calculated by the modified Garvie and Nicholson equation [24].

$$X_m = \frac{I_{m(1\,1\,1)} + I_{m(1\,1\,\bar{1})}}{I_{m(1\,1\,1)} + I_{m(1\,1\,\bar{1})} + I_{t(1\,1\,1)}} \tag{1}$$

$$V_m = \frac{1.311X_m}{1 + 0.311X_m} \tag{2}$$

$$V_t = 1 - V_m \tag{3}$$

where X_m is the intensity ratio. $I_{t(1\,1\,1)}$, $I_{m(1\,1\,\bar{1})}$, and $I_{m(1\,1\,1)}$ are the peak intensities corresponding to the tetragonal (1 1 1), monoclinic (1 1 $\bar{1}$), and monoclinic (1 1 1) planes, respectively.

Microstructural observations and compositional analyses were conducted by scanning electron microscopy (SEM; JXA-9800RL, JEOL, Japan) and electron probe micro-analyzer (EPMA; JXA-8800, JEOL), respectively. In addition, transmission electron microscopy (TEM; JEM-2010, JEOL) attached with energy-dispersive spectroscopy (EDS) was also used for microstructural evaluation. Thermal etching was performed by heating the polished samples in air to 1300 °C for 30 min for the purpose of measuring the grain sizes of the Al_2O_3 matrix. The average grain size of the Al_2O_3 matrix was determined quantitatively using the linear intercept method with the 1.56 correction, and the aspect ratio of the Ba-β-Al_2O_3 phase was obtained by directly measuring the length and width on SEM micrographs. These measurements were performed on two different random areas for each sample, which included at least 500 grains.