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High-temperature bending strength, internal friction and stiffness of ZrB_2–20 vol% SiC ceramics

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Abstract

Dense ZrB_2–20 vol% SiC ceramics (ZS) were fabricated by hot pressing using self-synthesized high purity ZrB_2 and commercial SiC powders as raw materials. The high temperature flexural strength of ZS and its degradation mechanisms up to 1600 °C in high purity argon were investigated. According to the fracture mode, crack origin and internal friction curve of ZS ceramics, its strength degradation above 1000 °C is considered to result from a combination of phenomena such as grain boundary softening, grain sliding and the formation of cavitations and cracks around the SiC grains on the tensile side of the specimens. The ZS material at 1600 °C remains 84% of its strength at room temperature, which is obviously higher than the values reported in literature. The benefit is mainly derived from the high purity of the ZrB_2 powders.

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Keywords: ZrB_2–SiC; UHTCs; High temperature strength; Internal friction; Crack origin; Grain sliding

1. Introduction

ZrB_2 is one of the ultra-high temperature ceramics (UHTCs), which are of considerable interest for applications that require exposure to extreme thermal and chemical environments such as the leading edge components and thermal protection systems in future hypersonic aerospace vehicles (7–20 Mach).[1–3] In order to improve the oxidation resistance and mechanical properties of ZrB_2, silicon carbide (SiC) is commonly used as an additive. ZrB_2–20 vol% SiC particulate composite (ZS) is regarded as the baseline materials in this research.

The densification process,[4–6] microstructure tailoring,[7–9] oxidation/ablation resistance,[1,10,11] thermo-physical[12] and mechanical properties of ZrB_2–SiC composites[13] have been extensively investigated in the last 10 years. The flexure strength of ZrB_2–SiC ceramics at elevated temperatures is an important factor for the applications in the extreme environment. However, there lack data of the high temperature flexure strength and fracture behavior of ZrB_2–SiC composites.

A number of previous studies[14,15] only reported some strength values of ZS material at a limited range of temperatures, but strength degradation mechanisms are not available; Bellosi et al. evaluated the strengths of ZrB_2–10 vol% SiC ceramics fabricated from commercial ZrB_2 and SiC powders with or without ZrC additions at 1500 °C in argon or air atmosphere. The results indicated that the loss of strength of the ZrB_2–10 vol% SiC ceramics was not obvious when the samples sintered by Spark Plasma Sintering (SPS). The favorable effects were mainly attributed to the clean grain boundaries in the as-SPSed samples.[16] However, their samples in the experiments were partially oxidized during the measurements. In addition, Hu[17] reported that the flexure strengths and behavior of ZrB_2 with 15 vol% and 30 vol% SiC additions at 1800 °C were controlled by the ZrB_2 and SiC grain sizes in the ZrB_2–SiC particulate composites. The fracture strength of the ZrB_2–15 vol% SiC composites at 1800 °C decreased from 217 MPa for coarse

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grains (ZrB_2 5 μm, SiC 2 μm) and to 112 MPa for fine grains (ZrB_2 2 μm, SiC 0.5 μm), and meanwhile the fracture behavior transformed from elastic fracture to plastic deformation. In Hu's studies, the authors only reported the fracture strengths at room temperature and 1800 °C, and did not offer any data concerning the temperature dependence of strength and strength degradation mechanisms.

Softening of grain boundary at elevated temperatures leads to strength degradation of ceramics, especially when low melting point phases exist at the grain boundaries. In order to minimize the grain boundary phases with low melting point and improve the high temperature strengths of ZS material, high purity ZrB_2 powders synthesized through a carbon-thermal/boron-thermal reduction method were used in the present research. The bending strength and Young's modulus (E) of ZS material at various temperatures were measured in a high purity argon atmosphere, i.e. the tested bars almost would not be oxidized. The effect of grain boundary characters on the strength loss at elevated temperatures was studied by high-resolution transmission electron microscopy (HRTEM) observations together with internal friction analysis. The strength degradation mechanisms of ZS were comprehensively investigated and discussed.

