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Fabrication and properties of porous anorthite ceramics with modelling pore structure

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ABSTRACT

A single-phased anorthite porous ceramics was fabricated by foam-gelcasting technique using α-Al_2O_3, $CaCO_3$ and SiO_2 as starting material. Through adjusting foaming agent concentration and solid content in the slurry, tailored porosity (62–91%), pore size (7–350 μm), thermal conductivity (0.04–0.27 W/m·K) and compressive strength (0.27–13.39 MPa) were obtained. The experimental thermal conductivity was in good agreement with the universal model. The dependence of compressive strength on porosity can be described by the Gibson model.

1. Introduction

Anorthite has many good properties, such as low theoretical density (about 2.76 g/cm^3), low thermal conductivity (about 3.67 W/m·K) and low thermal expansion coefficient (4.8×10^{-6}/K), making it a very promising material for thermal insulating applications [1–4]. Although anorthite has already been used as one of the main component in many commercialized refractory bricks, the relatively low purity and poor mechanical properties of these bricks limit their applications. Therefore, preparation of porous anorthite ceramics with high-purity, high strength and low thermal conductivity has become an area of focused research.

In past years, several techniques have been developed to prepare porous anorthite ceramics, including direct foaming [1], addition of foaming agent [4], organics burned [5], and foam-gelcasting [6]. Among those, foam-gelcasting leads to porous ceramics with good properties. Through this method, porous anorthite ceramics with high purity and ultra-low thermal conductivity (lowest to 0.018 W/m·K in vacuum) has been fabricated using γ-Al_2O_3, $CaCO_3$ and SiO_2 powders as raw materials [6]. But the particle properties of γ-alumina make it impossible to prepare porous anorthite ceramics with low porosity (less than 69.9%) and high strength.

Thus, in this paper, α-Al_2O_3 powder was selected as alumina resource to make porous anorthite ceramics by foam-gelcasting technique. Phase composition, pore structure and properties were thoroughly investigated. Thermal conductivity and compressive strength were compared with theoretical models, and discussed in terms of microstructure.

2. Experimental procedure

For anorthite synthesis, commercial powders α-Al_2O_3 (99.99%, Beijing boyu gaoke new material technology Co. Ltd., China), $CaCO_3$ (99.5%, Aladdin, China) and SiO_2 (99.4%, Shanghai fengchen powder material Co. Ltd., China) with particle size of about 0.3, 0.8 and 10 μm are used as raw materials. Materials used for aqueous foam-gelcasting processing is same as that used in reference [6] and [7].

The foam-gelcasting procedure used herein was performed as follows, which is similar to that described in reference [6] and [7]. First of all, ceramics powders in stoichiometric ratio of anorthite and premixed slurry were ball-milled for about 20 h to prepare even ceramics slurry. Secondly, Foaming agent was added into the slurry followed by rapidly mechanical stirring to form foamed slurries. Thirdly, the catalyst and initiator (the amount of catalyst and initiator were adjusted to ensure the solidification time was within 30 min.) were slowly added into the foamed slurries and then mixed rapidly. After adequately mixed, foamed

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Table 1
Processing and properties of porous anorthite ceramics made by foam-gelcasting.

Sample	Solid content (vol.%)	Foaming agent concentration (g/L)	Porosity (%)	Pore size (μm)	Compressive strength (MPa)	Thermal conductivity (W/m·K)
1	35	0.25	62.16 ± 0.69	7.77	13.39 ± 2.451	0.272 ± 0.0197
2		0.5	71.39 ± 1.18	14.91	4.98 ± 1.054	0.192 ± 0.0064
3		1	78.12 ± 0.05	21.62	3.13 ± 1.110	0.137 ± 0.0029
4		2	85.09 ± 0.40	68.69	1.03 ± 0.099	0.065 ± 0.0093
5		4	85.42 ± 0.50	63.23	1.50 ± 0.035	0.062 ± 0.0019
6		8	85.49 ± 0.20	59.71	2.96 ± 0.290	0.060 ± 0.0043
7	20	1	90.70 ± 1.85	82.10	0.27 ± 0.087	0.040 ± 0.0029
8	25		85.70 ± 0.19	69.67	1.43 ± 0.080	0.059 ± 0.0019
9	30		83.45 ± 0.42	59.70	1.65 ± 0.630	0.086 ± 0.0073
10	35		78.12 ± 0.05	21.62	3.13 ± 1.110	0.137 ± 0.0029
11	40		77.50 ± 0.11	23.52	5.28 ± 1.483	0.156 ± 0.0069

slurries were poured into molds and solidified at room temperature for 30 min. Then green bodies were removed from mold and dried in a microwave oven. Finally, the dried green bodies were sintered at 1400 °C for 5 h. To prepare porous anorthite ceramics with varied pore structure, the solid content and foaming agent concentration were tailored, as shown in Table 1.

