



Ultra-high porosity of open-cell alumina ceramic foams prepared by adding tiny amounts of γ -AlO(OH) sol

Youfei Zhang¹, Yongjiao Li¹, Zun Xia¹, Shihui Zhao², Xiuhui Wang¹, Hongbo Yu^{1,*}, Jinlong Yang^{1,2,*}

¹Key Lab of Preparation and Application of Inorganic Ultrafine Powder, Liaoning Provincial Department of Education, School of Materials Science and Engineering, Dalian Jiaotong University, Dalian 116028, China

²State Key Lab of New Ceramics and Fine Processing, School of Materials Science and Engineering, Tsinghua University, Beijing 100084, China

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Abstract

A creative and environmentally friendly preparation of high-porosity open-cell ceramic foams was investigated. The samples were prepared from the α -Al₂O₃ slurry with 25 wt.% solid loading in which a tiny amount of boehmite (γ -AlO(OH)) sol was added for the formation of particle-stabilized foams without introducing any sacrificial phases. The volumetric shrinking of γ -AlO(OH) enables the formation of open-cell while avoiding the generation of polluting gases. The resulting alumina ceramic foams have a synergistic increase in porosity and compressive strength, reaching a compressive strength of 1.17 MPa and an ultra-high porosity of 94.8%. This is due to the fact that the pore struts (connecting neighbouring cells) are almost defect-free, while the high sintering activity of γ -AlO(OH) promotes the sintering of ceramic phase and formation of alumina with small grains which has the strengthening effect on the obtained structures. By achieving open-cell, the heat conduction path increased, which reduces the thermal conductivity of the ceramic foam. This work provides an alternative and environmentally friendly new strategy for open-cell ceramics processing.

Keywords: ceramic foams, high porosity, open-cell alumina structure, γ -AlO(OH) additive

I. Introduction

Open-cell ceramic foams are widely used in catalysis, filtration, energy storage and other fields due to their unique three-dimensional interconnected structure [1]. It has been a hot topic of current research [2–4]. Light weight is one of the requirements for a growing number of applied engineering materials, and for ceramic foams this should be an advantage in itself due to their high porosity [5,6]. Unfortunately, open-cell ceramic foams with a porosity of more than 90% have been rarely reported.

Preparation methods for open-cell ceramic foams mainly include sacrificial fugitives, replica templates, polyurethane foaming method, etc. and the open-cell mechanism of the above methods is essentially achieved

by introducing and pyrolyzing sacrificial phases to form pores [7–9]. These sacrificial phases include organic sacrificial phases (polyurethane, starch, proteins, etc.) as well as inorganic sacrificial phases (carbon powders) [10–12]. The pyrolysis of the sacrificial phases produces a large number of gaseous pollutants including CO, CO₂, NO, NO₂, S₂O₃, SO₂, etc. which aggravates environmental pollution and bring harm to humans and nature [13]. At the same time, the sacrificial phases discharging process consumes more energy and the resulting corrosive gases can damage the heat treatment equipment, severely reducing the lifetime of the device. The introduction of the sacrificial phase also generally leads to a reduction in the mechanical properties of the ceramic product, and after pyrolysis of the sacrificial phase obvious defects inevitably occur in the ceramic matrix. When these defects are present in the pore column, they can severely affect its ability to withstand a load, thus reducing the mechanical properties of the foams [14]. Therefore, there is an urgent need for an en-

* Corresponding author: tel: +86 41184109266

e-mail: yhb961@163.com (H. Yu),

jlyang@mail.tsinghua.edu.cn (J. Yang)

environmentally friendly method to prepare open-cell ceramic foams that can satisfy conditions of high porosity and suitable mechanical properties.

To this end, a new open-cell mechanism must first be developed, i.e. where the opening of the pores can be achieved without introducing any sacrificial phases. Deng *et al.* [15] reported that when using aluminium hydroxide mixed with α -alumina prepared by cold-pressing, the 60% volume shrinkage that occurs when aluminium hydroxide is converted to α -alumina increases the porosity of ceramic samples. This is clearly a new porosity opening mechanism, but it has rarely been reported in the preparation of ultra-high porosity ceramic foams. Boehmite (γ -AlO(OH)) and aluminium hydroxide both belong to the class of hydrated alumina and can also achieve open-cell by volume shrinkage [16,17]. However, boehmite has superior properties, including grains of only a few nanometres in size, high specific surface area and sintering activity. As a result, boehmite is able to achieve open-cell while promoting sintering and thus increasing strength without generating contaminants.

