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Mechanical behavior of fibrous ceramics with a bird's nest structure

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ABSTRACT

Inspired by the structure of the bird's nest, a new fibrous ceramics with mullite fibers as the matrix and SiO_2–B_2O_3 phase as the high-temperature binder was designed and synthesized. The most important structure feature of this fibrous material was that the binder only bonded the fibers at the crossing points. The effects of sintering temperature on the ceramic properties were studied. The fibrous ceramics exhibited significant higher elasticity and pseudoductility compared to the ceramic matrix composites reinforced with continuous fibers. The fracture mechanism of this ceramics under compression was discussed. A high porosity (74.2–78.3%), a low thermal conductivity (0.231–0.248 W/m K), a relatively high compressive strength (1.3–3.2 MPa) and a high rebound-resilience (90–98%) measured from samples indicate that this fibrous ceramics is a potential material for the high-temperature sealing.

1. Introduction

The porous fibrous materials are considered to be a very interesting type of porous materials. On one hand, they are soft, porous, and voluminous. On the other hand, they show a relatively high resistance to mechanical deformation [1]. In addition, the fibrous porous materials require much less material (fiber) to form a stable structure because of the arrangement of the fibers, whereas the granular porous media require much more material (granules) [1–4]. Therefore, they are applied in numerous and diverse aspects such as catalyst supports, hot-gas filters, composite reinforcement, biomaterials, acoustic and thermal insulation and sealing materials [5].

Contrary to the monolithic ceramics which exhibit a brittle fracture, porous ceramics present a dissipative damage tolerant behavior [6–8]. Numerous research articles were published focusing on forming porous ceramics with special 3-D skeleton structures, such as the foams and cellular materials [9–12]. Besides, another branch of research works is devoted to developing new structures such as continuous fiber-reinforced matrix and multilayer laminates which could provide a high strength along the fiber direction but a weak strength across the fiber direction [13–16]. However, none of these materials satisfies the quasi-plastic and elastic requirement of sealing materials due to the inherent brittleness [17,18]. The aim of our study is to design a new fibrous material with both high strength and attractive compression–rebound property [19–21].

Highly porous fiber network materials are abundant in nature and in man-made environments. One convincing example is the bird's nest which is made of randomly arranged tree branches. Inspired by this fact, the idea of designing a fiber matrix porous ceramic with fibers as the skeleton structure bonded by proper binders at the crossing points was brought.

Mullite fiber has a good flexibility at relatively high stress, making it a promising candidate for preparing the sealing material [22,23]. In this research a new fibrous ceramic with the framework structure of polycrystalline mullite fibers was fabricated by infiltration method. During the preparation process, the organic and inorganic binders were impregnated in the fiber framework in order to bond the fibers with each other at room temperature and high temperature, respectively. The elasticity of FCFMF sintering at 1200 °C was investigated [24]. The study showed that the sample possessed a high degree of rebound resilience (98%) under a compression stress of 2 MPa. The present study is a continuous work of this previous study [24] and the effects of the sintering temperatures on the microstructure and physical and mechanical properties of the samples were analyzed.

2. Experiment

2.1. Raw materials

Commercially available polycrystalline mullite refractory fiber (PMF, 99.5%, Zhejiang Hongda Crystal Fiber Co., Ltd., China) was used as the starting material in this study. The organic binder (OB) was prepared by mixing sodium dodecyl benzene sulfonate (SDBS) with polyacrylamide (CPAM) in water with the weight ratio

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SDBS:CPAM:H_2O = 1:1:100. The inorganic binder (mixed sol) was made of silica sol and boric acid, with a final molar ratio of SiO_2 to B_2O_3 of 10:1. The silica sol was produced with the use of tetraethylorthosilicate (TEOS, AR grade, Tianjin Kewei Chemical Co., China) by one-step catalytic method. The weight ratio was TEOS:H_2O:ethanol:hydrochloride = $1:4:1:7.5 \times 10^{-4}$. The boric acid was obtained by dissolving the solid H_3BO_3 in the water at room temperature with a weight ratio of H_3BO_3:H_2O = 1:18.

Fig. 1(a) shows the micrograph of the mixed gels used as inorganic binder. The diameter of the mixed gels is around 5 μm and the shape is approximately ellipse. Fig. 1(b) presents the micrograph of the mullite fibers. It can be seen that the fiber possessed a diameter in the range of 8–15 μm and a length in the range of 300–500 μm.

