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Effects of alumina fiber content on microstructure and properties of alumina foam ceramics

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As a kind of heat insulating material, alumina foam ceramics have many advantages such as low thermal conductivity, high thermostable performance, good wear resistance, high hardness and excellent resistance to chemical corrosion. In this paper, a high-alumina foam ceramic was prepared by the gel-foaming method using water as dispersing medium, aluminum dihydrogen phosphate as gel and alumina fiber as reinforcing component, and the effects of alumina fiber content on the slurry viscosity as well as the porosity, phase composition, microstructure and mechanical properties of the prepared ceramic were studied. It was found that the viscosity of the slurry increased with the increase of fiber content. After sintering, there were C-$AlPO_4$, T-$AlPO_4$ and α-Al_2O_3 phases in the prepared samples; with the increase of fiber content, the porosity increased first and then decreased, whereas the compressive strength presented an opposite variation trend. In addition, the fracture toughness was also improved owing to the bridging effect of alumina fibers, the crack deflection and the fiber pulling-out mechanism.

Key words: Foam ceramics, Alumina fibers, Microstructure, Compressive strength.

Introduction

Heat insulating material is a kind of functional material that can block heat flux, and has many advantages including light weight, loose bonding, porous structure and low thermal conductivity. Porous ceramic is one of the most important heat insulation materials [1, 2], and has been widely used as the furnace lining material in industrial furnaces and as the critical part material that can be easily burned by high temperature air flow in aerospace engineering. As a kind of heat insulating material, alumina foam ceramic materials have the advantages of low thermal conductivity, good heat resistance, abrasion resistance, good oxidation resistance, good high temperature creep resistance, high hardness, low linear expansion coefficient, excellent resistance to chemical corrosion and so on [3]. However, there are also some obvious shortcomings that seriously restrict the practical applications of alumina foam ceramic materials in some fields [4, 5], such as poor plastic deformation ability (i.e., brittleness), poor ability to withstand intense mechanical shock, and poor resistance of thermal shock. Therefore, some methods, for example, grain size control and phase transformation toughening, should be explored to improve the toughness of alumina foam ceramics. Fibers can absorb the energy of the applied load through the pathways of cracking, debonding and pulling out in the fracture process of ceramic matrix, which not only improve the strength and toughness of ceramic composites, but also retain their good temperature resistance.

In recent years, SiC fibers, silicon nitride fibers and carbon fibers have been extensively used for the reinforcement of ceramic matrix composites. Liu et al. [6] studied the effects of fiber content on the mechanical properties of ceramic composites, and found that the strength and fracture toughness were significantly improved when the content was 5%. Mishra et al. [7] reported that glass fibers of 10 wt% could be evenly distributed in the slurry when L/D ratio was 1000. Lgor et al. [8] focused on the calculation of the effective viscoelastic properties of a short fiber reinforced composite. Chen et al. [9] studied the effects of high-alumina fibers on the strength, crack initiation and propagation of Al/Si refractory materials, and discovered that a lot of micro-cracks could be generated in the matrix when fiber content was up to a certain extent. Sun et al. [10] investigated the quasi-static and dynamic mechanical properties of ceramic fiber reinforced concrete, and the results showed that the compressive strength, tensile strength and bending strength all increased with the increase of fiber volume fraction. Ning et al. [11] prepared zirconium fiber reinforced foam ceramic materials through the organic foam impregnation process, and found that the strength

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of the ceramic foam materials was significantly improved when the content of zirconium fibers was 0.9%-1.4%. Li et al. [12] studied the effects of 2D, 2.5D, 3D braided and 3D needle acupuncture on C/SiC composites. It was found that the interface shear stress and interface debonding energy, as well as the dissipated hysteretic energy and hysteresis width decreased with increasing fiber volume fraction, whereas the hysteresis modulus increased. Unfortunately, the price of these fibers is relatively expensive; worse still, these fibers are easy to be oxidized in high temperature environment.

Alumina fibers are considered to be a better candidate for preparing reinforced composite materials because the melting point of alumina is about 2040 °C and the alumina fiber has high temperature oxidation resistance. In this paper, high-alumina foam ceramics were prepared with aluminum dihydrogen phosphate as inorganic gel and alumina fiber as reinforced phase by the gel-foaming method. The effects of alumina fibers content on the slurry viscosity, as well as the porosity, phase composition, microstructure and mechanical properties of the prepared ceramics were studied.

