A three-dimensional mullite-whisker network ceramic with ultra-light weight and high-strength prepared by the foam-gelcasting method

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ABSTRACT
A three-dimensional mullite-whiskers network ceramic with ultra-light weight and high-strength was obtained by the foaming gel-casting method with active mullite powders and NH₄F. Ultrafine active mullite powder was used as the raw material and SDS (sodium dodecyl sulfate) as a pore-forming agent. The effects of sintering temperature on the microstructure, flexural strength and thermal conductivity of the porous ceramics were systematically investigated. The mullite-whisker network porous ceramic has a fibrous-framework structure composed of interweaving mullite-whiskers. Because the pore-walls are composed of interlacing whiskers and the pores are filled with whiskers, the porous ceramics exhibited good mechanical properties and low thermal conductivity. The flexural strength of the obtained mullite-whisker porous ceramic sintered at 1500°C was up to 1.45 Mpa, while the porosity was as high as 89%. In addition, the thermal conductivity of the porous ceramic at room temperature was as low as 0.045 W/m-K.

1. Introduction

People today are paying more and more attention to energy saving and environmental protection, and thermal insulation and filtering materials have therefore been widely researched [1]. Ultra-light porous ceramics have attracted widespread interest due to their excellent characteristics such as antifouling properties, mechanical resistance, chemical stability and superior thermal stability [2,3]. In addition, mullite materials retain superior mechanical properties at high temperatures and display excellent creeping and thermal-shock resistance [4]. Therefore, mullite porous ceramic have been wildly applied in many engineering technologies, such as filters for waste water treatment and thermal insulation [5–7]. In the future, mullite porous ceramic with high porosity promise to be among the most important materials for engineering application.

Various technologies have been used to prepare mullite porous ceramics, such as consolidation [8], direct foaming [9], pore-forming [10], gel casting [11], gel freeze drying [12] and 3D printing [13]. Among these routes, the pore-forming method is considered one of the most efficient and economical methods [14]. However, the poor mechanical properties of mullite porous ceramics prepared by this method seriously hamper their application [15]. It is rare to produce porous ceramics with a continuous pore wall structure. Recently, porous ceramics toughened by mullite whisker grain have emerged as one of the most promising approaches to achieve porous ceramics with excellent mechanical strength [16,17]. Thus, the production of mullite whiskers in the pores and pore walls of porous ceramics provides a novel idea for the manufacture of mullite porous ceramics with excellent properties. These whiskers can be used as bridges between the pore walls and pores, and can improve the compressive strength of the porous ceramics [18]. Porous ceramic materials with a three-dimensional mullite-whisker framework have been attracting much attention during the past decade due to their unique whisker and pore structure [19,20]. Few studies have been reported for preparation of low-density mullite porous ceramics with perfect whisker skeletons. Haught et al. [21] successfully prepared mullite whiskers from AlF₃ and SiO₂ as raw materials by the one-step sintering method. They found that gas-solid reactions among the raw materials boost mullite whisker generation at high temperatures. However, the mullite whiskers obtained by this method had an uneven morphology, rough surface and small aspect ratio. Rashad et al. [22] developed a mullite porous ceramic from clay and aluminum fluoride trihydrate (AlF₃·3H₂O) with Al₂O₃. In their work, needle-shaped mullite formed an interlocking network at above 1300°C. However, the small aspect ratio of the mullite whiskers resulted in low porosity and strength of the resulting porous ceramic. Hence, it is meaningful to prepare a mullite porous ceramic with a perfect whisker skeleton framework for practical applications.

In this study, we successfully developed a simple and facile way to a prepare high-performance porous ceramic with a mullite whisker framework using dextrin and SDS as pore-forming agents. Interestingly, the
sillimanite phase was observed at low temperatures, and was then transformed into mullite whiskers with increases in the sintering temperature. During the preparation process, pores of different sizes were evenly distributed throughout the porous ceramic. These pores not only reduced the bulk density but also provided growth space for the whiskers through a gas-solid reaction, where the pore-walls were composed of interlacing whiskers and the pores were filled with whiskers. The as-prepared porous ceramic with a mullite-whisker framework structure showed low thermal conductivity and good mechanical strength after heat treatment at high temperature. The mullite-whisker porous ceramic also possesses relatively high flexural strength, ultra-low thermal conductivity and superior high porosity. Therefore, this mullite whisker porous ceramic with high porosity and strength can be used as a high-temperature thermal insulation and filtering material.