In this study, we utilized α -Al₂O₃ as a raw material and added a small amount of γ -AlO(OH) to fabricate open-cell alumina foams via particle-stabilized foaming. The relevant properties of ceramic foams were characterized, mainly including microstructure, porosity and mechanical properties and thermal conductivity. The analysis focuses on the mechanism of compressive strength enhancement, supporting the application of open-cell ceramic foams in industrial areas.

II. Experimental

2.1. Preparation of ceramic foams

α -Al₂O₃ particles (CT3000 SG, 99.8%, Almatix Co. Ltd.) were mixed with deionized water by ball grinding to obtain the slurry with a 25 wt.% solid loading. Then, long-chain surfactant sodium alpha-olefin

sulfonate (AOS in short; chemically pure; Shandong Yousuo Chemical Technology Co. Ltd, Linyi, China), used as the hydrophobic modifier, was dropwise added at a lower stirring speed of around 500 rpm, at concentrations of 0.03–0.05 wt.% based on the total mass of the suspension [18]. Then γ -AlO(OH) (Hangzhou Zhihuajie Technology Co. Ltd, Hangzhou, China) was added in a concentration of 2, 4 and 6 wt.% in relation to α -Al₂O₃. Once well mixed, the stirring speed increased gradually to 2000 rpm and held for 10 min to prepare stable wet foams. Afterwards, 2 mol/l ammonia solution (Sinopharm Chemical Reagent, Shanghai, China) was added to adjust the pH value to 6.0–8.0, and the agitation remained for another 5 min to stabilize the foams. The stable foams were then poured into the moulds (stainless steel, 50 mm × 68 mm × 68 mm). After setting for 5–10 min, the wet foams could hold their shape, followed by demoulding. The wet foams were allowed to dry at room temperature for 24–48 h. The dried foams were sintered in air at a temperature of 1500 °C, with a heating rate of 5 °C/min and a dwelling time of 2 h. The preparation procedure for open-cell alumina ceramic foams is depicted in Fig. 1.

2.2. Characterization

Morphology of specimens was observed using a scanning electron microscope (SEM; Merlin VP Compact, Carl Zeiss, Jena, Germany). The appropriate amount of the boehmite particles was dispersed in ethanol, sonicated for 20 min, and then deposited onto a copper electron microscope grid to observe their microstructure by transmission electron microscopy (TEM, S-5500, Hitachi, Japan). Phase composition of the ceramic foams was characterized by X-ray diffraction using Cu K α radiation (XRD, D8 ADVANCE, Bruker, Karlsruhe, Germany).

Compressive strength of the alumina ceramic foams was measured using a universal material testing machine (AG-IC 5 kN; Shimadzu Corporation, Kyoto,