Phase composition was identified by XRD (D/MAX-IIIB, Rigaku, Japan) and microstructure was observed using SEM (JSM 6700F, JEOL, Tokyo, Japan). Archimedes method in distilled water was used to test porosity. Pore size distribution was analyzed using mercury intrusion method (Auto Pore IV 9510, Micrometrics, USA). Room temperature thermal conductivity was measured on $5 \times 5 \times 3\ mm^3$ machined specimens using Thermal Transport Option (TTO) parts of Physical Properties Measurement System (PPMS, Model 6000, Quantum Design, USA) in high vacuum environment. Compressive strength was measured with cylindrical specimens of 20 mm diameter and of 20 mm height by a mechanical testing machine (WDW3020, Changchun, China) with a crosshead speed of 0.5 mm/min. At least three specimens were measured for each material.

3. Results and discussion

Fig. 1 shows typical XRD patterns of the prepared materials. It is seen that all diffraction peaks can be identified as anorthite, and no other phase is detected. XRD patterns for other samples are almost the same, which are not shown here. This result indicates that a single-phased porous anorthite ceramics can be prepared by this technique.

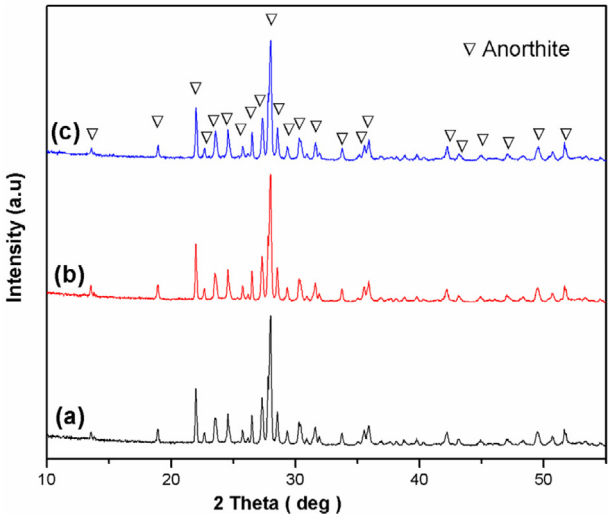

Fig. 1. Typical XRD patterns obtained from three different ceramics: sample 1, (b) sample 3, (c) sample 6.

SEM images and pore size distribution curves (Fig. 2) show the pore characteristics of the ceramics fabricated using different foaming agent concentrations (Table 1 samples 1–6). SEM images show that materials mainly contain two types of pores: large spherical pores generated by foaming process and small cellular holes within the internal walls derived from the removal of organic matter. The images also reveal that pore size and porosity vary with foaming agent concentration. These results consist with the pore size distribution curves. As the foaming agent concentration increases from 0.25 to 2 g/L, the size of the large spherical pores and the porosity increases. However, upon further increasing it to 8 g/L, the size of the large spherical pores decreases and the porosity remains nearly the same. Observation (SEM image not show) of the ceramics prepared with different solid phase loads revealed that the size of the large pores decreases upon increasing the load from 20 to 35 vol%, whereas the size remains the same when the solid load further increases to 40 vol%. Effects of foaming agent concentration and solid content on pore structure can be understood by considering foaming mechanism. Surface energy and viscosity of the slurry are two main factors during foaming. Foaming agent is a kind of surfactant which can lower surface energy, and solid content may change the viscosity of the slurry. Low surface energy and viscosity is helpful for bubble to form and grow.

The thermal conductivity of porous anorthite ceramics was measured at room temperature in a vacuum (Table 1). The results demonstrate that the thermal conductivity can be tailored within a very large range by changing foaming agent concentration or solid content, suggesting that the technique employed herein is very useful for preparing thermal insulating porous ceramics. Further analysis of the thermal conductivity data was performed by comparing the experimental data to theoretical models (Fig. 3). From Fig. 3, the experimental data cannot be described by the five classic models (series, parallel, Maxwell-Eucken-I, Maxwell-Eucken-II, and Effective Medium Theory) but fit well with the universal model (Eq. (1)) when $d_i = 3$ and $k' = 0.26$. High porosity and complex pore structure are the main reason for low thermal conductivity.

$$K_e = \frac{\sum_{i=1}^{2} k_i v_i \frac{d_i k'}{(d_i - 1)k' + k_i}}{\sum_{i=1}^{2} v_i \frac{d_i k'}{(d_i - 1)k' + k_i}} \quad (1)$$

where k and v are thermal conductivity and volume fraction [8]. Here, thermal conductivity of high vacuum and dense anorthite ceramics are chosen as 1×10^{-9} and 3.67 W/m·K.