2. Experimental procedures

2.1. Raw materials

Aluminum sulfate power (Al$_2$(SO$_4$)$_3$·18H$_2$O, 99.9% purity, Aladdin, China), sodium silicate power (NaSiO$_3$·9H$_2$O, 99.95% purity, Aladdin, China), hydrochloric acid (HCl, 36-38%wt, Shanghai, China) and ammonia water (NH$_4$OH, 25-28%wt, Shanghai, China) were used as the raw materials to obtain the precursor mullite powder. Ammonium fluoride (NH$_4$F, Anshan Chemical Reagents Factory, China) was selected as an additive to obtain the mullite whiskers. Dextrin and SDS ((C$_6$H$_{10}$O$_5$)$_n$, 99% purity, Shanghai, China) were selected as the foaming agents to fabricate porous ceramics. All chemicals used in this study were analytic reagent grade.

2.2. Sample preparation

In this study, mullite precursor powders were prepared by the coprecipitation reaction-azeotropic distillation method (shown in Figure 1(a)), in which the molar ratio of the raw materials were calculated according to Equations (1) and (2). First, the determined amounts of aluminum sulfate and sodium silicate were dissolved in a moderate amount of water distilled in a beaker. The excess hydrochloric acid was poured into the beaker slowly with continuous stirring for 30 min to assure a full reaction among the raw materials. The excess ammonia water was then poured into the beaker slowly as well with continuous stirring for 30 min. Next, the well-stirred stable suspension slurry was poured into a Buchner funnel and filtrated by vacuum pump until the water was gone. This process was repeated 3–5 times to remove the Na$^+$ and SO$_4^{2-}$ completely. Finally, the well-filtrated mullite precursor powder was entered into a rotary evaporator with moderate n-amyl alcohol selected as the solvent to improve the homogeneity of the precursor powder. The rotary evaporator was heated in a water bath with the temperature raised slowly to 99°C, and the mullite precursor powder was then dried at 110°C for 12 h (Figure 1(a)).

$$Na_2SiO_3 + 2HCl \rightarrow H_2SiO_3 + 2NaCl \quad (1)$$

$$Al_2(SO_4)_3 + 6NH_4OH \rightarrow 2Al(OH)_3 + 2(NH_4)_2SO_4 \quad (2)$$

The fabrication process for the mullite-whisker-framework porous ceramic is illustrated in Figure 1. Firstly, ammonium fluoride was dissolved in 50 ml deionized water with stirring for 10 min. Secondly, dextrin and SDS (0.8 g, respectively) were added the solution as the foaming agent and foam stabilizer, respectively. After stirring,
the precursor mullite powders were added to the premix solution in a molar ratio of Al: Si: F = 3:1:12. In order to ensure complete mixing of the raw materials, the mixture was mechanically stirred for 10 min to achieve a stable slurry. Finally, the stable suspension was cast into molds while the gelation process continued to produce specimens 10 × 10 × 50 mm in size. After drying for 24 h, the green bodies were removed from the molds and dried at 40°C for 12 h. Then the mixed materials were dried at 110°C for 24 h. After drying, the samples were sintered at the final temperature for 3 h under an air atmosphere. The heating rate was 5°C/min, and the sintered samples were then cooled naturally.

### 2.3. Characterization method

Crystalline phase evolution of the mullite precursor powder was followed by X-ray diffraction (XRD, CuKa source (λ = 0.154056 nm), 6º/min, 10º < 2θ < 90º). The bulk density (D_b) of the mullite-whisker porous ceramic was calculated from the mass-to-volume ratio of the specimens. Porosity (P) was calculated from the bulk density of the ceramic (D_b), and the actual density of the dense mullite ceramic was determined using the equation P = (1-Db/Dt) × 100%. Pore size distribution was measured using a mercury intrusion porosimeter (AutoPore IV 9510, Micromeritics, Norcross, GA, USA). A thermal conductivity instrument (TC-3000, Xian Xiaxi Electric, China) was used to test the thermal conductivity of the samples at room temperature by the transient hot-wire method. The thermogravimetry differential scanning calorimetry of the green bodies (DSC/TG, Zeiss, Germany) was measured from room temperature to 1400°C at a heating rate of 10°C/min in an air flow. A weak endothermic peak is seen at around 720°C with a weight loss of about 5 wt %. In this stage, the slight weight loss was attributed to a chemical reaction of AlF_3 with air to form a gas phase. Combined with the XRD pattern, the AlF_3 phase disappears, and sillimanite becomes the main phase at 730°C. Hence, in this stage AlF_3 reacts with amorphous SiO_2 to form sillimanite. The sillimanite phase is then transformed into mullite whiskers at 1000°C. According to the TG-DSC curve and XRD pattern, the sintered temperature of green bodies of the mullite-whisker framework was determined to be below 1200°C.