Experimental

Sample preparation

Alumina foam ceramics were prepared using OP-10 (it belongs to nonionic surfactant and its main ingredient is nonylphenol polyoxyethylene ether) as emulsifier, water as dispersion medium, aluminum dihydrogen phosphate as gels, and alumina fibers as reinforced component. With Al_2O_3 content as the benchmark, alumina powder, aluminum hydroxide powder and alumina fibers were added into the aluminum dihydrogen phosphate solution to obtain four samples in which the content of alumina fibers was 10 wt%, 15 wt%, 20 wt% and 25 wt% respectively. After stirring for 3-5 min, PVA powder (as a foam stabilizer) was added to the mixture, and deionized water was used to adjust the viscosity of the slurry. Then, the mixture was stirred for 2-3 hrs until the slurry volume no longer expanded to form uniform and fine foam. Afterwards, the slurry was casted into a disc-shaped mold with a diameter of 13.5 cm. The mold was stood for 24 hrs at room temperature until the slurry gelled. The green body was oven-dried at 50 °C for about 2 days, and then sintered at 1580 °C for 4 hrs.

Characterization

Phase composition of the sintered samples was analyzed using X-ray diffractometer (XRD, D8 Advanced, Germany) with Cu-$K_{\alpha 1}$ radiation at an accelerated voltage of 40 KV and a scanning step of 0.02 degree. Microstructure was observed with a scanning electron microscopy (SEM, Sirion 200, Holland). Compression strength was tested by an electronic universal testing machine. Open porosity of the samples was measured by the Archimedes method. Thermal conductivity of the samples was determined with a THQDC-1 type thermal conductivity detector. Slurry viscosity was examined by a rotational viscometer. Indentation method was chosen to measure the fracture toughness, which was calculated based on the hardness from a Vickers micro hardness tester.

Results and Discussion

Effect of fiber content on the slurry viscosity

Fig. 1 shows the effect of alumina fiber content on the ceramic slurry viscosity. It can be seen that the ceramic slurry viscosity increases with the increase of fiber content. When fiber content is less than 20 wt%, the slurry viscosity increases slowly and the effect of fiber content on the ceramic slurry viscosity is not obvious, so that the fibers can be well dispersed in the ceramic slurry. With further increase of fiber content from 20 wt% to 25 wt%, the slurry viscosity increases from 1258.5 mPa·s to 3005.3 mPa·s, which can be attributed to the increase of total solid content in the slurry when fiber content exceeds a certain value. This

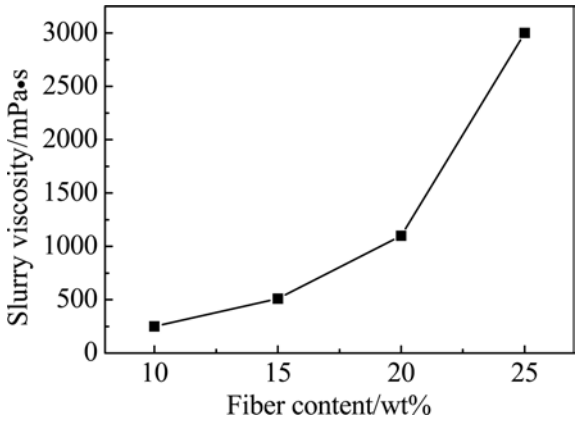

Fig. 1. Effect of the alumina fiber content on the slurry viscosity.

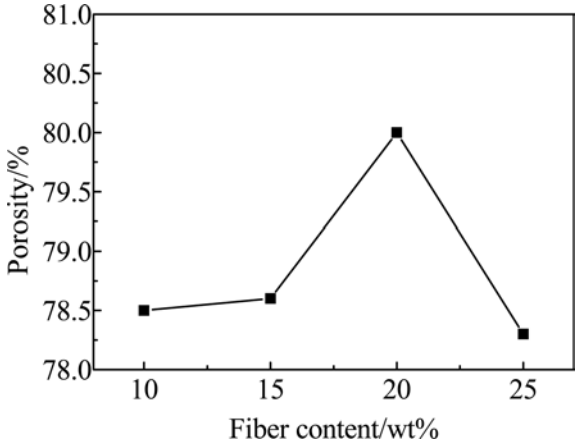

Fig. 2. Effect of the alumina fiber content on the porosity of the prepared ceramics.

rapid increase of slurry viscosity makes it difficult for fibers to disperse in the ceramic slurry, so that the slurry becomes more viscous and the mobility becomes worse.

