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Effect of sintering temperature on compressive strength of porous yttria-stabilized zirconia ceramics

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Abstract

Porous yttria-stabilized zirconia (YSZ) ceramics were fabricated by tert-butyl alcohol (TBA)-based gel-casting method for potential applications in heat-insulation materials. The effect of sintering temperature on compressive strength of porous YSZ ceramics was investigated on the basis of measurements linear shrinkage, porosity and pore size. As the sintering temperature increased from 1350 to 1550 °C, a decrease of porosity from 77 to 65%, a decrease of average pore size from and an increase of linear shrinkage from 15.4 to 31.8% were observed. The compressive strength increased remarkably from 3 to 27 MPa with increasing sintering temperature from 1350 to 1550 °C, which was related to the corresponding change of linear shrinkage, porosity, pore size and microstructure. A remarkable decrease of compressive strength with increasing porosity was observed. The compressive strength decreased also with increasing pore size.

Keywords: B. Porosity; C. Strength; D. ZrO_2; Gel-casting

1. Introduction

Zirconia-based ceramics have high strength, low thermal conductivity compared with other ceramics. It is therefore of interest to consider them as thermal protections. Many studies of zirconia-based ceramics focused on fabricating them as thermal barrier coatings (TBCs). Although TBCs offer advantage in thermal insulating, they provided little potential in load-bearing. TBCs cannot meet the strength requirement of thermal protections which require not only low thermal conductivity but also high strength. Therefore, it is important to study zirconia-based ceramics as bulk materials which meet both thermal and mechanical requirements.

It has been confirmed that thermal conductivity of zirconia-based ceramics is lowered by introducing pores to form porous ceramics [1]. Meanwhile, the strength of porous ceramics decreases significantly with increasing porosity. The decrement of strength relates directly to the microstructure of porous ceramics which is influenced by fabrication methods. Many studies reported on the methods of fabricating highly porous ceramics, including employing pore forming agents [2–10], partial sintering [11] and template methods [12–14]. However, fabrication of porous ceramics with both high porosity and high strength were rarely reported. Gel-casting process is a recently developed method for fabricating porous ceramics with uniformly distributed pores which accounts for relatively high strength [15].

In this paper, porous yttria-stabilized zirconia (YSZ) ceramics were successfully fabricated by tert-butyl alcohol (TBA)-based gel-casting process. The compressive strength was influenced by sintering temperature besides composition of the sample and initial solid loading. The effect of sintering temperature on compressive strength was investigated on the basis of measurements linear shrinkage, porosity and pore size.

2. Experimental procedure

2.1. Materials

Commercially available yttria-stabilized zirconia (ZrO_2-8 mol% Y_2O_3, YSZ) powder (AR grade, Shanghai Chemical Regent Co., China) was used as the starting material. This YSZ powder has an average particle size of 1.26 μm and a specific

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surface area of 6.49 m^2/g. TBA (chemical purity, Beijing Yili Chemical Corporation, Beijing, China) was used as the shaping solvent and the pore forming agent in gel-casting process. A premix solution of monomers (acrylamide, AM, $C_2H_3CONH_2$) and cross-linker (N,N′-methylenebisacrylamide, MBAM, $(C_2H_3CONH)_2CH_2$) was prepared in TBA with a concentration of 14.5 wt% AM and 0.5 wt% MBAM. Initiator and catalyst for gelation reaction were ammonium persulfate (APS) and N,N,N′,N′-tetramethylethylenediamine (TEMED), respectively.

2.2. Fabrication procedure

The TBA-based gel-casting technique was applied to fabricate bulk, porous, yttria-stabilized zirconia (YSZ) ceramics. This method typically consists of preparing a liquid suspension (slurry) and then molding, drying, removing the binder and sintering. Slurries, including YSZ powders, TBA and acrylamide (AM), were prepared by ball milling for 5 h. To adjust the suspension to a proper flowability during casting, selected alkali solution was added into the slurries. After ball milling, initiator and catalyst were mixed into the slurries. These slurries were poured into glass molds and dried in a nitrogen atmosphere. During the drying procedure, TBA gradually volatilized, and the AM polymerized to form a strong network. Green bodies were then produced after demolding. Subsequently, they were sintered at different temperatures of 1350, 1400, 1450, 1500 and 1550 °C for 2 h.