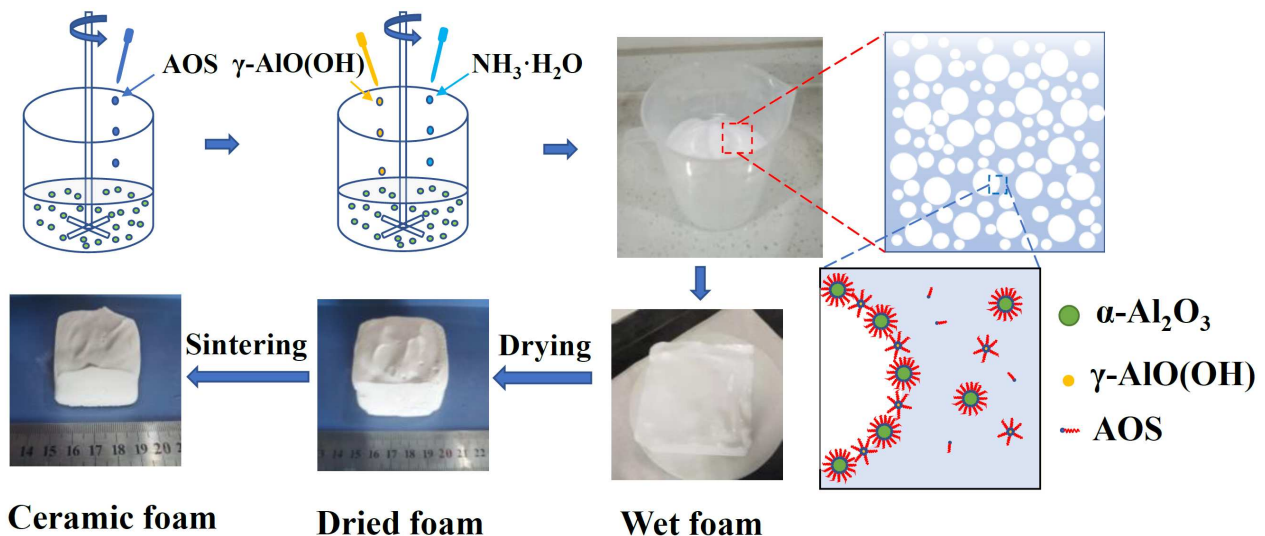


Figure 1. Schematic diagram of the procedure for alumina ceramic foams preparation

Japan) at a load of 1 mm/min. Three samples with dimensions of approximately 10 mm × 20 mm × 20 mm were tested, and the mean and standard deviation of compressive strength were determined. The thermal conductivity of the ceramic foams was characterized by a thermal constant analyser (TPS 3500, Hot disk, Sweden) at room temperature. The pore size distribution of the ceramic foams was determined by Nano Measurer software (invented by Fudan University, Shanghai, China), where more than 200 pore diameters were collected and analysed. The open porosity (P) and bulk density (D) of ceramic foams were measured by the Archimedes principle using intelligent ceramic porosity (MAY-120SD; MiaoZhun Technology Co. Ltd, Guangdong, China) according to following equations:

$$P = \frac{m_1 - m_2}{m_1 - m_3} \cdot 100 \quad (1)$$

$$D = \frac{m_2}{m_1 \rho_{H_2O}} \cdot \rho_{H_2O} \quad (2)$$

Herein, m_1 , m_2 and m_3 represent wet weight, dried weight and weight of samples when suspended in water, respectively. The linear shrinkage during the sintering process (S) was calculated by Eq. 3:

$$S = \frac{l_d - l_s}{l_d} \cdot 100 \quad (3)$$

where l_d and l_s are the side length of dried foam and sintered ceramic foam.

III. Results and discussion

3.1. Microstructure and sintering of ceramic foams

SEM micrographs of the raw powders (Fig. 2) show that the α -alumina has particles with the size of about few hundred nanometres, while particles of the γ -AlO(OH) are much smaller, only a few nanometres.

XRD analyses of the ceramic foams are shown in Fig. 3. Both α -Al₂O₃ and γ -AlO(OH) phases co-existed in the dried foams and all of them were converted into α -Al₂O₃ after sintering at 1500 °C. This provides sup-

port for the preparation of single component alumina ceramic foams.

Figure 4 shows SEM micrographs of the prepared ceramic foams. In the ceramic foams without γ -AlO(OH) the pore morphology definitely belongs to the closed-cell structure, but after the addition of γ -AlO(OH) open pore structure was obviously formed. The pore structures consist of the cells referring to the larger pore in the foam (Fig. 4e), struts acting as a support for the pore structure (where the walls of the cell meet, as shown in Fig. 4f) and windows, i.e. the pores that appear in the wall of the cells (as shown in Fig. 4g).

Figure 5 shows the sizes of the cell and window at different amounts of γ -AlO(OH), which gradually decreased from $102.6 \pm 1.6 \mu\text{m}$ in the sample without the addition of γ -AlO(OH) to $97.8 \pm 0.7 \mu\text{m}$ in the foam with 6 wt.% γ -AlO(OH). With the increase of the γ -AlO(OH) amount from 2 to 6 wt.%, the size of window gradually increased from $14.8 \pm 0.9 \mu\text{m}$ to $37.2 \pm 3.7 \mu\text{m}$. This demonstrates the ability of γ -AlO(OH) to participate in the formation of open-cell structure.

