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Short communication

High-temperature elasticity of fibrous ceramics with a bird's nest structure

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Abstract

New fibrous ceramics with polycrystalline mullite fibers as the matrix and silica–boron sols as the high temperature binder, which was inspired by the bird's nest structure in nature, were synthesized. The most important structure characteristic of this fibrous material is that the silica–boron binder only fixed the fibers at the crossing points rather than filled the pores among the fibers. The elastic behavior was investigated, both at room temperature and elevated temperature. Compared to conventional ceramic matrix composites, the samples show a much higher degree of elasticity because of the bending of the fibers. The rebound resilience decreased slowly with the increase of the temperature, but it still remained 86% of that at ambient temperature at 1000 °C. The sample exhibits good elasticity performance, relatively high strength (2.25 MPa) and high porosity (83%) indicating it is a potential high-temperature seal material.

Keywords: Mullite fiber; Porous ceramics; Microstructure; Compressive strength; High-temperature elasticity

1. Introduction

In recent years, high temperature sealing problem has received much attention in the fields of aerospace and energy.[1,2] In order to seal the gap between the components effectively, the sealing materials must possess good elastic property. Elastic materials have been widely used in the fields of mechanical engineering due to the rapid recovery to its approximate initial shape and dimensions after substantial deformation by force and subsequent release of that force.[3,4] It is well known that the traditional study of elastic behaviors mainly focuses on the metallic materials and polymers; however, neither of these materials can be used in high-temperature environments.[5,6]

Ceramics exhibit many intrinsic properties which make them promising high-temperature structural materials.[7–9] Related reports show that the ceramic matrix composites reinforced with continuous fibers present a dissipative damage tolerant behavior, contrary to the monolithic ceramics which exhibit a brittle fracture.[10–12] However, in the system of the ceramic matrix composites reinforced with continuous fibers, all the introduced fibers were embedded in the ceramic matrix and were consequently constrained by the matrix. This dense structure results in small deformation of the sample under a certain stress, which means that this kind of material system exhibits poor elasticity.[13,14]

It is well known that the bird's nest structure in nature, consisting of randomly arranged tree branches, is characterized by high compression ratio. Inspired by this, a new porous material with a bird's nest-like structure was designed in this paper. Polycrystalline mullite fiber (PMF) is an attractive alternative which is applied at high temperature in oxidizing environment because of the combination of high thermal stability, high mechanical strength, good chemical stability and creep resistance.[15–18] In this experiment, 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 into the fiber framework in order to bond the fibers

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with each other at room temperature and high temperature, respectively.

In this system of porous PMF, the idea was to fix the fibers by the binders only at the crossing points rather than filled the pore among the fibers, which allows the fibers bending in a certain range under the pressure and recovering to its approximate initial shape after removing the load. The compression rebound characteristics of the samples were investigated at different temperatures (from room temperature to 1000 °C).

2. Experimental procedure

2.1. Raw materials

Commercially available polycrystalline mullite fibers (99.5%, Zhejiang Hongda Crystal Fiber Co., Ltd., China) were used as starting materials. The organic binder (OB) was prepared by mixing sodium dodecyl benzene sulfonate (SDBS) and polyacrylamide (CPAM) in water with weight ratios SDS:CPAM:H_2O = 1:1:100. The inorganic binder (mixed sol) was composed of silica sol and boric acid, with a weight ratio of Si to B of 10:1. The silica sol was produced with the use of tetraethylorthosilicate (TEOS, AR grade, Tianjin Jiangtian Chemical Co., China) by one-step catalytic method and the weight ratio was TEOS:H_2O:ethanol:hydrochloride = $1:4:10:7.5 \times 10^{-4}$. The boric acid was obtained by dissolving the solid H_3BO_3 in the water and the weight ratio was H_3BO_3:H_2O = 1:18. All chemicals used in this study were analytical (AR) grade.

2.2. Process

The process of bird's nest structures from the PMF was illustrated in Fig. 1(a)–(d). First, the fibers (6 g) were mixed with the organic binder (30 g) by stirring, and after infiltration the fibers formed into the fiber block with a dimension of 30 mm in diameter and 15 mm height (Fig. 1(a) and (b)). Then the mixed sols were impregnated into the fiber block (Fig. 1(c)). The content of the mixed sols (characterized by the SiO_2–B_2O_3 content) relative to the weight of PMF was 15 wt%. The green bodies were sintered at 1200 and 1300 °C for 2 h with a heating rate of 5 °C/min, assisted by holding at 600 °C for 1 h to decompose the organic phase. After sintering, the organic binder burned out and the mixed sols melt into a glass phase at the crossing points of the fibers, consequently acting as necks at high-temperature (Fig. 1(d)). In Fig. 1(e)–(g), the process of the compression-rebound test is exhibited.

2.3. Characterization

Differential scanning calorimetry and thermogravimetry (DSC/TG) of a green body were conducted from ambient to 1300 °C at a heating speed of 10 °C/min in flowing air with a thermal analyzer (Netzsch Sta 449C, Germany), in order to follow up transformations occurring during the heating of the samples. 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 the water-immersion technique using the Archimedes method. The compressive strength was measured by a universal testing machine (XWW, Beijing Shengxin Detecting Instrument, China) with a loading speed of 0.05 mm/min in accordance with GB/T 1964-1996. Compression ratio and rebound resilience tests of the porous PMF were carried out at room temperature and high temperature on an electro-universal testing machine (Instron -5569, USA) in accordance with GB/T 1964-1996. During the test, a load of 2 MPa was first applied to the sample with a loading speed of 0.05 mm/min, and then removed with an unloading speed of 0.05 mm/min. The compressive ratio and the rebound resilience were determined by the following equations: compressive set = $[(t_0 - t_1)/t_0] \times 100\%$, 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.

3. Results and discussion

3.1. Thermal analysis and phase characterization

Fig. 2(a) shows the TG and DSC curves of the green body. It can be seen that the weight loss took place in the following three ranges. The first weight loss of 2.5% was attributed to the dissociation of the absorbed water and ethanol from 60 °C to 100 °C. The second weight loss (about 0.2%) which resulted from the decomposition of H_3BO_3 occurred between 100 °C and 300 °C. The third weight loss (about 0.8%) attributed to the organic phase of the fibers arised between 400 °C and 700 °C. It should be noted that the second weight loss ranges (100–300 °C) and the third weight loss (500–600 °C) were together with a broad endothermic peak which was related to the oxidation of the –OR and –OH bonds of the mixed gels and the removal of the organic binder on the surface of the fibers. Moreover, there is a broad endothermic peak (from 700 °C to 1200 °C) on the DSC curve, which was caused by the melting of SiO_2–B_2O_3 gels.

Fig. 2(b) shows the diffractograms of the sintered bodies after heating at 1200 and 1300 °C. The patterns of the sintered bodies heated at 1200 °C are composed of mullite characteristic peaks and a broad background. The background indicates that the silica–boron gel turned into the glassy phase after heating at 1200 °C. The patterns of the sample heated at 1300 °C exhibit the α-cristobalite characteristic peaks. It meant that silica-based glassy phase crystallized into the β-cristobalite at 1300 °C and then β-cristobalite underwent a β → α displacive phase transition when the temperature decreased to room temperature.[19,20] This transition was accompanied by 3–5% volume change, which often generated thermal stresses and resulted in a cracking of the binder.

According to the analysis of DSC curve and XRD, the sintering temperature of the sample was determined to be 1200 °C for the following tests.