2.3. Characterization

The linear shrinkage of samples during sintering process was determined by the following equation:

$$\text{shrinkage} = \left(\frac{l_g - l_s}{l_g}\right) \times 100\% \quad (1)$$

where l_g and l_s are the height of green sample and sintered sample, respectively. The bulk density of the sintered sample was measured from the sample mass and dimension. The relative density was obtained from the ratio of the measured bulk density to the theoretical density, which was taken as 6.0 g/cm^3 for this YSZ material. The open porosities of the as-prepared YSZ ceramics were measured by Archimedes method in distilled water. Pore size distribution was analyzed by a mercury intrusion porosimetry (AutoPore IV 9510). Microstructure was observed with a scanning electron microscope (SEM, JSM 6700F, JEOL, Tokyo, Japan). The compressive strength was measured by using a CSS-2220 testing machine with cylindrical specimens with a diameter of 20 mm and a height of 20 mm. Three specimens were used to determine the average compressive strength.

3. Results and discussion

3.1. Linear shrinkage

Fig. 1 shows the variation of linear shrinkage of the porous YSZ ceramics with sintering temperature. The linear shrinkage

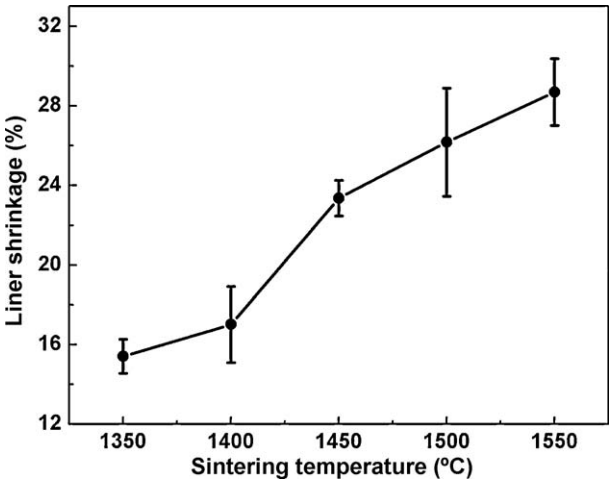

Fig. 1. Variation of linear shrinkage of porous YSZ ceramics with sintering temperature.

of the samples increased from 15.4 to 31.8% as the sintering temperature increased from 1350 to 1550 °C. The change of linear shrinkage between the 1400 °C sample and the 1450 °C sample was the most substantial. The authors believe that the shrinkage occurred as the TBA and polymer were removed and the grains became stronger interconnected to each other. The increase of linear shrinkage with increasing temperature was attributed to increasingly intensive interconnection between grains as shown in Fig. 2 below.

3.2. Microstructure

Fig. 2 shows the SEM micrographs of the porous YSZ specimens sintered at different sintering temperature of 1350, 1400, 1450, 1500 and 1550 °C. After the sintering processes, both TBA and PMA were successfully removed and porous YSZ bodies without processing cracks were obtained. The interconnected pores were homogeneously dispersed in the zirconia matrix. Adjacent grains interconnected to form a strong skeleton. It could be observed from Fig. 2a–e that the grains grew up and the porosity decreased as the sintering temperature increased from 1350 to 1550 °C. This porous structure was a consequence of the pore formation during the experimental process: TBA volatilized at a high temperature, and polymer was burned out when the temperature reached approximately 600 °C. Both of the two components then travelled along numerous irregular channels from inside the ceramic to the surface. As a result, the space left by TBA and polymer formed the pores.

3.3. Porosity

Fig. 3 shows the variations of porosity and open porosity of porous YSZ ceramics with sintering temperature. As the sintering temperature increased from 1350 to 1550 °C, the porosity decreased from 77 to 65% due to the process of sintering densification. Open porosity exhibited a general decrease trend with increasing sintering temperature except for

