



# Preparation and characterization of foamed microporous mullite ceramics based on kyanite

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## Abstract

A new approach has been developed for preparing microporous mullite ceramics based on kyanite obtained by foaming with aluminate cement as the gel-former. By using this method, foams in slurries are effectively stabilized, microporous mullite ceramics with high open porosity (53–77.3%), high compressive strength (5.5–50.5 MPa), small mean pore size (2.5–9.1  $\mu\text{m}$ ) and low thermal conductivity (0.37–0.45 W/mK at 1100 °C) are prepared. Effects of foaming agent addition on the microstructures and properties of samples are investigated. With increasing foaming agent addition, the number and size of pores increase firstly and then tend to be constant. The pore structure looks like a bird's nest, in which there are a lot of needle-like mullites, which results in the effective pore size smaller.

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## 1. Introduction

Due to their excellent chemical stability, high permeability, high refractoriness, low bulk density and thermal conductivity as well as specific heat [1–4], porous ceramics have attracted much attention in the scientific community in recent years, which have been used in a wide range of engineering applications, such as refractory linings for furnaces [5,6], filters for molten metal and hot gas [7,8], catalyst carriers [9,10], or thermal and acoustic insulation applications [7].

During the past decades, variety of manufacturing methods have been developed for fabricating porous ceramics, including the replica, the sacrificial template and the direct foaming methods [9,11,12]. Among them, the direct foaming method is recognized as the optimum method for the fabrication of porous ceramics with higher porosity and smaller-sized closed pores. However, the foam is in a state of imbalance, several transformations in the bubble structures may occur between the

foam generation and the slurry solidification, which will influence the final microstructures and properties of solid foams [5,13]. Therefore, it is important to take effective measures to stabilize the foams. In recent years, it has been proposed that the use of gelcasting technique is a promising route for fabricating porous ceramics, which can stabilize the foams through the in situ polymerization of organic monomers [14]. However, industry has been reluctant to use this technology, not only considering it is high cost, but also due to that the acrylamide monomer has neurotoxin [15]. In this study, the aluminate cement is first used as the gel-former and binder to stabilize the foams for fabricating microporous mullite ceramics, it is an eco-friendly and cost-effective method. When foams is produced either by mechanical frothing or by the injection of gas into ceramic suspensions, the cement and other particles will deposit on the bubble film surface, then the hydration reaction of the gelatinization materials takes place, the new generating hydration products will fill into the blanks among water molecules, which can stabilize the foams by fixing other particles. The bubbles

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finally transform into pores among solid particles in the porous ceramics through caking and heating processes.

In addition, kyanite belongs to  $\text{Al}_2\text{O}_3\text{-SiO}_2$  system material, the irreversible reaction between  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  in kyanite forms mullite at high temperature, which accompanies ca 16–18% volume expansion. This property of kyanite makes it widely used as an expanding agent to counteract the shrinkage of samples during the heating process in the refractory field. However, there are few reports on porous mullite ceramics fabricated by using kyanite as the main starting material for the applications in high-temperature thermal insulations or furnace linings.

In this paper, microporous mullite ceramics are fabricated by the direct foaming method with kyanite as the main starting material, aluminate cement as the stabilizer of foams. The effects of foaming agent addition on the microstructures and some properties (including bulk density, open porosity, mechanical strength, pore size distributions and thermal conductivity) of microporous mullite ceramics are investigated.

## 2. Experimental procedure

### 2.1. Experimental materials

The starting material used to prepare microporous mullite ceramics is commercial kyanite powder with particle size  $d_{50}$  of 75  $\mu\text{m}$  and  $\text{Al}_2\text{O}_3$  content  $50 \pm 1$  wt% (China Kyanite Company). Sodium lauryl sulfate (SLS, chemical purity, Tianjin Chemical Reagent Co., China) is selected as foaming agent. Aluminate cement (Secar 71, Lafarge, France) is used as the gel-former to stabilize foams and providing mechanical strength for the manipulation with raw foam before heating treatment.

