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Mechanical properties of laminated ZrB_2–SiC/SiC_w ceramics

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The mechanical properties of ZrB_2–SiC/SiC_w layered ceramics obtained by strip casting and hot pressing are studied. In the ceramics, ZrB_2–SiC layers and ZrB_2–SiC_w layers alternate. The bending strength and fracture toughness of the ceramics can reach 360 MPa and 10.83 MPa·m$^{1/2}$, respectively. It is shown that, compared with ZrB_2–SiC monolithic ceramics, the improvement in fracture toughness is explained by SiC whiskers and layered structure.

Keywords: laminated structure, ZrB_2, SiC_w, mechanical properties.

Исследованы механические свойства слоистой керамики ZrB_2–SiC/SiC_w, полученной методом литья ленты и горячего прессования. В керамике слои ZrB_2–SiC и слои ZrB_2–SiC_w попеременно чередуются. Прочность на изгиб и вязкость разрушения керамики может достигать 360 МПа и 10.83 МПа·м$^{1/2}$ соответственно. Показано, что по сравнению с монолитной керамикой ZrB_2–SiC улучшение вязкости разрушения объясняется усиками SiC и слоистой структурой, которые полезны для отклонения трещин, разветвления трещин и вытягивания усов.

Механічні властивості шаруватої кераміки ZrB_2–SiC/SiC_w. *Yupeng Xie, Yuxiang Wang, XianDe Wang.*

Досліджено механічні властивості шаруватої кераміки ZrB_2–SiC/SiC_w, отриманої методом лиття стрічки і гарячого пресування. У кераміці шари ZrB_2–SiC і шари ZrB_2–SiC/SiC_w поперемінно чергуються. Міцність на вигин і в'язкість руйнування кераміки може досягати 360 МПа і 10,83 МПа·м$^{1/2}$ відповідно. Показано, що в порівнянні з монолітною керамікою ZrB_2–SiC поліпшення в'язкості руйнування пояснюються вусиками SiC і шаруватою структурою, які корисні для відхилення тріщин, розгалуження тріщин і витягування вусиків.

1. Introduction

Zirconium diboride (ZrB_2), as a member of ultra-high temperature ceramics (UHTCs), has the lowest theoretical density (6.09 g/cm^{-3}), high melting point (3245°C), high hardness (23 GPa), high thermal conductivity, and excellent chemical stability, making it a promising material for high temperature structural applications [1–3]. However, ZrB_2 ceramics shows low fracture toughness, poor thermal shock resistance and poor sinter ability due to its intrinsic characteristics, resulting in limitation of its applications [4, 5]. Introduction of a second phase to the ZrB_2 matrix can effectively improve its mechanical properties and antioxidant properties [6–10]. Among the ZrB_2-based ceramics, ZrB_2–SiC ceramics have been paid more attention to studies [8, 11]. In spite of many excellent properties of the ZrB_2–SiC ceramics, the inherent brittleness of the ZrB_2–SiC ceramics limits its application. A laminated structure is considered as an effective way to improve fracture toughness of the ZrB_2-based ceramics [12]. Compared with conventional approaches, the laminated structure can consume more energy as the crack deflected and lead to residual stress between layers. The laminated ZrB_2–SiC ceramics with weak interface (graphite and boron nitride) have been reported in many works, which obtain good fracture toughness [13, 14]. However, the poor oxidation resistance of the weak interface limits their application at high temperature. In order to solve the above problem, the laminated ZrB_2–SiC ceramics with strong interface have been studied [11, 15–17]. Especially, the laminated ZrB_2–SiC ceramics with SiC whiskers (SiC_w) not only increase oxidation resistance, but also improve fracture toughness.

In the present work, the samples of the laminated ZrB_2–SiC/SiC_w ceramics were successfully prepared using tape casting and hot-pressing processes. The microstructure and mechanical properties were investigated. The toughening mechanisms were discussed.

2. Experimental

ZrB_2 powders (average particle size of 0.8 μm, purity of 99.9 %, Shanghai Shuitian Co., Ltd, China), SiC powders (0.5 μm, 99.9 % purity, Shanghai Shuitian Co., Ltd, China), and β-SiC whiskers (average length of 18 μm and diameter of 1.5 μm, 99 % purity, Alfa Aesar, MA, USA) were used as raw materials. AlN powders (50 nm, purity 99.0 %, Qinhuangdao Eno High-Tech Material Development Co., Ltd., China) were used as the sintering aid. Ethyl alcohol and acetone were used as a solvent. Triethyl phosphate (TEP) was used as a dispersant. PVB, glycerol and di-*n*-octyl phthalate (DOP) were used as a binder and a plasticizer, *n*-butanol and ethylene glycol were used as a defoamer.