Effect of fiber content on the porosity of the prepared ceramics

Fig. 2 shows the effect of alumina fiber content on the porosity of the prepared ceramics. When fiber content increases from 10 wt% to 20 wt%, the porosity gradually increases and reaches to the maximum value at the fiber content of 20 wt%. However, when fiber content is greater than 20 wt%, the porosity decreases sharply. This is because that the fibers can form a unique three-dimensional network structure when a large amount of alumina fibers are added. This structure can prevent the shrinkage of the samples, thus decreasing the porosity of the samples during sintering. In combination with Fig. 1, when excessive fibers are added, the slurry viscosity increases dramatically. Thus, the fibers may wrap together because of the lack of space, which destroys the original 3D mesh structure.

Phase composition analysis of the foam ceramics

Fiber content has no significant effect on the phase composition of the foam ceramics because the fibers only account for a small fraction of the total α-Al_2O_3. In this paper, the ceramic samples which the fiber content is 20 wt% were analyzed. Fig. 3 shows the XRD pattern of the ceramic samples after sintering. As can be seen, there are C-$AlPO_4$, T-$AlPO_4$ and α-Al_2O_3 in the samples. Among these, the peak intensity of C-$AlPO_4$ is larger, which means a higher crystallinity. In the high-temperature environment of 1580 °C, $AlPO_4$ exists as the steady phase C-$AlPO_4$, and T-$AlPO_4$ is also transformed to C-$AlPO_4$, so that the crystallinity of C-$AlPO_4$ is high. It is noticed that the peak intensity of α-Al_2O_3 is weaker than that of C-$AlPO_4$ because the relative content of α-Al_2O_3 is less than that of C-$AlPO_4$. However, the diffraction peaks of α-Al_2O_3 are very sharp, which indicates a better crystallinity.

Microstructure analysis of the samples

Fig. 4 shows the microstructure of the alumina foam ceramics with the fiber content of 20 wt%. Fig. 4(a) presents the enhancement mechanism of fiber fracture [13]. As can be seen, the fiber connects the two fracture surfaces as a "bridge". When an external force is applied to the sample, the fibers break to consume the energy, so that the strength of ceramic composites can be improved. Fig. 4(b) presents the enhancement mechanism of microcrack deflection [13]. The propagation of microcracks formed during the forming, drying and sintering of the green body is hampered by alumina fibers when an external force is applied. Along with the increase of this expansion resistance of microcracks, more energy is consumed in the fracture process, thus

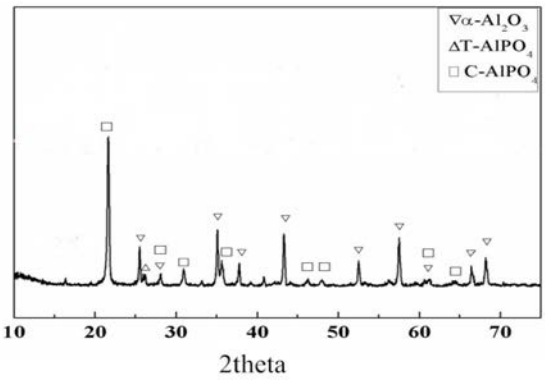

Fig. 3. XRD pattern of the sintered sample.

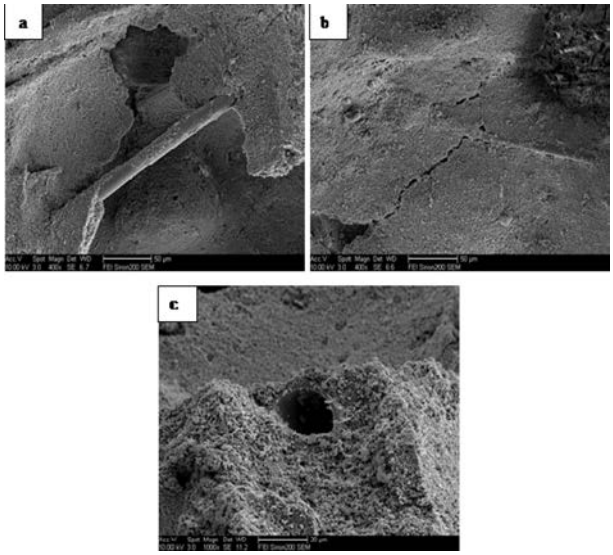

Fig. 4. Microstructure of the sample with the fiber content of 20 wt%.

enhancing the toughness of ceramic matrix composites. Fig. 4(c) presents the image of a hole after a single fiber is pulled out [13]. With the increase of external force, the fibers break and are separated from the base material. This process consumes a lot of energy, which enhances the toughness of the ceramic composites.

