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Mechanical properties and structure of zirconia-mullite ceramics prepared by in-situ controlled crystallization of Si-Al-Zr-O amorphous bulk

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Abstract: Zirconia-mullite nano-composite ceramics were fabricated by in-situ controlled crystallization of Si-Al-Zr-O amorphous bulk, which were first treated at 900−1 000 ℃ for nucleation, then treated at higher temperature for crystallization to obtain ultra-fine zirconia-mullite composite ceramics. The effects of treating temperature and ZrO_2 addition on mechanical properties and microstructure were analyzed. A unique structure in which there are a lot of near equiaxed t-ZrO_2 grains and fine yield-cracks has been developed in the samples with 15% zirconia addition treated at 1 150 ℃. This specific microstructure is much more effective in toughening ceramics matrix and results in the best mechanical properties. The flexural strength and fracture toughness are 520 MPa and 5.13 MPa·$m^{1/2}$, respectively. Either higher zirconia addition or higher crystallization temperature will produce large size rod-like ZrO_2 and mullite grains, which are of negative effect on mechanical properties of this new composite ceramics.

Key words: Si-Al-Zr-O amorphous bulk; crystallization; zirconia-mullite composite; structure

1 Introduction

Mullite has been widely investigated because of its outstanding importance as potential candidate of single-phase ceramics, composite ceramics for high temperature structured applications due to its favorable thermal and mechanical properties[1−4]. However, wider applications would be obtained only if its low flexural strength (150 MPa) and low fracture toughness (1.8 MPa·$m^{1/2}$) could be improved. Many strategies have been developed to improve the mechanical properties of mullite ceramics such as adding ZrO_2 component[5−7] and dispersing SiC particles[8], carbon nanotubes[9] and other micro-or nanoparticles in the mullite ceramic matrix as reinforcing phases.

Dispersing metastable tetragonal zirconia (t-ZrO_2) particles in a mullite matrix is a well-known and relatively cheap route to reinforce mullite[6−7]. The principles of the reinforcement are based on the existence of tetragonal zirconia in which the phase transformation toughening effect could be resulted. Particularly, by adding stabilizing agents, transformation of tetragonal zirconia (t-ZrO_2) to monocline zirconia (m-ZrO_2) in cooling process could be prohibited and much better toughening effect could be obtained[6−8, 10−11].

Many other unconventional ways have been used to prepare zirconia-mullite ceramics[12−14]. In this work, ultra-fine zirconia-mullite composite ceramics were prepared by in-situ controlled crystallization of Si-Al-Zr-O amorphous bulk[15−16]. This method can avoid aggregation of nano-size starting powder, and receive higher densification which is hard to be achieved by nano-sized powder sintering. MONICA et al[17] has ever reported the crystallization of quenched Al_2O_3-ZrO_2-SiO_2 glasses but failed to receive high quality composite ceramics with high performance.

The purpose of this work is to report the processing and characterization of zirconia-mullite ceramics prepared by the new method. Mechanical properties, microstructure and the crystallization behavior were also evaluated.

2 Experimental

2.1 Samples preparation

The batch powder contented 30%−45% SiO_2, 30%

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−40% Al_2O_3, 10%−25% ZrO_2 (mass fraction) and a small amount of MgO, CaO, etc. They were mixed and homogenized by conventional ball milling with zirconia balls for 10 h. Then 20 g of mixed powders were put into Al_2O_3 crucible which was heated in air at temperatures in the range of 1 620−1 700 ℃ for 2−4 h in an electric furnace. The homogeneous flux was thereafter poured into a stainless mold and cooled with liquid nitrogen to produce amorphous bulks. Afterward, the as-received amorphous bulks were first treated at 900−1 000 ℃ for nucleation, then treated at higher temperature for crystallization to obtain nano-composite zirconia-toughened mullite ceramics. Samples numbered as Z15, Z18 and Z20 indicated zirconia mass fraction of 15%, 18% and 20%, respectively.