The open-cell structure is achieved by the volume self-shrinkage that occurs when γ -AlO(OH) is trans-

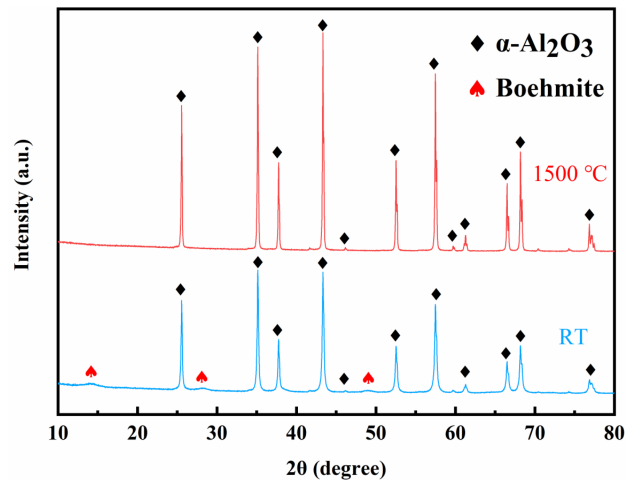


Figure 3. XRD patterns of the foams dried at room temperature (RT) and sintered at 1500 °C (precursor suspension contained 4 wt.% γ -AlO(OH))

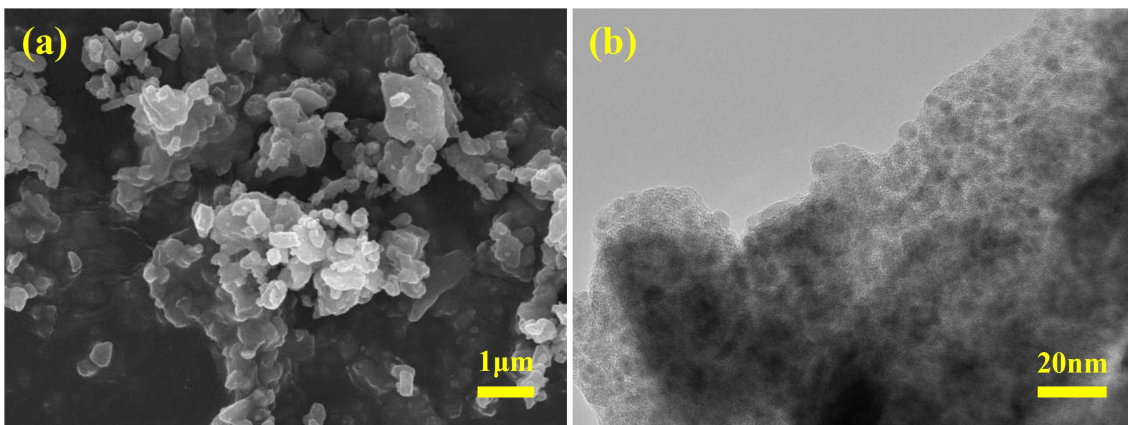


Figure 2. SEM image of α -Al₂O₃ particles (a) and TEM image of boehmite particles (b)

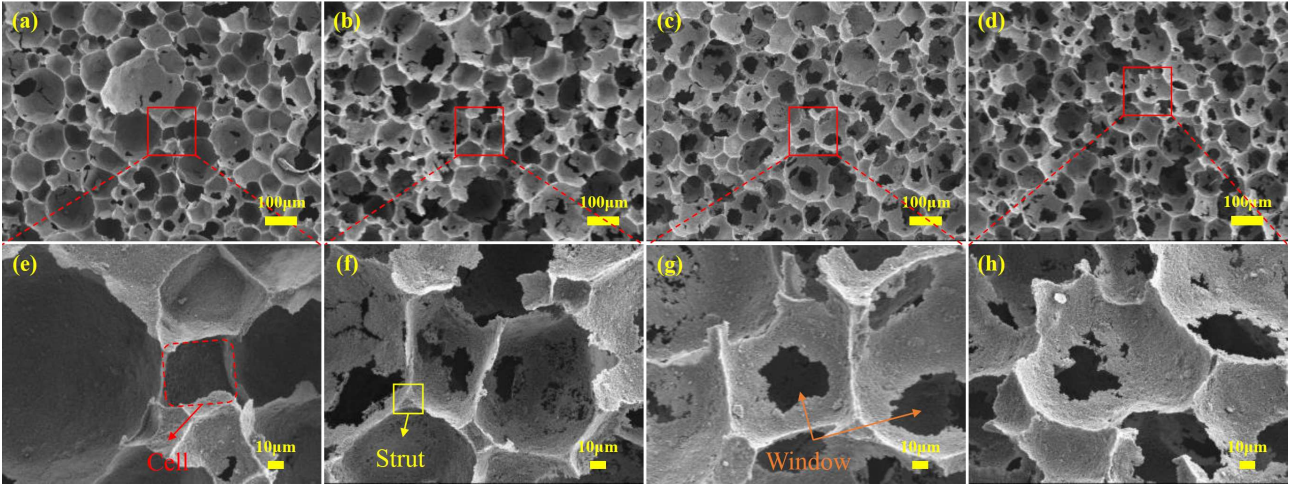


Figure 4. SEM images of alumina ceramic foams with a solid loading of 25 wt.% α -Al₂O₃ and different amount of γ -AlO(OH): (a, e) 0 wt.%, (b, f) 2 wt.%, (c, g) 4 wt.% and (d, h) 6 wt.% (samples were sintered at 1500 °C)