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\begin{align*}
(NH_4)_3AlF_6 & \rightarrow 2NH + NH_2AlF_4 + 2HF \\
(NH_4)_2SiF_6 + 2H_2O & \rightarrow 2NH_3 + SiO_2 + 6HF
\end{align*}
\]

### 3. Results and discussion

#### 3.1. Thermal analysis and phase characterization

Figure 2 shows the TG-DSC pattern of a green body heated in a dry air atmosphere. A broader endothermic peak was observed at about 120°C due to the evaporation of the absorbed water and volatile portions of the mixture, with a mass loss in the TG curves. In the range of 204-297°C, a sharp exothermic peak with a quick mass loss is seen in the DSC curve. In this stage, the decomposition of ammonium hexafluoroaluminate ([(NH_4)_3AlF_6]) and (NH_4)_2SiF_6 and combustion of the residual alcohol from the mullite precursor powder according to the chemical formulas shown in (3) and (4) comprise the main reaction. A large exothermal peak is observed at around 551.3°C, indicating that the combustion process of the foaming-agent dextrin and the thermal decomposition of ammonium tetrafluoroaluminate (NH_4AlF_4) are the main reactions, which corresponds with Figure 3. A weak endothermic peak is seen at around 720°C with a weight loss of about 5 wt %. In this stage, the slight weight loss was attributed to a chemical reaction of AlF_3 with air to form a gas phase. Combined with the XRD pattern, the AlF_3 phase disappears, and sillimanite becomes the main phase at 730°C. Hence, in this stage AlF_3 reacts with amorphous SiO_2 to form sillimanite. The sillimanite phase is then transformed into mullite whiskers at 1000°C. According to the TG-DSC curve and XRD pattern, the sintered temperature of green bodies of the mullite-whisker framework was determined to be below 1200°C.

![Figure 2. TG-DSC curves of the slurry mixture conducted in dry air.](image)

![Figure 3. XRD patterns of green bodies fired at various temperatures.](image)
Figure 3 shows the XRD spectrum of green bodies fired at 260°C, 580°C, 730°C, 1000°C, and 1200°C for 2 h and active precursor mullite powder dried at 110°C, respectively. The active mullite precursor powder was synthesized from amorphous Al₂O₃ and amorphous SiO₂. Only diffraction peaks of (NH₄)₂AlF₆ and (NH₄)₂SiF₆ in the green bodies can be clearly observed at 110°C, as shown in Figure 3. When the green bodies were fired at 260°C, the diffraction peaks of (NH₄)₂AlF₆ completely disappeared, and NH₄AlF₄ became the primary crystalline phase. At the same time, the diffraction peaks of (NH₄)₂SiF₆ completely disappeared. Subsequently, when the firing temperature was increased to 580°C, the NH₄AlF₄ phase was transformed into AlF₃. At 730°C, sillimanite became the main crystalline phase and a few weak diffraction peaks of Al₂O₃ were also observed. The diffraction peaks of mullite appeared when the samples were fired at 1000°C. The mullite phase without other obvious impurity phase peaks was observed when the samples were fired at 1200°C. These results indicate that the Mullite transformation from sillimanite may be completed at a temperature of approximately 1200°C. Thus, in this study, mullitization was achieved at below 1100°C, as the degree of mixing of raw materials was achieved at the molecular level, which is beneficial for mullite synthesis.

