

Al₂O₃-fiber-reinforced porous YSZ ceramics with high mechanical strength

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Abstract

Porous YSZ ceramics were prepared by a gel-casting method using tert-butyl alcohol (TBA) as solvent and Al₂O₃ fiber as reinforcement. Effects of sintering temperature, soaking time and Al₂O₃ fiber content on porosity, microstructure, compressive strength, and fracture toughness of the porous ceramics were studied. Compressive strength of porous YSZ ceramics was improved while the porosity decreased with the increase of sintering temperature and soaking time. Addition of Al₂O₃ fibers significantly strengthened and toughened porous YSZ ceramics; however, the laws of strengthening and toughening were different. The strength of the porous YSZ ceramics reached maximum at the addition of 10 vol% Al₂O₃ fibers, and the corresponding compressive strength and flexural strength were 100.2 ± 25.4 MPa and 61.5 ± 11.3 MPa, respectively. Fracture toughness of the porous YSZ ceramics monotonically increased with increasing of Al₂O₃ fibers, and could be improved from $0.5 \text{ MPa m}^{1/2}$ to $1.2 \text{ MPa m}^{1/2}$ (improved by 140%). The strengthening and toughening mechanisms of Al₂O₃ fibers were discussed.

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1. Introduction

Yttria-stabilized zirconia (YSZ) is widely used as refractory materials, aerospace structural materials, and biological materials due to its excellent high mechanical strength, chemical stability, high temperature stability, and low thermal conductivity [1–4]. As heat insulation materials, introducing pores into the zirconia ceramics can obviously lower thermal conductivity because air trapped in the pores is a better thermal insulator. Recently, widespread interests have been focused on fabricating porous zirconia ceramics to lower their thermal conductivity. However, the strength of the porous ceramics decreases exponentially with increasing porosity as a result of the weakened skeleton structure of the ceramic materials. The main contradiction between high porosity (light weight) and mechanical strength has limited zirconia ceramics in applications as heat insulation structural materials. Therefore, preparing high-porosity yet high-strength zirconia ceramics is a challenging but valuable task.

One way to strengthen porous ceramics is either to tailor the pore microstructures or to strengthen the skeletons. On one hand, many research works were focused on improving the uniformity of pore microstructure, reducing pore size, optimizing the pore shape and so on. These works have got some good progress [5–10]. For example, Rizwan Ahmad improved porous Al₂O₃ ceramic mechanical properties through crack healing and improvement of the surface condition by dip-coating. In his paper [5], the compressive strength is improved by 200%. And Guogang Xu says in his paper [7], “The narrower pore size distribution and regular microstructure of the pores are beneficial to improve the filtration accuracy and strength of the porous ceramics.”

On the other hand, strengthening the skeleton can remarkably improve the strength of porous ceramics [11,12]. Bin Zhao improved mechanical properties of porous ZrB₂–SiC specimens by doping with tungsten which support a high strength skeleton for the samples [12]. Fiber-reinforced ceramic matrix composites have been extensively studied for fully-dense ceramics, and it has been shown that fibers as reinforcements show good reinforcing effects for fully-dense ceramics [13–16]. However, few studies [17] have shown fiber reinforcements for

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porous ceramics. So the question whether fibers can effectively reinforce porous ceramics is still unresolved.

In this paper, high-strength Al_2O_3 fiber is chosen to reinforce porous YSZ ceramics because the Al_2O_3 fiber was proved to be stable with YSZ at high temperature and to effectively reinforce porous YSZ ceramics in the earlier work of the present authors [18]. Porous YSZ ceramics are prepared by gel-casting and pressureless sintering process. The influences of sintering conditions and fiber content on porosity, microstructure, compressive strength, and fracture toughness were investigated, and the reinforcing mechanism of Al_2O_3 fiber-reinforced porous YSZ ceramics was also discussed. This work is one of first studies on the fiber-reinforced porous materials.

