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Mechanical Properties and Interfacial Microstructure of SiC Whisker-Reinforced ZrO_2–Y_2O_3 Composites

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Abstract: Mechanical properties and fracture mechanisms of 20 vol% SiC whisker-reinforced ZrO_2 (Y_2O_3) composites were investigated. The phase composition, fractography and the microstructure of the whisker–matrix interface were studied by XRD, SEM, TEM and HREM. The results show that flexural strength and fracture toughness of SiCw/ZrO_2 (with 2 mol% Y_2O_3) composites increase by 453 MPa and 2·6 MPa $m^{1/2}$, respectively. However, in the composites with 6 mol% Y_2O_3, the increment is only 17 MPa and 1·6 MPa $m^{1/2}$, respectively. It reveals that the ZrO_2 (2 mol% Y_2O_3) composites have a good toughening effect by incorporating SiC whiskers. HREM observations indicate that the whiskers in ZrO_2 (2 mol% Y_2O_3) composites are directly bonded with the ZrO_2 matrix, only few atom disorder zones exist at the interface. In contrast, for ZrO_2 (6 mol% Y_2O_3) composites, there is a thin, uniform layer of amorphous phase at the interface between SiC whisker and ZrO_2 matrix, indicating that high Y_2O_3 content promotes the formation of an interfacial layer. The study of fracture surfaces by SEM indicates that the main toughening mechanisms in SiCw/ZrO_2 (with 2 mol% Y_2O_3) composites are crack deflection, crack bridging, whisker pull-out and dynamic t→m transformation. Crack deflection is the main toughening mechanism in SiCw/ZrO_2 (with 6 mol% Y_2O_3) composites.

1 INTRODUCTION

A whisker is one of the main reinforcements incorporated into a ceramic matrix. SiC whisker-reinforced ceramic composites have superior properties at room and elevated temperatures,[1,2] and thus have an advantage over transformation toughening. The effect of toughening (such as crack deflection, whisker pull-out and bridging) is sensitively influenced by the microstructure of the composite, and particularly by the characteristics of the whisker–matrix interface.[3,4] The object of this study is to assess the microstructure of SiCw/ZrO_2 (with 2 or 6 mol% Y_2O_3) composites using SEM, TEM and HREM techniques, and to analyse the mechanical behaviour and properties of the composites.

2 EXPERIMENTAL

The materials used in this study were solid solutions of ZrO_2 containing 2 or 6 mol% Y_2O_3, reinforced by 20 vol% SiC whiskers. ZrO_2 powders stabilized by 2 or 6 mol% Y_2O_3, with an average size of about 0·65 μm, were used as the basic materials and the chemical compositions are shown in Table 1. β-SiC whiskers were presented by Toksi Cabon, Japan, having a diameter of 0·1–1 μm

Table 1. Chemical composition of the ZrO_2 (with Y_2O_3) powders (mol%)

Composition	ZrO_2	Y_2O_3	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO
ZrO_2 (2Y)	97·86	2·056	0·017	0·020	<0·01	<0·01	<0·01
ZrO_2 (6Y)	93·75	6·168	0·017	0·020	<0·01	<0·01	<0·01

and a length of 20–30 μm and the chemical composition shown in Table 2. The composites were hot-pressed at 1600°C for 1 h under a pressure of 25 MPa. The final specimen density was measured by the Archimedes method.

Flexural strength and fracture toughness of the composites were measured in air at room temperature using an Instron 1186 machine. All flexural bars were fabricated with the tensile surface perpendicular to the hot-pressing direction. Flexure strength measurements were performed on bar specimens (3 mm × 4 mm × 36 mm) using a three-point bend fixture with a span of 30 mm. Fracture toughness measurements were performed on single-edge-notched bar (SENB) specimens (2 mm × 4 mm × 25 mm) with a span of 16 mm and a half-thickness notch was made using a diamond wafering blade.

Fracture surfaces of the composites were examined using a HITACHI S-570 scanning electron microscope (SEM). The volume percentages of t-ZrO_2 in total ZrO_2 were estimated by X-ray diffraction on the as-polished and the fracture surfaces. The microstructure of the composites were characterized by TEM. Thin foil specimens taken normally to the hot-pressing axis were prepared by dimpling and subsequent ion-beam thinning. The characterizations of whisker/matrix interface were employed in a JEM-2000EX-II high resolution electron microscope at atomic level resolution.