3.2. Microstructure of the high-porous mullite whisker-framework ceramic

Figure 4 presents the SEM images of a mullite porous ceramic without NH₄F sintered at 1400°C. The as-prepared sample with uniform distribution of spherical pores and pore sizes possessed high porosity. This porous ceramic exhibited a typical pore structure prepared by the foam gel-casting method, including large spherical pores from the foaming process and small pores in the internal walls from the removal of the organic phase during the sintering process. Due to the relatively low solid content of the slurry, solid particles adhering to bubbles exhibit different degrees of shrinkage during the drying process, a similar situation to that reported by Liu [23]. In addition, the as-prepared sample does not form a continuous pore wall structure.

A comparison with Figure 4 shows that the microstructure of the obtained mullite porous ceramic has obviously changed with the introduction of additives. A large number of fine needlelike mullite whiskers is observed in the pores and pore walls (as shown in Figure 5). The growth of mullite whiskers mainly involved two mechanisms, gas-solid (VS) and gas-liquid-solid (VLS), which are universal in mullite materials [16]. As seen from Figure 9(c), the prepared mullite whisker surface is smooth, and no liquid phase occurs during the growth of the mullite whiskers. This indicates that the formation of whisker growth takes place via a VS process. As shown in Figures 3 and 2, the micron particles (NH₄)₂AlF₆ and (NH₄)₂SiF₆ are decomposed into AlF₃ and amorphous SiO₂ [24]. With increases in the heating temperature, the AlF₃ reacted with O₂ to become transformed into gaseous AlOF. During this process, amorphous SiO₂ was decomposed by catalysis, resulting in the generation of gaseous SiF₄ in the crucible [25]. Then gaseous AlOF reacted with the SiF₄ to form a sillimanite nucleus, as is confirmed by Figure 3. With increasing heat, mullite crystals were directly transformed from sillimanite nucleus, and the nucleus then grew into whiskers with a continuous supply and reaction of the AlOF and SiF₄ gases. The generation of in-suit mullite whiskers took place continuously until a network structure eventually formed.

Figure 5 shows SEM images of samples sintered at different temperatures. Due to the poor strength with no practical value of the sample sintered at 1200°C, we have not characterized its performance. At 1300°C, a uniform porous microstructure is observed, composed entirely of mullite whiskers forming a skeleton for the pore structure. Because of the short length (16 μm) and diameter (0.24 μm) of the whiskers, as shown in Figure 6, a mass of unfixed lap-joint points can be observed in the pores and pore walls. At 1400°C (Figure 5(c)), the mullite whiskers grew thicker and longer and interlocked with each other. On the sample sintered at 1500°C, the average length of the mullite whiskers achieved the maximum of 58 μm, an aspect ratio of 18.81. In addition, mullite whiskers interlocked with each other formed a 3D network framework structure in both the pores and pore walls. At 1600°C, meanwhile, the sintering behavior of the sample ceased to be promoted, and the average length and aspect ratio of the elongated mullite decreased to 0.6 μm and 7.83,
respectively. Long columnar mullite grains were present in the pore walls, moreover, but not inside the pores. At the same time, the elongated mullite grains were wrapped by the glass phase and combined with the glass phase to form blockage in the large pores (Figure 5(d)).

3.3. Mechanical properties of highly-porous mullite-whisker framework ceramic

Figure 7 shows the pore size distributions, bulk density and porosity of porous ceramics, and confirms the presence of a close relationship between these results and the terminal sintering temperature. Figure 7(b) illustrates the porosity and bulk density of mullite-whisker porous ceramic sintered at different temperatures. As can be seen from Figure 7(b), with an increase in the sintering temperature from 1300 to 1600°C, there is little change in the porosity of the mullite-whisker porous ceramic, which varies from 78% to 89%. In addition, the change in the bulk density is also small, varying from 0.46 to 0.57 g/cm³. Combined with Figure 5, this
shows that the formation of a mullite-whisker skeleton is favorable for improving the porosity. The length and aspect ratio of the mullite whiskers increased with increases in the sintering temperature from 1300 to 1500°C, and the porosity of the ceramic then increased slightly.