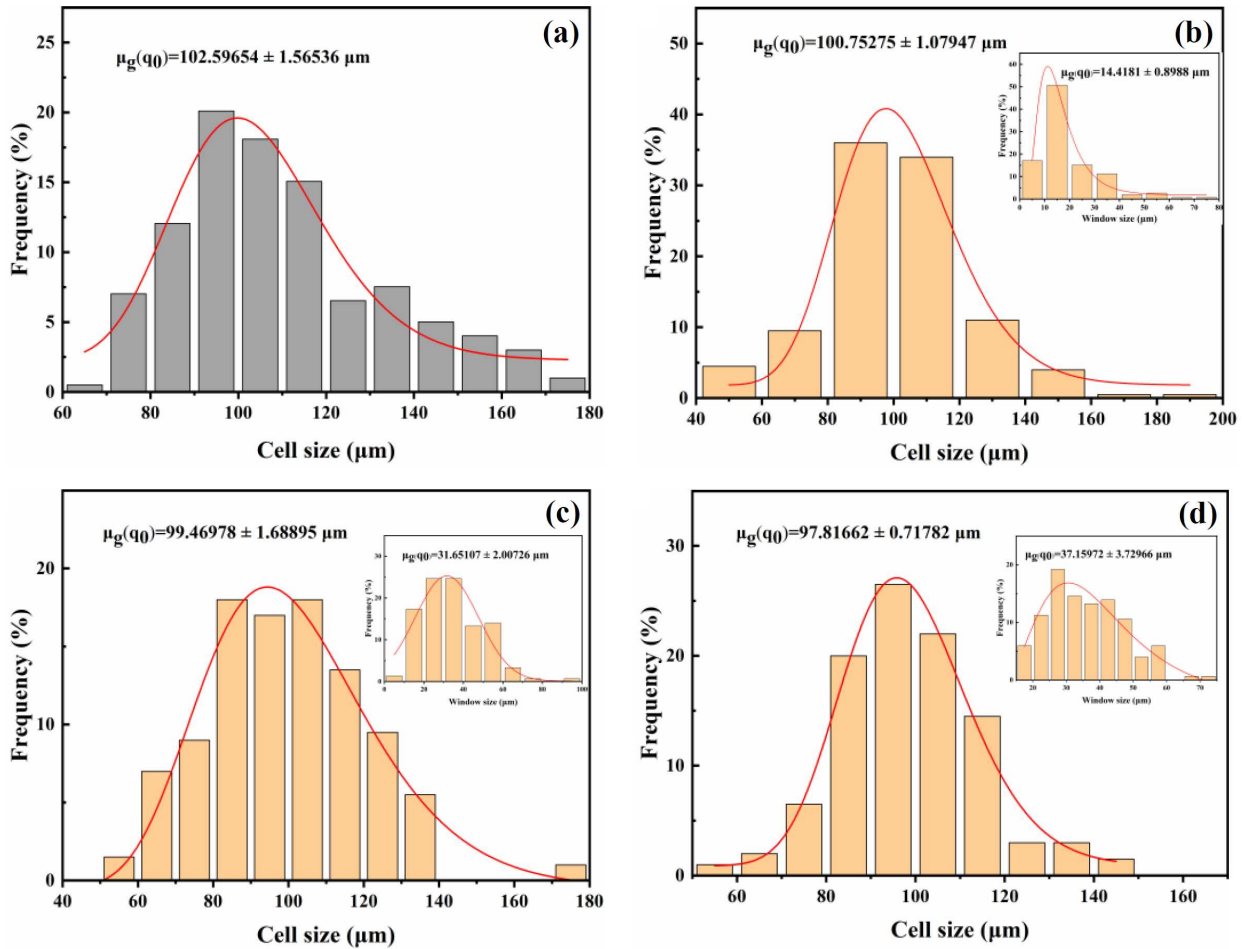


Figure 5. Cell and window sizes distributions of alumina ceramic foams prepared with different amount of γ -AlO(OH): a) 0 wt.%, b) 2 wt.%, c) 4 wt.% and d) 6 wt.% (samples were sintered at 1500 °C)