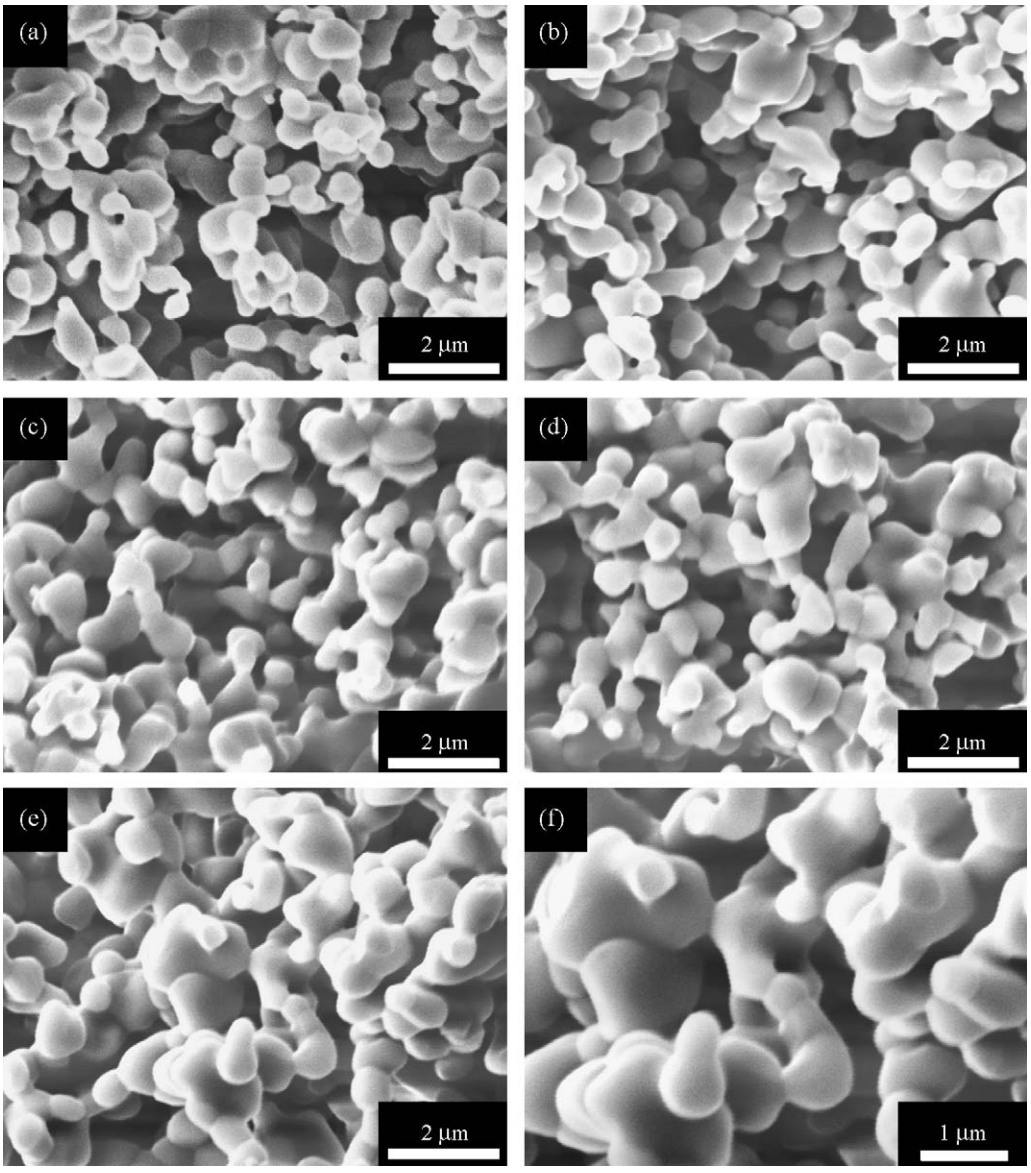

Fig. 2. SEM micrographs of porous YSZ specimens at different sintering temperatures: (a) 1350 °C, (b) 1400 °C, (c) 1450 °C, (d) 1500 °C, (e) 1550 °C and (f) typical interconnection of grains.

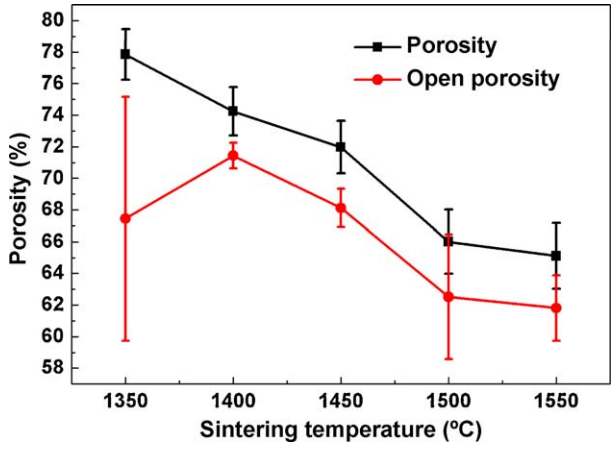

Fig. 3. Variations of porosity and open porosity of porous YSZ ceramics with sintering temperature.

the unexpected low value of samples sintering at 1350 °C. The authors believe that the unexpected low value of samples sintered at 1350 °C resulted from the measuring error: part of sample was lost into the boiling water due to the low strength of samples sintered at 1350 °C, leading to declined values of water mass and volume in the sample which represented the open pore volume. After 1400 °C, porosity and open porosity revealed a similar decreasing trend.