When samples were sintered at 1300°C, 1400°C, 1500°C and 1600°C, the value of their average pore size was 11, 2.8, 1.0 and 71 μm, respectively. With increases in the sintering temperature, the average pore size value decreased at first and then increased, and the minimum value was obtained at 1500°C. The change in pore size distribution may have been caused by the formation of the whiskers in the pores and pore walls. In addition, the curve of the pore size distribution mainly exhibited a double-peak, as confirmed by the SEM images, presented in Figure 5. When a sample was sintered at 1300°C, only short, fine needlelike whiskers were produced, the filling and separation capability of the pores of mullite-whiskers was insufficient, and the average pore size was relatively large. With increases in the sintering temperature, the aspect ratio of the mullite-whiskers became shorter. At 1500°C, both the aspect ratio and number of mullite-whiskers increased, resulting an improved ability to fill the pores. Meanwhile, the high aspect ratio of the mullite-whiskers protruding from the pore walls, not only filling the pores but also dividing the large pores into a small pores. Therefore, large numbers of secondary micro-pores were formed between these mullite-whiskers network structures in the pores and pore walls by interweaving of the mullite-whiskers. All of these factors contributed to reducing the average pore size and forming more micro-pores in the samples. When the sintering temperature was 1600°C, the needlelike mullite-whiskers were transformed into elongated mullite grains and some micro-pores became larger, which led to enlargement of the average pore size, as seen in Figure 5(d). It was confirmed that the numbers and aspect ratio of the mullite whisker were reduced, and that the ability of the whiskers to fill and divide pores declined, leading to sequential and enlargement of the average pore size.

As illustrated in Figure 8, with increases in the sintering temperature from 1300 to 1600°C, the flexural strength of the mullite-whisker framework porous samples increased initially and then decreased. The flexural strength of the sample sintered at 1500°C was as high as 1.45 MPa, which was much greater than that of porous mullite prepared using the conventional foaming method [26,27]. The much improved mechanical properties of the mullite porous ceramic discussed in this paper could be attributed to the excellent microstructure of the sample. As seen in Figure 5 as well, with increases in the sintering temperature, both the length and diameter of the mullite whiskers increased. The temperature rose to 1500°C, the whiskers deep in the pores and pore walls interlaced with each other to form a network structure. This represents a reinforcement phase featuring mullite-whiskers with a high aspect ratio, which is beneficial to improving the strength of porous samples [28,29]. When samples were sintered at 1600°C, the structure changed remarkably (as seen in Figure 5d), the ratio of the whiskers’ length to diameter decreased, and they became elongated mullite grains. This resulted in a weak interface of whisker interaction and a reduction in the strength of the samples. At the same time, the flexural strength (1.259 MPa) was

![Figure 7](image_url) (a) Pore size distribution and (b) porosity of samples sintered at different temperatures.

![Figure 8](image_url) Flexural strength and thermal conductivity of samples sintered at 1300, 1400, 1500 and 1600°C.
higher than that (1.06 MPa) of porous mullite ceramics obtained by the conventional foaming method [23].

We hypothesize that the mullite-whisker framework is responsible for the outstanding mechanical properties of the porous ceramic, primarily the structure that allows for large loading without fracture. On the structure with independent large pores shown in the as-prepared samples sintered at 1300-1400°C (Figure 9(a) and (b)), the length of the mullite whiskers was too short to form a mullite-whisker framework in the pores, but they did form one in the pore walls, giving them relatively high strength. At 1500°C, the high aspect ratio of the mullite whiskers in larger pores and pore walls enabled them to intersect with each other to form a 3D interlocking framework. The diameter of mullite whiskers sintered at 1500°C was larger than that of those sintered at 1300°C. In addition, the whiskers in the sample sintered at 1500°C were more flourishing than those in the sample sintered at 1400°C. What is more, those lap-joint whiskers acted as bridges between the pore walls during the loading process (Figure 9(c)). In this case, loading stress could be transferred uniformly to the pores and pore walls inside the samples, showing a high resistance to load. On the sample sintered at 1600°C, elongated mullite whiskers were observed only in the pore walls (Figure 9(d)) as a reinforcement phase, contributing further to improvement of the mechanical properties [30]. Meanwhile, as shown in Figure 8, the obtained mullite-whisker porous ceramic exhibited ultra-low thermal conductivity, which increased slightly from 0.039 W/m-K to 0.066 W/m-K with sintering temperatures from 1300 to 1600°C. As illustrated in Figures 5 and 9, because of the whiskers bundled at lap-jointing points, heat transfer in the samples was significantly obstructed [31]. Therefore, the as-prepared porous ceramic has potential to be applied in high temperature-resistant thermal insulation materials.