formed into α -Al₂O₃ during the sintering process. The density of γ -AlO(OH) is 2.42 g/cm³, while that of α -Al₂O₃ is 3.97 g/cm³ [19,20]. A 39.04% volume shrinkage occurs when γ -AlO(OH) is converted to α -Al₂O₃ during the sintering process, providing the required volume for the opening pores. Although only a slight amount of γ -AlO(OH) is added, due to the volume

shrinkage, the particles of γ -AlO(OH) and the adjacent alumina particles produce obvious gaps. When this occurs in large quantities, distinct weak regions on the cell wall and tearing effects are generated [21]. As the sintering progresses, the tearing effect becomes more pronounced as the grain grows, and these weak regions gradually form windows of tens of microns in size. It

is important to note that the windows almost invariably appear on the cell walls rather than on the cell struts, that is, the structure of the cell struts is not significantly altered. This is quite different from the way how the opening is achieved with the introduction of a sacrificial phase. This plays a very crucial role in the stability of the mechanical properties of ceramic foams [22].

3.2. Porosity and compressive strength

Figure 6a shows the porosity and compressive strength of the obtained alumina ceramic foams. With the increase of the γ -AlO(OH) addition, the porosity first increased and then decreased, and the corresponding compressive strength showed opposite trend. Compared with the control group (i.e. the foam without γ -AlO(OH)) with a compressive strength of 0.94 MPa at the porosity of 94.02%, the porosity and strength of the ceramic foam after the addition of γ -AlO(OH) increased at the same time. The porosity reached 94.81% and the compression strength reached 1.17 MPa for the sample with 4 wt.% γ -AlO(OH), while the density was only 0.21 g/cm³. The increase in porosity is mainly due to the opening of the pores formed by the volume shrinkage of γ -AlO(OH).

Compared with the mechanical properties of the alumina ceramic foams prepared by the sacrificial phase

method, the specific strength of the alumina foams prepared in this work is significantly higher, as shown in Fig. 6b [9,23–26]. The synergistic increase in porosity and strength of the alumina ceramic foam after the addition of γ -AlO(OH) is mainly due to the following points:

- Firstly, the cell struts, which are the main load components, do not have apparent defects due to the opening, as shown in Fig. 7a,b. As mentioned earlier, the presence of a window in the cell wall has a minor effect on the compressive strength of the ceramic foams. This is significantly different from the way the sacrificial phase was introduced to achieve the opening, which resulted in large defects in the pore pillars.
- Secondly, γ -AlO(OH) has the effect of ceramic binder. With the increase of γ -AlO(OH) addition, due to the tiny grain size of γ -AlO(OH), it has elevated specific surface area and high sintering activity. This promotes sintering and form strong grain bonding through reactive bonding [15,27]. At the same time, the small α -Al₂O₃ grains, formed by transformation of γ -AlO(OH), play a role in the dispersion strengthening of the ceramic matrix [28], as shown in Fig. 7c. In addition, the reduction of pore size due to the addition of γ -AlO(OH) (Fig. 5) can also improve the compressive strength of alumina ceramic foams [29,30].