3.4. Pore size distribution

Fig. 4 shows the pore size distribution of the porous YSZ ceramics sintered at 1350, 1400, 1450, 1500 and 1550 °C, respectively. At about the 1 μm of pore size, the sharp increase from 0 to a stable value of cumulative volume indicated monomodal and uniform pore size distribution. It was noticed that the mean pore size generally decreased with increasing

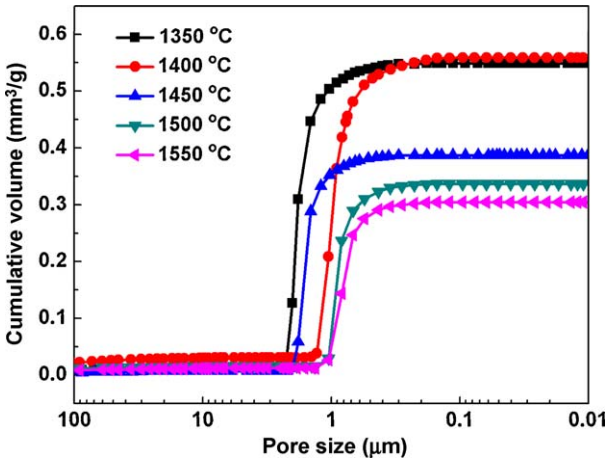

Fig. 4. Pore size distribution of the porous YSZ ceramics at different sintering temperatures.

sintering temperature from 1350 to 1550 °C. The total cumulative volume generally decreased with increasing temperature except for the unexpected low value of the sample sintering at 1350 °C, indicating that the pore volume decreased as the temperature increased, which was consistent with the open porosity–temperature dependence shown in Fig. 3. The unexpected low value of the sample sintering at 1350 °C was believed to result from the similar measuring error which is stated above.

3.5. Compressive strength

Fig. 5a shows the variation of compressive strength of porous YSZ ceramics with sintering temperature. The compressive strength increased from 3 to 27 MPa with the increase of sintering temperature from 1350 to 1550 °C. A remarkable increase of compressive strength was observed from 1500 to 1550 °C. Due to the high porosity which led to an exponential decrease of strength, the level of strength was much lower than the corresponding value (∼2068 MPa [16]) of dense zirconia ceramics. The effect of sintering temperature on compressive strength of porous YSZ ceramics can be investigated by linear shrinkage, porosity and pore size, which were discussed in detail as below.

Fig. 5b shows the variation of compressive strength of the porous YSZ ceramics with linear shrinkage. The compressive strength increased from 3 to 27 MPa with increasing linear shrinkage from 15.4% to 28.7%. The strength–shrinkage behavior was observed to be similar to the strength–sintering temperature behavior. With the increase of sintering temperature, ceramic particles connected closer and the linear shrinkage became larger. Consequently, the structure of the