Considering the widespread application of porous mullite ceramics in the filtration field, the sample sintered at 1500°C was selected to demonstrate the filtration effect of the obtained porous ceramic. An image of oil-in-water emulsions before and after the separation is shown in Figure 10. The oil-in-water emulsion exhibited a slightly turbid, milky appearance before the separation test. After filtration through the mullite-whisker framework porous ceramic at a pressure of 0.2 MPa, the turbid oil-in-water solution had become clear and transparent. We consider that the color change of the solution can be attributed to high-efficiency oil-in-water separation by the mullite-whisker framework porous ceramic. As shown in Figure 5, it can be seen that the secondary micro-pores composed by the interweaving of mullite-whiskers in large pores is beneficial to the filtering effect of porous ceramics. These result demonstrate that the obtained mullite-whisker framework porous ceramic is potentially suitable for application in the filter field.

4. Conclusion
In this study, a mullite-whisker self-reinforced framework porous ceramic has been successfully prepared from active-mullite powders and NH₄F with dextrin and SDS as pore-forming agents using the foam-gelcasting method. The mullite porous ceramic is reinforced by a three-dimensional mullite-whisker network, which acts as a bridge between the pores and forms an interlocking framework in the pore walls. In addition, large numbers of secondary pores shaped by the intersection of mullite-whiskers in the pores and pore walls are instrumental in reducing the thermal conductivity of the porous ceramic. The obtained mullite-whisker skeleton porous ceramic possesses very high porosity (81.3–89.3%), ultra-light weight (0.46–0.57 g/cm³), ultra-low thermal conductivity (0.045–0.067 W/m-K) and excellent flexural strength (0.76–1.45MPa). Unlike conventional porous ceramics with mostly closed pores and dense cell walls prepared by the direct foaming method, the mullite-whisker framework ceramic is easily permeable, due to its unique three-dimensional mullite-whisker framework with plenty of channels between whiskers. The mullite-whisker skeleton porous ceramic also possesses excellent filtering capabilities. Thus, the as-prepared mullite-whisker network porous ceramic exhibits low

![Figure 9](image-url). Schematic diagram illustrating the load distribution and secondary pore formation processes in mullite-whisker porous ceramics.
density and thermal conductivity and high strength, and it can be expected to find use as an insulating and filtering material.

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Disclosure statement

No potential conflict of interest was reported by the authors.