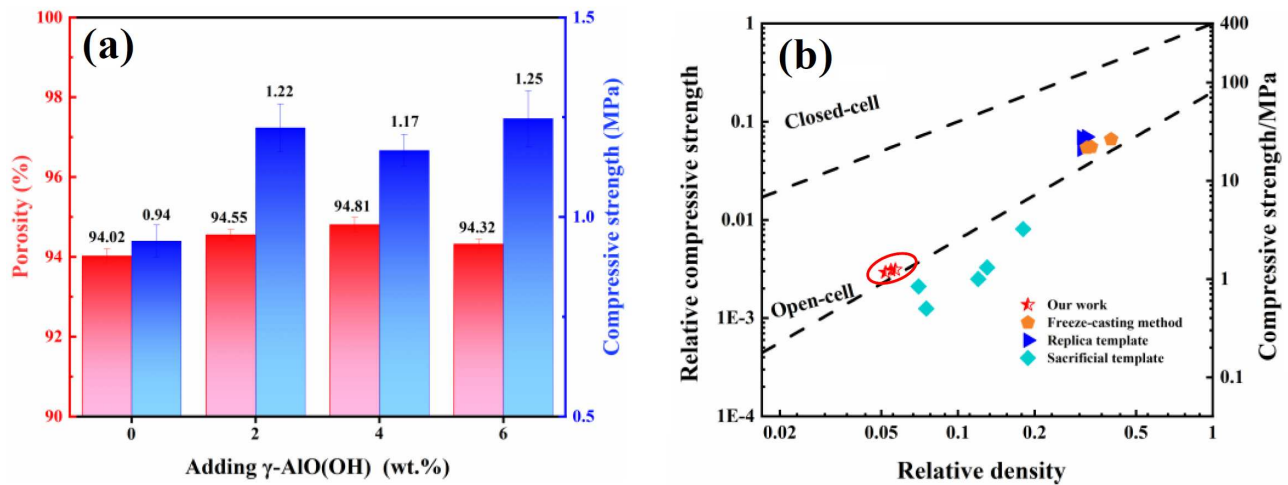


Figure 6. Porosity and compressive strength of ceramic foams prepared with different amount of γ -AlO(OH) and sintered at 1500 °C (a) and strength of the open-cell ceramic foams obtained in this work compared with the strength of bulk alumina foams reported in the literature [9,23–26] (b)

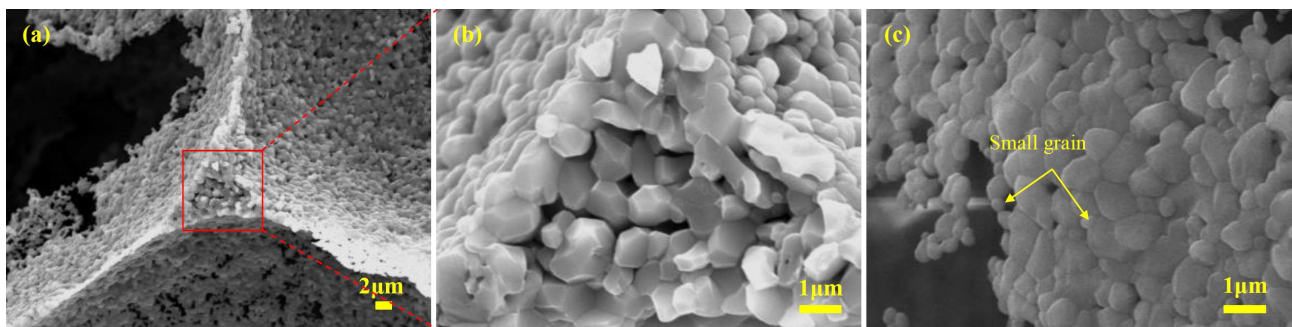


Figure 7. SEM images of the strut in alumina foams with 2 wt.% γ -AlO(OH) (a, b) and SEM image of the small grain of α -Al₂O₃ produced by the reaction of γ -AlO(OH) (c)