2. Experiment

2.1. Raw materials

Commercially available YSZ powder (ZrO_2 –8 mol% Y_2O_3 , Fanmeiya Powders Co., Ltd., Jiangxi province, China) was used as raw materials, which has a mean particle size (d_{50}) of 1.26 μm . Al_2O_3 fiber (Zhejiang Osmun crystal fiber Co., Ltd., Deqing, Huzhou, Zhejiang province, China) is about thousands of micrometers in length and 4–8 μm in diameter. Fibers were cut into 1–2 mm long before mixing with the YSZ powder. Tert-butyl alcohol (TBA, chemical purity, Beijing Yili Chemical Co., Beijing, China) was used as a solvent and pore forming agent in the gel-casting process. A premix solution of monomers and cross linkers was prepared in TBA with a concentration of 14.5 wt% of acrylamide (AM, $\text{C}_2\text{H}_3\text{CONH}_2$) and 0.5 wt% N,N'-methylenebisacrylamide (MBAM, $(\text{C}_2\text{H}_3\text{CONH})_2\text{CH}_2$). The initiator and catalyst were ammonium persulfate (APS) and N,N,N,N-tetramethylethylenediamine (TEMED), respectively. All the chemicals used in this work are analytical reagent grade.

2.2. Preparation

All samples were prepared by the TBA-based gel-casting method. The fabrication process included preparing slurry, ball milling, deairing, molding, gelation, drying and sintering. YSZ powder and 0–15 vol% fiber were weighed in proportion. The slurries were prepared with 15 vol% solid content, 85 vol% premix solution by ball milling with a rotary speed of 300 r/min for 0.5 h. After ball milling and deairing, initiator (ammonium persulfate (APS)) and catalyst (N,N,N,N-tetramethylethylenediamine (TEMED)) were added into the slurries. The slurries were poured into molds and dried in air at 50 °C. During the drying procedure, the polymerization of AM occurred as well as TBA gradually volatilized. After demolding, the green bodies were calcined at 500 °C for 2 h to remove the organics, followed by pressureless sintering in air at 1400–1550 °C for 0.5–4 h.

2.3. Characterization

Microstructure was observed using a scanning electron microscope (SEM, JSM 6700F, JEOL, Tokyo, Japan). Porosity was measured by calculating the difference between the theoretical density (the density of dense YSZ material is about 6.0 g/cm³ and the density of Al_2O_3 /YSZ composites were calculated with the rule of mixture) and the actual density. Compressive strength was measured on cylinder samples (20 mm in diameter and 20 mm in height) and loaded with a crosshead speed of 0.5 mm/min using a CSS-2220 test machine (kexin Co., Ltd., Changchun province, China). Flexural strength was measured by three-point bending test (test bars 4 mm × 3 mm × 36 mm) with a span of 30 mm and a cross-head speed of 0.5 mm/min. Fracture toughness was measured by the SENB method (test bars 4 mm × 6 mm × 30 mm) with a cross-head speed of 0.05 mm/min and the width of the notch is less than 0.25 mm. Four samples were used to determine the average porosity and mechanical properties.

3. Results and discussion

3.1. The influence of the sintering conditions

In order to optimize the sintering conditions, all samples were prepared with 10 vol% Al_2O_3 fiber content. Fig. 1 shows the effects of sintering conditions on porosity and compressive strength of YSZ ceramics with 10 vol% Al_2O_3 fiber. When the sintering temperatures increased from 1400 to 1550 °C, as shown in Fig. 1a, the porosity decreased gradually while the compressive strength increased. A remarkable increase of compressive strength was observed when the sintering temperature changed from 1450 to 1500 °C. This indicated that the YSZ green body underwent well sintering at 1500 °C, which led to stronger grain boundary among YSZ grains and stronger interfacial bonding between YSZ grains and Al_2O_3 fibers. As a result of strengthened structure skeleton, the compressive strength of the porous YSZ ceramics was improved while the porosity decreased. The higher sintering temperature of 1550 °C promoted shrinking, while the compressive strength increased slowly.

Fig. 1b shows the soaking time dependencies of porosity and compressive strength of YSZ ceramics. Porosity decreased while the compressive strength increased with increasing soaking time. With the soaking time changing from 2 h to 4 h, the compressive strength increased slowly, while the porosity monotonically decreased.

Fig. 2 shows the microstructure of samples sintered at different temperatures and the insert images on top-right corners show the grain-connections at higher magnification. As seen in Fig. 2a and b, the YSZ particles are poorly connected at 1400 and 1450 °C. However, YSZ particles start forming sintering necks at 1500 °C (Fig. 2c). At 1550 °C, the sintering neck grew and form block structure (Fig. 2d). The Al_2O_3 fibers remained their smooth surface at temperatures below 1500 °C. The fiber and the particles did not conjoin tightly below 1500 °C, and the gap between the fiber and

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