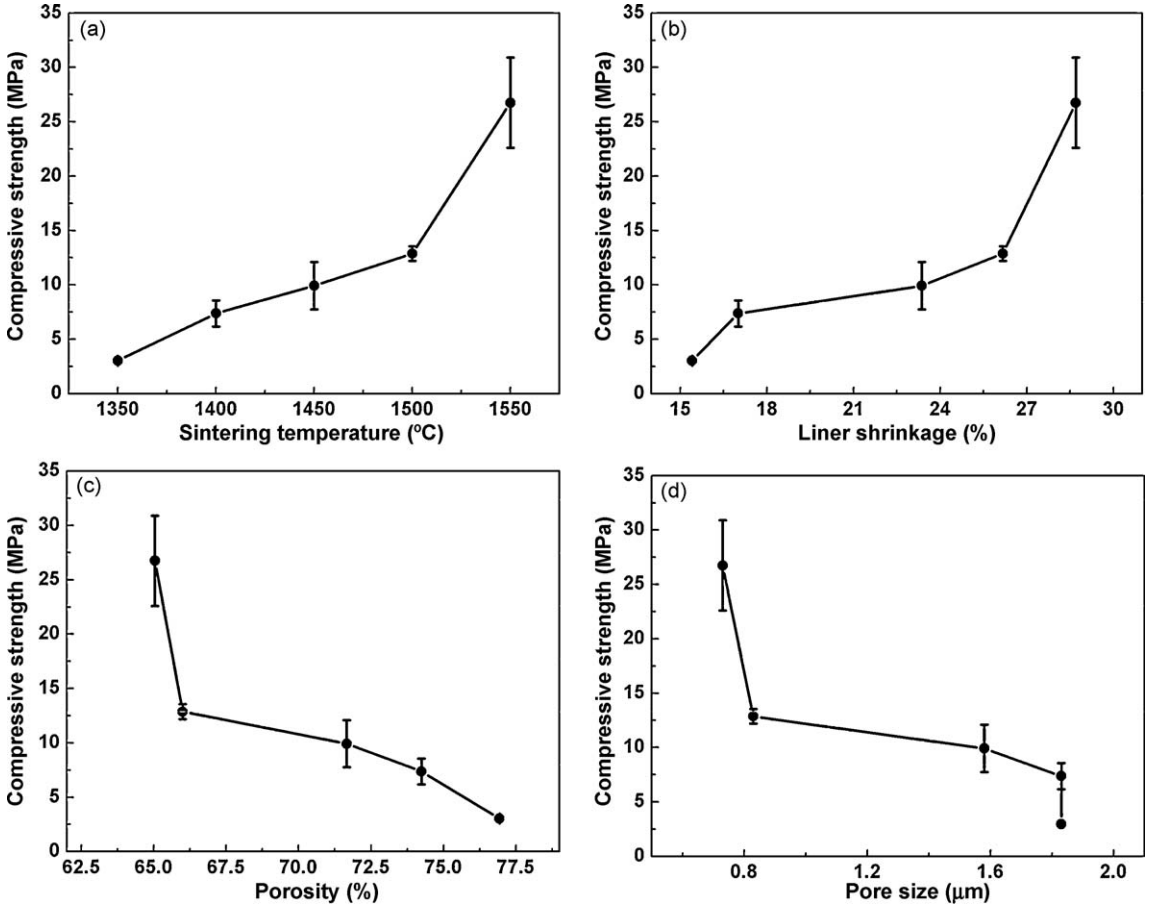

Fig. 5. Variation of compressive strength of porous YSZ ceramics with (a) sintering temperature, (b) linear shrinkage, (c) porosity and (d) pore size.

sintered compacts became stronger, which led to higher strength of the final products.

The compressive strength of porous YSZ ceramics also depends on the porosity. Fig. 5c shows the variation of compressive strength of the porous YSZ ceramics with porosity. The compressive strength of the porous YSZ ceramics with porosity of 65% was 27 MPa. As the porosity increased, the compressive strength decreased remarkably. In the porous YSZ ceramics which contained 77% pores, the compressive strength was about 3 MPa. The compressive strength appeared to be insensitive to the total pore volume at relatively high level of porosity, e.g. >70%; however, the difference in the compressive strength–porosity behavior became pronounced as the porosity decreased. When the porosity was 77%, the pores in the sintered sample were big, and the grains were loosely bonded, so the strength was relatively low. With the decrease of the porosity, the pore became smaller, and the grains interconnected tightly to form a strong skeleton, resulting in relatively high strength.

The compressive strength of the porous YSZ ceramics depends not only on the linear shrinkage and total pore volume but also on the dimensions of pores. Fig. 5d shows the variation of compressive strength of porous YSZ ceramics with pore size. The values of pore size denoted the average pore size analyzed by a mercury intrusion method. It was noticed that the compressive strength of the porous YSZ ceramics decreased from 27 to 3 MPa with increasing pore size from 0.73 to 1.82 μm.

4. Conclusions

Porous YSZ ceramics were prepared using TBA-based gel-casting method. The sintering temperature of porous YSZ ceramics influenced significantly the linear shrinkage, porosity and pore size, and thus exerted impact on compressive strength. Therefore, it is possible to adjust sintering temperature to control pore size and thus compressive strength of porous YSZ ceramics for potential applications in thermal insulations.

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