2.2. Experimental procedure

Fig. 2 shows the processing steps of ceramic composites preparation and the schematic diagram of the structure of the FCFMF. First, the organic binder (30 g) and PMF (6 g) were mixed together by stirring (Fig. 2(a)). The fibers formed into a fiber block by infiltration with the help of a certain amount of organic binder coated on the fiber surface (Fig. 2(b)). After infiltration, the fiber surface became negatively charged due to the formation of the negatively charged organic coating. Then the mixed sols were impregnated into the fiber block. Since the fiber surface and the mixed sols were both negatively charged, there was a great repulsion between the fiber surface and the mixed sols. Therefore, the mixed sols coated on the fiber surface can be easily taken away by infiltration. However, the mixed sols at the crossing points of the fibers were forced to stay in the original place after infiltration because of the big obstacles at the crossing points of the fibers (Fig. 2(c)). The green bodies were sintered at different temperatures from 1100 to 1400 °C for 2 h assisted by holding at 600 °C for 0.5 h to decompose the organic phase. After sintering, the organic binder was burned out and the mixed sols melt into continuous phase at the crossing points of the fibers, consequently acting as a high-temperature binder (Fig. 2(d)). The mullite fibers with the bonding points constituted a special 3-D skeleton structure.

2.3. Characterization

Phases were analyzed via X-ray diffraction (XRD, D/Max-2500 Rigaku, Japan). Microstructure of the sintered samples was observed by scanning electron microscope (SEM, XL-30Philips, Japan). Open porosities and densities of the sintered samples were determined by Archimedes method namely the water-immersion technique. The linear shrinkage of samples during drying and sintering process was determined by the following equation: shrinkage = $[(l_a - l_b)/l_a] \times 100\%$, where l_a and l_b were the diameter of initial samples and dried or sintered samples, respectively. Compression ratio and rebound resilience tests of the FCFMF were carried out at room temperature on an electro-universal testing machine (Instron 5569, USA) in accordance with GB/T 1964–1996. During the test a set of loads were applied to the samples at a loading speed of 0.05 mm/min, and removed at an unloading speed of 0.05 mm/min. The compressive ratio and the rebound resilience were determined by the following equations: compressive ratio = $[(t_0 - t_1)/t_0] \times 100\%$, rebound-resilience = $[(t_2 - t_1)/(t_0 - t_1)]$, where t_0, t_1 and t_2 are the height of preloading, loading and unloading, respectively. Thermal conductivity at room temperature was measured by the thermal-conductivity instrument (C-3000, Xian Xiaxi Electric Co., Ltd., Shanxi, China). The dimensions of measured samples were 30 mm in diameter and 5 mm in height. Each value represented an average of five measurements of five different specimens.

3. Results and discussion

3.1. The typical structure of the FCFMF

Fig. 3(a) shows the typical SEM image of the FCFMF. After sintering, the organic binder was removed completely, and the fibers lapped with each other forming a loose skeleton structure. It could be clearly seen that the fibers were randomly arranged in the block providing the ceramics with isotropic mechanical properties. Meanwhile, the adjacent fibers were bonded by the surrounding melted SiO_2–B_2O_3 binders at the crossing points which endowed the fibrous ceramics with relatively high strength. Fig. 3(b–d) shows the typical bonding points in the fiber block. This special bird's nest structure provides this material with unique properties, which were discussed below.

3.2. Phase characterization

The sintering temperature plays an important role in the state of SiO_2–B_2O_3 among fibers and the wettability between the SiO_2–B_2O_3 and the mullite fiber, thus determining the properties of the samples.

Fig. 4 shows the XRD patterns of the green body and samples sintered at different temperatures. A wide peak was observed in the green body and the peak of mullite was not very strong, which suggest that the fibers were covered by the organics. When the temperature was below 1200 °C, the patterns of the sintered bodies composed of mullite characteristic peaks and a broad background which indicates that the silica–boron gel transformed to the glassy phase. However, when the sintering temperature was above 1200 °C, the patterns of the sample exhibited the α-cristobalite characteristic peaks besides the mullite characteristic peaks. With the temperature increasing from 1200 to 1400 °C, the intensity of cristobalite increased gradually. The existence of the α-cristobalite characteristic peaks proved that silica-based glassy phase crystallized into the β-cristobalite at 1300 °C and then the β-cristobalite underwent a β → α displacive phase transition when

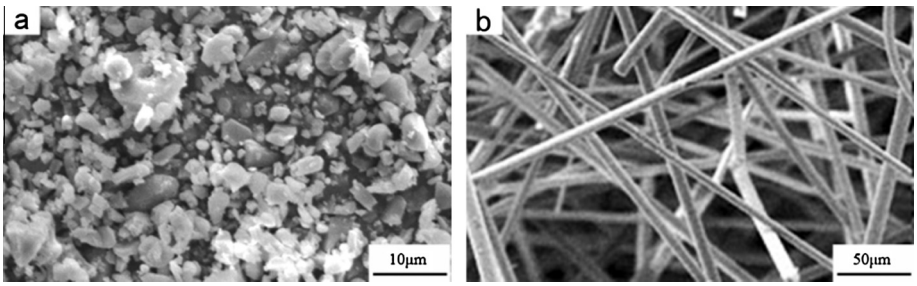

Fig. 1. The micrograph of (a) the silica–boron gels used as binder and (b) the fibers.