Effect of fiber content on the mechanical properties of the foam ceramics

Fig. 5 shows the effect of alumina fiber content on the toughness of the foam ceramics. At the beginning when a small amount of alumina fibers are added, the toughness of the foam ceramics varies little. When alumina fiber content is more than 15 wt%, the addition of alumina fibers can effectively improve the toughness of the foam ceramics. The toughness of the material refers to the ability of absorbing energy during plastic deformation and fracture. After adding fibers, when the foam ceramics break, the fibers in the fracture surface may be pulled out or tightly bond with the matrix to change the direction of microcracks, which leads to the crack deflection. With the increase of fiber content,

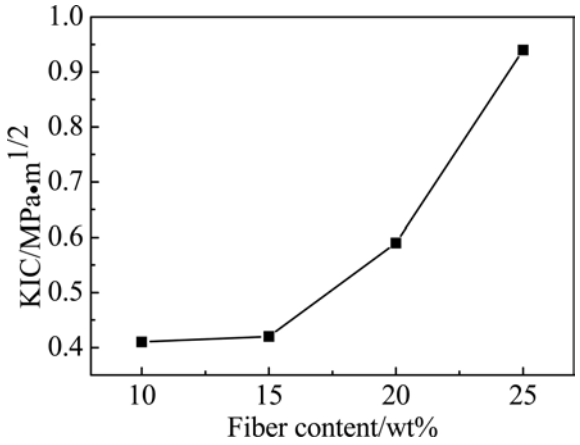

Fig. 5. Effect of the alumina fiber content on the toughness of the foam ceramics.

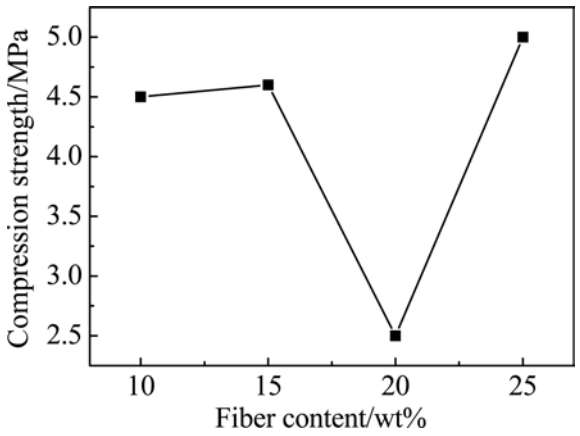

Fig. 6. Effect of the alumina fiber content on the compressive strength of the foam ceramics.

there are more fibers appearing in the cross section, which significantly improves the toughness of the ceramics.

Fig. 6 shows the effect of fiber content on the compressive strength of the foam ceramics. The compressive strength increases slowly with the increase of fiber content from 10 wt% to 15 wt%, and then decreases rapidly from 15 wt% to 20 wt%. With further increase of fiber content, the compressive strength increases sharply. The addition of alumina fibers produces both positive and negative effects on the strength of the foam ceramics. On the one hand, the porosity of the foam ceramics increases due to the formation of the three-dimensional network structure of alumina fibers, but the increase of porosity can lead to the decrease of the compressive strength of the ceramics. Hence, adding alumina fibers can reduce the compressive strength of the ceramics. On the other hand, alumina fibers can enhance the structure of the ceramics, so that the fibers can increase the strength. That is to say, when alumina fibers are added, the effects of increasing the porosity and enhancing the structure of the fibers are coexistent. As a result, the compressive strength of the alumina fiber reinforced alumina foam ceramic is affected by two effects. When alumina fibers are mainly used to improve the porosity, the addition of alumina fibers may reduce the strength of the ceramics; when the fibers are mainly used for reinforcing effect, the addition of alumina fibers can enhance the strength of the ceramics. It can be seen from Figs. 2 and 6 that when alumina fiber content is less than 15 wt%, the reinforcing effect is not obvious; when fiber content is between 15 wt% and 20 wt%, the porosity increases, the compressive strength presents just an opposite variation trend with the porosity; when fiber content is more than 20 wt%, the reinforcing effect of alumina fibers dominates, so that the compressive strength still shows an increasing trend although the porosity decreases.

Conclusions

(1) The slurry viscosity increases with the increase of alumina fiber content. After sintering, there are C-$AlPO_4$, T-$AlPO_4$ and α-Al_2O_3 in the prepared samples.

(2) With the increase of alumina fiber content, the porosity increases first and then decreases, while the compressive strength presents just an opposite variation trend. Due to the bridging effect of alumina fibers, the crack deflection and the fiber pulling-out mechanism, the fracture toughness increases continuously with increasing alumina fiber content.

(3) The addition of alumina fibers produces both positive and negative effects on the strength of the foam ceramics. The compressive strength first decreases with the increase of fiber content, and then increases when fiber content is more than 20 wt%.

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