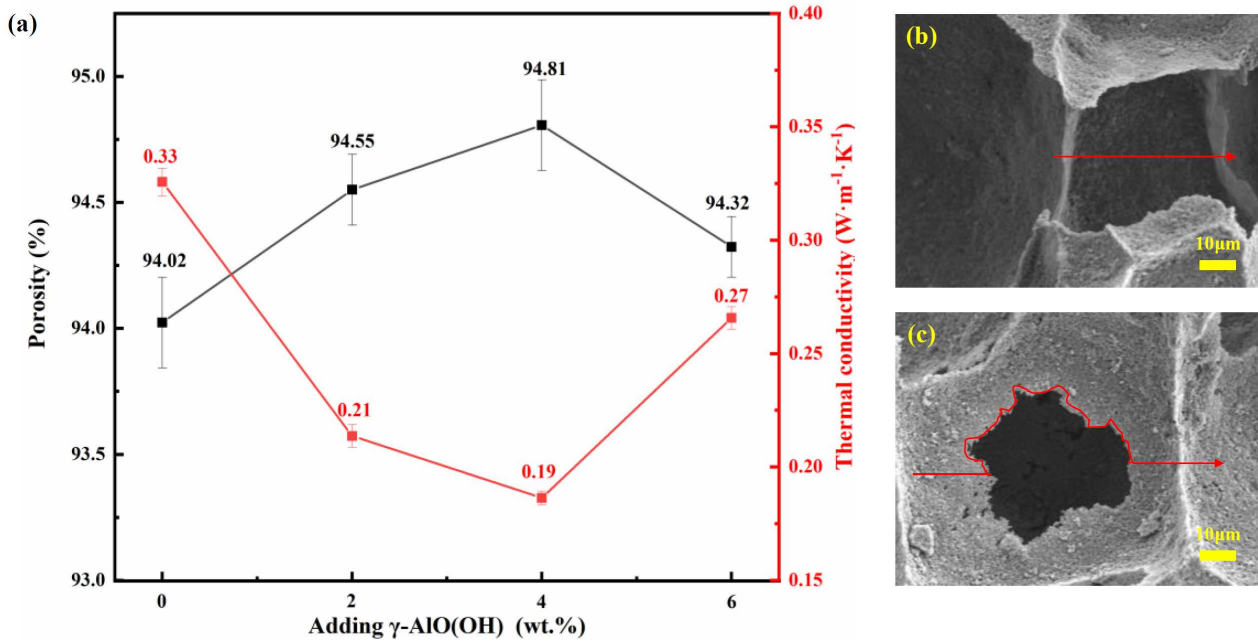


Figure 8. Porosity and thermal conductivity of alumina ceramic foams prepared with a solid loading of 25 wt.% α - Al_2O_3 and different amount of γ -AlO(OH) and sintered at 1500 °C (a). Schematic representation of the conduction path of heat on the wall of the ceramic foams: b) closed-cell and c) open-cell

3.3. Thermal conductivity

Figure 8a shows the porosity of the alumina ceramic foam and the corresponding thermal conductivity of the samples with different γ -AlO(OH) amounts. The thermal conductivity of the open-cell alumina ceramic foam was significantly reduced compared to the control group with a closed-cell structure. The porosity of the ceramic foam prepared without γ -AlO(OH) is 94.02% and the corresponding thermal conductivity is $0.33 W \cdot m^{-1} \cdot K^{-1}$. With the increase of the γ -AlO(OH) amount the thermal conductivity decreased at first and then increased. The thermal conductivity was $0.19 W \cdot m^{-1} \cdot K^{-1}$ when the porosity reached 94.81%. When γ -AlO(OH) is further added, the porosity decreases and the thermal conductivity increases. This confirms that porosity still has a significant effect on the thermal conductivity in open-cell ceramic foams.

The reduced thermal conductivity of the resulting ceramic foam is mainly due to the open-cell structure, since in porous ceramics heat is mainly conducted through the solid material. A large number of windows appear compared to the closed-cell structure in the control group without γ -AlO(OH) addition (Fig. 8b), resulting in an increased path during heat conduction, then resulting in a decrease in thermal conductivity (Fig. 8c) [31]. At the same time, increasing the porosity by implementing open pores is also an influential cause of the decrease in thermal conductivity [32]. Therefore, the preparation of open-cell structures is an effective method to reduce the thermal conductivity of ceramic foams. This provides a current strategy for the application of ceramic foams in the field of thermal insulation.

IV. Conclusions

In this paper, instead of introducing a sacrificial phase, a small amount of γ -AlO(OH) (boehmite) was added to the α - Al_2O_3 slurry to produce open-cell alumina ceramic foams with high porosity and strength. The 39.04% volumetric shrinkage of γ -AlO(OH) achieves the open-cell structure while avoiding the generation of polluting gasses. The resulting open-cell alumina ceramic foams have a very high porosity of 94.32–94.81% and a corresponding compressive strength of 1.16–1.26 MPa, which is significantly higher than the sacrificial phase method of open-cell ceramic foams. This synergistic increase in porosity and strength is mainly due to the fact that the opening of the pore is achieved without creating significant defects on the pore struts. In addition, the added γ -AlO(OH) has a high sintering activity, which promotes the sintering of ceramic grains, and the small grains of α - Al_2O_3 formed by the γ -AlO(OH) reaction are evenly distributed in the matrix, which plays a role in the dispersion strengthening. The implementation of an open-cell structure makes the path of heat conduction longer and thus reduces the thermal conductivity. This paper provides a new strategy for the preparation and application of new environmentally friendly open-cell ceramics that can be widely used in filtration, catalyst support, adsorption, thermal insulation and other fields.

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