2. Experimental procedure

The ZrB_2 powder was synthesized by the reaction between ZrO_2 and B_4C at 1600 °C in vacuum. A similar synthesis procedure could be found as for HfB_2 powder.[18] The crystalline phase, impurities content, average particle size and suppliers of ZrB_2 and SiC powders are listed in Table 1. Apart from Hf, the total impurities (0.6 wt%) and the oxygen content (0.5 wt%) in the as synthesized ZrB_2 powder are obviously lower than those in commercial ZrB_2 powders.[12,16,17] Furthermore, the carbon content in ZrB_2 powders is as low as 0.10 wt%, i.e. the residual boron carbide in the as-synthesized ZrB_2 powder is limited. The ZrB_2 and SiC powders were mixed with acetone in a resin jar for 12 h using Si_3N_4 media. Then the slurry was dried and sieved. The compacts of the mixed raw powders were hot pressed at 2000 °C and at 30 MPa for 1 h in a flowing argon atmosphere. The processing details and densification procedures have been reported in our previous reports.[19]

Three-point bending tests were performed at temperatures in the range from ambient temperature to 1600 °C and at the cross-head speed of 0.5 mm/min. The dimension of the specimens was 2 mm × 2.5 mm × 25 mm (thickness × width × length). The tensile surfaces of the specimens were polished to a 1.5 μm diamond finish and chamfered. Then, the polished specimens were placed on a graphite bend ring in a furnace equipped with tungsten mesh heating elements. The total pressure in the furnace was vacuumed to $<10^{-3}$ Pa by a diffusion pump, followed by introducing high purity argon (>99.999%) at flowing rate of 100 ml/min to keep a high oxygen-free environment in the furnace and to prevent the test bars from oxidation during measurements. The bars were soaked at the testing temperatures for 20 min to reach a thermal equilibrium. The reported flexure strengths are the average of five specimens at room temperature and of three specimens at high temperatures. The microstructures of ZS before and after testing were observed by scanning electron microscopy (SEM, TM3000, Hitachi, Japan) and HRTEM (JEM 2100, JEOL, Japan) equipped with an X-ray energy dispersive detector systems (EDS, Model Link-ISIS, Oxford Instruments, UK).

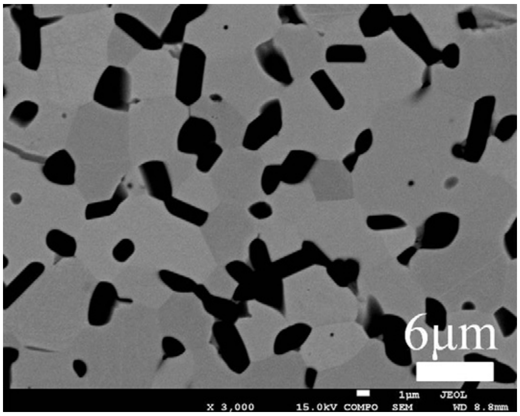

Fig. 1. Microstructure of ZrB_2–20% SiC ceramics hot pressed sintered at 2000 °C for 1 h in argon.

The Young's modulus (E) and internal friction (Q^{-1}) of the ZS specimens with the dimension of 5 mm × 1.5 mm × 40 mm were measured by an impulse excitation technique (HTVP-1750-C, IMCE, Diepenbeek, Belgium) in argon atmosphere at temperatures from 25 °C to 1300 °C with a heating rate of 2 °C/min as previously described.[20,21]

3. Results and discussions

3.1. General microstructure

The microphotograph of ZS shown in Fig. 1 indicates that the average particle size of ZrB_2 grains is 6.1 ± 1.4 μm, plate-like SiC grains with the average length of 5.3 ± 2.1 μm and width of 1.1 ± 0.8 μm are homogenously dispersed in the ZrB_2 matrix. It should be noted that the color contrasts of some small regions are different from those of ZrB_2 and SiC. It is very hard to identify these phases simply by SEM. According to the detailed microstructural analysis of ZS material conducted by TEM, two secondary phases have been found, and the representative ones are shown in Fig. 2. The first is crystallized hexagonal boron nitride (Fig. 2a), the other one with the composition of Zr–Ca–(Al)–Si–O (Fig. 2b and c, sometimes Al could not be detected from EDS) is an amorphous phase, as confirmed by its electronic diffraction pattern (Fig. 2d). The formation of BN should result from the reaction between B_2O_3 (from starting powders) and Si_3N_4 (incorporated with the milling balls) through reaction (I),[22] while the Zr–Ca–(Al)–Si–O phase presumably comes from the impurities in the starting powders as well as the milling balls (Si_3N_4 with CaO and Al_2O_3 additions). However, these impurities are very difficult to find during TEM observation, indicating that their amounts in ZS ceramics are extremely low, which is in accordance with the element content analysis of ZrB_2 and SiC powders.

$$2B_2O_3 + Si_3N_4 = 4BN + 3SiO_2 \quad \text{(I)}$$

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