2.2 Characterization

Fracture toughness was determined for all the samples using a Vicker's hardness tester with a Vicker's indentor and a load of 9.8 N (MICRODUROMAT4000). Flexural strength was tested in three points bending on 3 mm×4 mm×35 mm, using a 30 mm span and a crosshead speed of 0.5 mm/min(CSS-44100). The reported values were the average data obtained from four tests of samples with the same raw powder composition and heat-treating procedure.

Phase compositions were measured by X-ray diffraction using Japanese D/MAX 2500VB instrument in a step-scanning mode with Ni-filtered Cu K_α as the radiation source and the radiation was over a range of 10°−80°. The volume fractions of tetragonal zirconia (φ_t) were calculated by the following equations[18]:

$$\varphi_t=1-\varphi_m \quad (1)$$

where φ_m is the volume fraction of m-ZrO_2, which can be calculated by

$$\varphi_m=\frac{PX_m}{1+(P-1)X_m} \quad (2)$$

where X_m is the integrated intensity ratio, and P=1.340.

$$X_m=\frac{I_{m(\bar{1}11)}+I_{m(111)}}{I_{m(\bar{1}11)}+I_{m(111)}+I_{t(101)}} \quad (3)$$

where I_m and I_t are the peak heights of m-ZrO_2 and t-ZrO_2, respectively.

After crystallization, the bulk density of samples was measured using the Archimedes' technique. The microstructure was examined by a Siri-on200s canning electronic microscope(SEM). The bulk samples for SEM testing were etched by 1% (volume fraction) hydrofluoric acid-water solution after being polished and washed 3 times with deionized water. The crystallization status of t-ZrO_2 was examined by selected-area electron diffraction to powder samples using a Tecnai $G^2$20 S-TWIN transmission electron microscope(TEM).

3 Results and discussion

3.1 Effects of ZrO_2 content and heat-treatment temperature on mechanical properties

Fig.1 shows the flexural strength and fracture toughness data of samples Z15, Z18 and Z20 treated at 1 150 ℃ and 1 200 ℃ for 1 h crystallization, respectively. From the results, it can be found that the mechanical properties decrease with the increasing of heating temperature. Samples heat-treated at 1 150 ℃ for 1 h have better mechanical properties than those treated at 1 200 ℃, but with an exception of the fracture toughness of Z18. Sample Z15 heat-treated at 1 150 ℃ has the conditional optimized properties with 520 MPa of flexural strength and 5.13 MPa·$m^{1/2}$ of fracture toughness, respectively. The flexural strength is 40% higher than that of the zirconia-toughened mullite ceramics which are fabricated by conventional means [10]. This suggests that in order to obtain better mechanical properties, it is better to control the heat-treatment temperature and let it not exceed 1 150 ℃. From Fig.1, it also can be found that the mechanical properties decrease with the increasing content of ZrO_2 in starting materials after the content of ZrO_2 exceeds 15%. This means that the further increase of zirconia after 15% ZrO_2 addition has little help for the mechanical property improvement. That is, the optimum content of ZrO_2 in starting materials should not be over 15%.

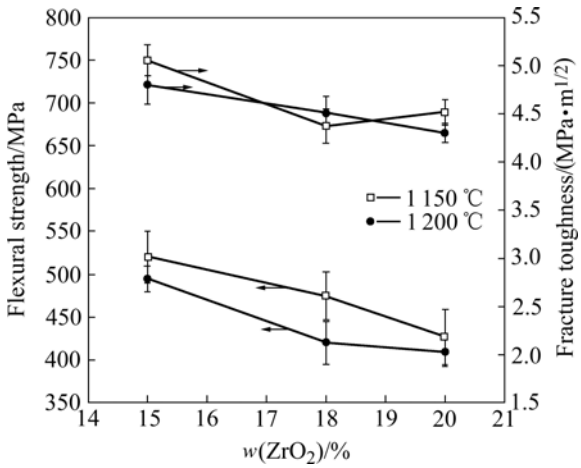

Fig.1 Mechanical properties of samples Z15, Z18 and Z20 treated at different temperatures

3.2 Phases analysis

Fig.2 shows the X-ray diffraction patterns of samples Z15, Z18 and Z20 treated at different temperatures. The nucleation test results at 900 ℃, 1 000 ℃ for Z20 sample are also given. The precipitated

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