References

[1] Xu L, Xi X, Zhu W, et al. Investigation on the influence factors for preparing mullite-whisker-structured porous ceramic. J Alloy Compd. 2015;649:739–745.
[2] Zhu L, Dong Y, Hampshire S, et al. Waste-to-resource preparation of a porous ceramic membrane support featuring elongated mullite whiskers with enhanced porosity and permeance. J Eur Ceram Soc. 2015;35 (2):711–721.
[3] Zhu L, Dong Y, Li L, et al. Coal fly ash industrial waste recycling for fabrication of mullite-whisker-structured porous ceramic membrane supports. RSC Adv. 2015;5 (15):11163–11174.
[4] Hua K, Shui A, Xu L, et al. Fabrication and characterization of anorthite–mullite–cordundum porous ceramics from construction waste. Ceram Int. 2016;42(3):6080–6087.
[5] Vogt U, Garbar M, Dimopoulos-Eggenschwiler P, et al. Improving the properties of ceramic foams by a vacuum infiltration process. J Eur Ceram Soc. 2010;30(19):3005–3011.
[6] Li N, Zhang XY, Qu YN, et al. A simple and efficient way to prepare porous mullite matrix ceramics via directly sintering SiO2-Al2O3 microspheres. J Eur Ceram Soc. 2016;36(11):2807–2812.
[7] Yang M, Li J, Man Y, et al. A novel hollow alumina sphere-based ceramic bonded by in situ mullite whisker framework. Mater Des. 2020;15(186):108334.
[8] Barea R, Osendi M, Miranzo I, P Ferreira J. Fabrication of highly porous mullite materials. J Am Ceram Soc. 2005;88(3):777–779.
[9] Zhao J, Yang C, Shimai S, et al. The effect of wet foam stability on the microstructure and strength of porous ceramics. Ceram Int. 2018;44(1):269–274.
[10] Sciamanna V, Nait-Ali B, Gonona M. Mechanical properties and thermal conductivity of porous alumina ceramics obtained from particle stabilized foams. Ceram Int. 2015;41(2):2599–2606.
[11] Liu YF, X Q L, Wei H, et al. Porous mullite ceramics from national clay produced by gelcasting. Ceram Int. 2001;27(1):1–7.
[12] Ding S, Zeng YP, Jiang D. Fabrication of mullite ceramics with ultrahigh porosity by gel freeze drying. J Am Ceram Soc. 2007;90(7):2276–2279.
[13] Elsayed H, Rebesan P, Crockave MC, et al. Biosilicate scaffolds produced by 3D-printing and direct foaming using preceramic polymers. J Am Ceram Soc. 2019;102:1010–1020.
[14] Minas C, Carnelli D, Tervoort E, et al. 3D printing of emulsions and foams into hierarchical porous ceramics. Adv Mater. 2016;28:9993–9999.
[15] Liu J, Ren B, Lu Y, et al. Novel design of elongated mullite reinforced highly porous alumina ceramics using carbonized rice husk as pore-forming agent. Ceram Int. 2019;45(11):13964–13970.
[16] Chen N, Wang H, Huo J, et al. Preparation and properties of in-situ mullite whiskers reinforced aluminum chromium phosphate wave-transparent ceramics. J Eur Ceram Soc. 2017;37(15):4793–4799.
[17] Xiao Peng LW, Li Z, Yu X, et al. Thermal cycling behavior and oxidation resistance of SiC whisker–toughened mullite/SiC coated carbon/carbon composites in burner rig tests. Corros Sci. 2016;106:179–187.
[18] Wang X, Guo A, Liu J, et al. Effects of in situ synthesized mullite whiskers on compressive strength of mullite fiber brick. Ceram Int. 2016;42(11):13161–13167.
[19] Su L, Wang H, Niu M, et al. Ultralight, recoverable, and high-temperature-resistant SiC nanowire aerogel. ACS Nano. 2018;12:3103–3111.
[20] Jung SM, Jung HY, Fang W, et al. A facile methodology for the production of in situ inorganic nanowire hydrgels/aerogels. Nano Lett. 2014;14 (4):1810–1817.
[21] Haught D, Talmy I, Divecha D, et al. Mullite whisker felt and its application in composites. Mater Sci Eng A. 1991;144(1–2):207–214.
[22] Rashad M, Sabu U, Logesh G, et al. Development of porous mullite and mullite-Al2O3 composite for microfiltration membrane applications. Sep Purif Technol. 2019;219:74–81.
[23] Liu W, Xu J, Wang Y, et al. Processing and properties of porous PZT ceramics from particle stabilized foams via gel casting. J Am Ceram Soc. 2013;96(6):1827–1831.
[24] White MA, Shi HY, Leiper JC. Preparation and characterization of (NH4)3AlF6 and ND4AlF4 including the perdeutero salts (ND4)3AlF6 and ND4AlF4. J Fluor Chem. 1993;62(2–3):211–216.
[25] Okada K, Yosuka N. Synthesis of mullite whiskers and their application in composites. J Am Ceram Soc. 1991;74:2414–2418.
[26] Guo HS, Li W, Ye FB. Preparation of micro-porous mullite ceramics by foaming for high temperature thermal isolation. Ceram Int. 2016;42(15):17332–17338.

[27] Huo W, Zhang X, Chen Y, et al. Ultralight and high-strength bulk alumina/zirconia composite ceramic foams through direct foaming method. Ceram Int. 2019;45(1):1464–1467.

[28] Wang X, Li JH, Guan WM, et al. Emulsion-templated high porosity mullite ceramics with sericite induced textured structures. Mater Des. 2016;89:1041–1047.

[29] Ozturk C, Tur YK. Processing and mechanical properties of textured mullite/zirconia composites. J Eur Ceram Soc. 2007;27(2–3):1463–1467.

[30] Deng X, Ran S, Han L, et al. Foam-gelcasting preparation of high-strength self-reinforced porous mullite ceramics. J Eur Ceram Soc. 2017;37(13):4059–4066.

[31] Huo W, Zhang X, Xu J, et al. In situ synthesis of three dimensional nanofiber knitted ceramic foams via reactive sintering silicon foams. J Am Ceram Soc. 2019;102 (5):2245–2250.