ZrB_2-20 vol.% SiC and ZrB_2-35 vol.% SiC_w slurries were prepared by mixing the powders with TEP and then dispersing the mixture in the solvent. The slurries were ball-milled for about 12 h by using zirconium ball as a grinding medium followed by adding PVB, glycerol, DOP, *n*-butanol and ethylene glycol. After adding other chemical agents, the slurries were ball-milled for another 12 h to obtain well-dispersed slurries with good fluidity. The slurries were hold in vacuum to remove gas bubbles. The casted sheets were dried naturally, and stacked alternately with ZrB_2-20 vol.% SiC layers and ZrB_2-35 vol.% SiC_w layers until the desired compositions were obtained. The

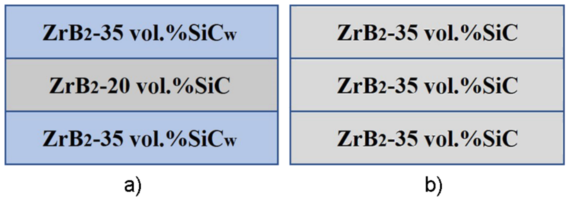

Fig. 1. Schematic illustration of the laminated structure of (a) ZSW and (b) ZS.

monolithic ZrB_2-35 vol.% SiC ceramics were prepared using the same composition and processing as the laminated ZrB_2–SiC sheet. The schematic illustration of the laminated ceramics and monolithic ceramics is shown in Fig. 1. The sintering aid was 5 vol.% AlN. The stacked bodies were pressed by cold isostatic pressing at room temperature. The pressed bodies were sintered at 600°C for 2 h to remove the binder, and then were sintered at 1950°C for 1 h under a uniaxial load of 30 MPa in Ar atmosphere. The laminated ZrB_2–SiC/SiC_w ceramics and monolithic ZrB_2–SiC ceramics were respectively marked as ZSW and ZS.

The bulk density was measured by Archimedes' method. The flexural strength of the samples 3×4×22 mm^3 was tested by three-point bending method with a span of 30 mm and a crosshead speed of 0.5 mm/min. The fracture toughness of the samples 2×4×22 mm^3 was evaluated by a single-edge notched beam (SENB) test with a 16 mm span and a cross-head speed of 0.05 mm/min on the same jig used for the flexural strength.

The microstructures were analyzed with a scanning electron microscope (SEM, S-4700, Hitachi, Japan).

3. Results and discussion

3.1 Microstructures

Fig. 2 shows the cross-section morphologies of the ZSW samples. As shown in Fig. 2a, the ZrB_2–SiC layers and ZrB_2–SiC/SiC_w layers were alternately arranged. There were no obvious defects on the surface, indicating that the material was densified. The thickness of the ZrB_2–SiC layers and ZrB_2–SiC/SiC_w layers was about 60 μm. From the magnified image of the ZrB_2–SiC layer and ZrB_2–SiC/SiC_w layer (Figs. 2b and 2c), SiC particles and SiC whiskers were homogeneously dispersed in the ZrB_2 matrix and well combined with the matrix. Holes can be clearly observed due to the high frac-

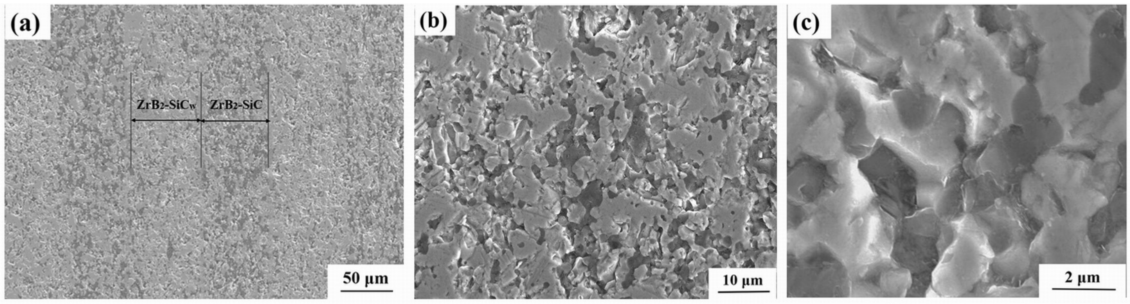

Fig. 2. SEM micrographs of ZSW samples: (a) cross-sectional; (b) low magnification of a ZrB_2–SiC layer; (c) higher magnification of a ZrB_2–SiC_w layer.