### 2.2. Preparation of foamed slurries

Slurries were first prepared by well dry mixing powders, which are composed by the kyanite and aluminate cement with a weight ration of 95:5, then distilled water was added into the mixer (JJ-1, Jieruier Co., China) with solid content of 45 vol%. The density of kyanite powder was taken as 3.5  $\text{g}/\text{cm}^3$  in the solid content calculations. After 1 min of wet mixing, the homogeneous and good flowable slurries were obtained. In the early period of tests, the hydration of aluminate cement had just began, but its rate was very small. Then, different amounts (0, 0.1, 0.2, 0.3, 0.4 and 0.5 wt%) of the foaming agent were added into the slurries, after 5 min of vigorously whisking, the slurries became homogeneous foamed slurries.

### 2.3. Preparation of ceramic foams

After foaming, the foamed slurries were poured into molds, which were coated with silicon oil (Henan garlway machinery Co, China) before the casting process for the convenience of demolding and kept better surface qualities. Then drying in room temperature for 2–3 days, the samples gained sufficient strength. After demolding, the green bodies were dried under 50–120  $^\circ\text{C}$  for approximately 24 h, and then sintered in a

chamber furnace with a heating rate of 1.5 K/min until 600  $^\circ\text{C}$ , following by a ramp of 3 K/min up to 1450  $^\circ\text{C}$  and a dwell time of 180 min for sintering.

### 2.4. Characterization

Bulk density ( $\rho_b$ ) and open porosity ( $\epsilon_0$ ) of the sintered samples were determined by an Archimedes method. Total porosity ( $\epsilon_t$ ) of the sintered samples was evaluated using Eq. (1)

$$\epsilon_t = 1 - \rho_b / \rho_t \quad (1)$$

Here,  $\rho_t$  is true density, which was examined according to ISO5018-1983 standard.

Compressive strength was measured by a uniaxial compressive tester (HT-8391, Hongta Co, China) with a crosshead speed of 0.5 mm/min. Three to five samples were used to determine the average compressive strength. The phase composition was analyzed using Philips X-ray diffractometer (XRD) with Cu  $K_\alpha$  radiation (40 kV, 40 mA) in the  $2\theta$  range of 10–80  $^\circ\text{C}$  for a period of 3 $^\circ$ /min in the step scan mode. Polished sections of samples were prepared for microstructure analysis by scanning electron microscopy (SEM, JSM-5610LV, Japan) at an acceleration voltage of 15 kV. Pore size distribution was determined with mercury porosimetry (PM-60GT, Quantachrome instruments, America). Thermal conductivity at 1100  $^\circ\text{C}$  was measured by a flat plate thermoconductivity tester (PBDR-02P, Luoyang Precondar, China).

## 3. Results and discussion

### 3.1. Bulk density and open porosity

Bulk density and open porosity of microporous mullite samples with different foaming agent additions are shown in Fig. 1, showing that with the foaming agent addition increases, the bulk density initially decreases and then tends to be constant, while the change of open porosity exhibits a reverse tendency. When the foaming agent addition reaches to 0.4 wt%, the bulk density of samples shows the minimum value

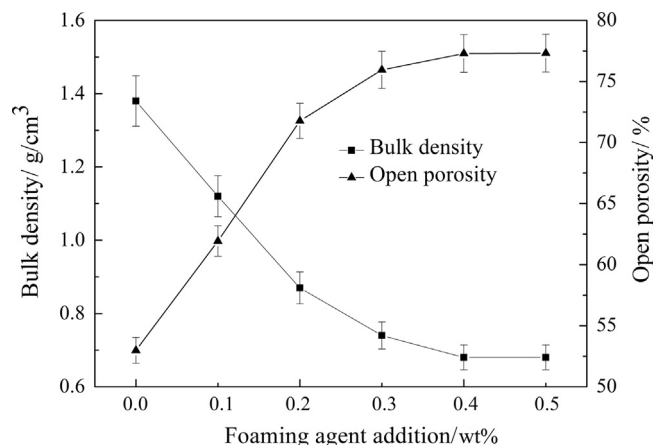


Fig. 1. Dependence of the bulk density and open porosity of microporous mullite samples on the foaming agent addition.

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