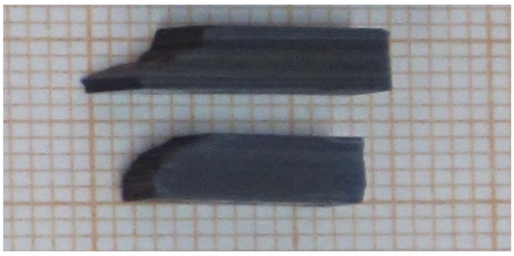

Fig. 3. Crack propagation in ZSW and ZS samples.

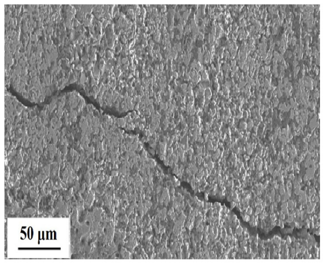

Fig. 4. SEM image of crack propagation in the ZrB_2–SiC_w layer.

tion of the SiC whiskers which were difficult to be fully densified, as shown in Fig. 2c.

The crack propagation paths in the ZS and ZSW samples after the three-point test are depicted in Fig. 3. A zigzag crack propagation path was observed in the ZSW sample. The crack propagation was vertical in the ZrB_2–SiC layer, and then the crack deflected in the ZrB_2–SiC/SiC_w layer due to the effect of the SiC whiskers. The vertical and horizontal cracks propagated repeatedly until the sample fractured. In addition, the crack branching was also observed in the ZrB_2–SiC/SiC_w layer, as shown in Fig. 4. The crack deflection and branching significantly decreased the crack propagation; this was beneficial for improvement of toughness.

3.2 Mechanical properties

The fracture surfaces of the ZSW samples are shown in Fig. 5. The SiC whiskers are observed and holes resulting from whisker pull-out are also detected, as shown in Fig. 5a. Furthermore, some whiskers are fractured during the test, indicating a strong interface bonding between the whisker and matrix. It can be seen from Fig. 5b that the fracture mode is mixed transgranular and intergranular. The whisker pull-out and crack deflection around the whisker (Fig. 5c) can be observed in the fractured surface of the ZSW sample, leading to increasing the fracture energy and improving toughness.

The bulk density, relative density, flexural strength and fracture toughness of the ZS and ZSW samples are listed in Table. It can be seen that bulk densities of ZS and ZSW samples were 5.12 $g \cdot cm^{-3}$ and 5.09 $g \cdot cm^{-3}$, which corresponded to the relative densities of 99.4 % and 98.9 %, for ZS and ZSW samples, respectively. The relative density of the ZSW sample was lower than that of the ZS sample, indicating that the pores existed in the layer; this corresponds to the results of Fig. 2 and Fig. 5. As shown in Table, the fracture toughness of the ZSW sample was higher than that of the ZS sample, owing to the multiple toughening mechanisms.

4. Conclusions

Laminated ZrB_2–SiC/SiC_w ceramics were successfully prepared by tape casting and hot-pressing processes. The ZSW was alter-

Table. Mechanical properties of ZSW and ZS samples

Samples	Flexural strength, MPa	Fracture toughness, $MPa \cdot m^{1/2}$	Bulk density, $g \cdot cm^{-3}$	Relative density, %
ZSW	360±23	10.83±0.43	5.09	98.9
ZS	455±34	4.20±0.26	5.12	99.4

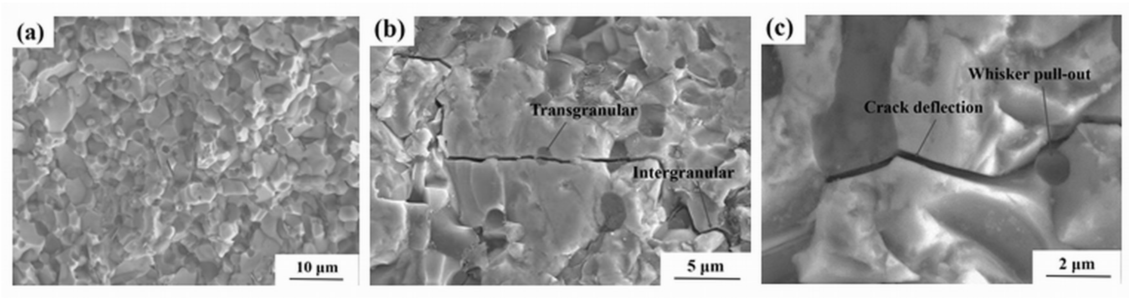

Fig. 5. SEM micrographs of fracture surfaces of ZSW samples: (a) whisker pull-out; (b) fracture mode and (c) crack deflection.

nately composed of ZrB_2-20 vol.% SiC layers and ZrB_2-35 vol.% SiC_w layers with sintering aid of 5 vol.% AlN. The fracture toughness of the ZSW was effectively improved, owing to the crack deflection, crack branching and whisker pull-out.

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