3 RESULTS AND DISCUSSION

3.1 Microstructure of SiCw–ZrO_2 matrix interface

As shown in Fig 1, the whiskers in ZrO_2 + SiCw composites after fabrication were seen mainly along the hot-pressing axis because of their preferential alignment in this plane. The serrate surfaces of the whiskers appeared to have a good bond with the matrix. Although the geometric mismatch of the whisker and the matrix is large, the relative density of composites is nearly 99% due to the proper hot-pressing technique.

Interface characterization of ZrO_2 (with 2 mol% Y_2O_3) composites is shown in Fig. 2, indicating that the whiskers have a good bond with the matrix. The interfacial region in the matrix shows

Table 2. Mechanical properties of SiCw/ZrO_2 (Y_2O_3) composites

Material	ZrO_2 (2Y)	ZrO_2 (6Y)	SiCw/ZrO_2 (2Y)	SiCw/ZrO_2 (6Y)
Flexure strength (MPa)	888 ±70	293 ± 11	1341 ± 23	310 ± 27
Fracture toughness (MPa m$^{1/2}$)	13·71 ± 1·85	3·42 ± 0·15	16·31 ± 1·06	4·78 ± 0·05

the lath structure of monoclinic phase ZrO_2 (2Y) transformed by induction of residual thermal stress due to the difference in the thermal expansion coefficient between the whisker and matrix ($\alpha_{ZrO_2} = 10 \times 10^{-6}$ K^{-1}, $\alpha_{SiCw} = 5 \times 10^{-6}$ K^{-1}). This t→m transformation could dissipate some of the interfacial residual stress and hence be of benefit to the properties of the composites.[5] Extensive contrast bands across the matrix, shown in Fig. 2, further confirm the presence of residual stresses at the interface.

The structure of the ZrO_2 (6Y) composite is shown in Fig. 2(b), revealing that the whiskers are also well bonded with the ZrO_2 matrix (observed under transmission electron microscopy (TEM)) and there is no transformation trace due to the high Y_2O_3 content, which is consistent with the results of XRD.

High resolution electron microscopy micrographs of the interface between ZrO_2 (with 2 mol% Y_2O_3) matrix and SiC whisker are shown in Fig. 3. It reveals that the whisker is directly bonded to the ZrO_2 matrix except for a several nm thickness disordered atom layer at some regions (Fig. 3(b)). The lattice image taken from the ZrO_2 (with 6 mol% Y_2O_3) composite indicates that the whisker and matrix are separated by a thin layer of amorphous phase, showing that the high Y_2O_3

Fig. 1. TEM image of SiCw/ZrO_2 (with 2 mol% Y_2O_3).

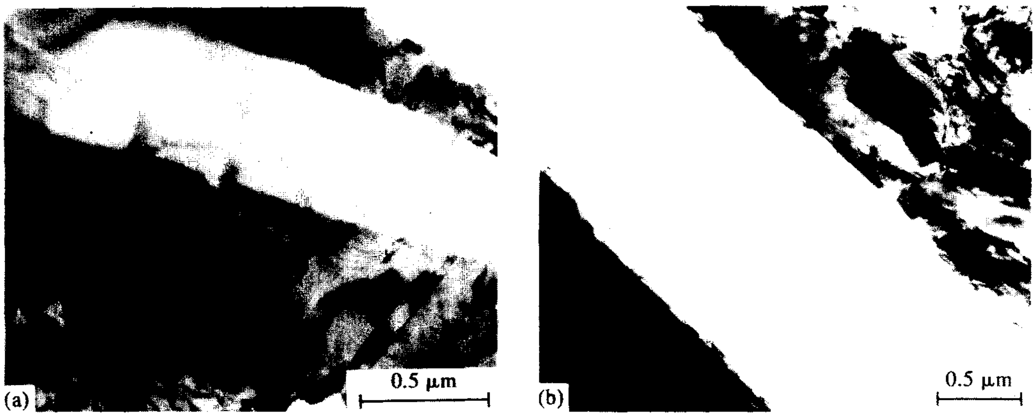

Fig. 2. TEM images of SiCw/ZrO_2 (with Y_2O_3) composites. (a) SiCw/ZrO_2 (with 2 mol% Y_2O_3) composite. (b) SiCw/ZrO_2 (with 6 mol% Y_2O_3) composite.

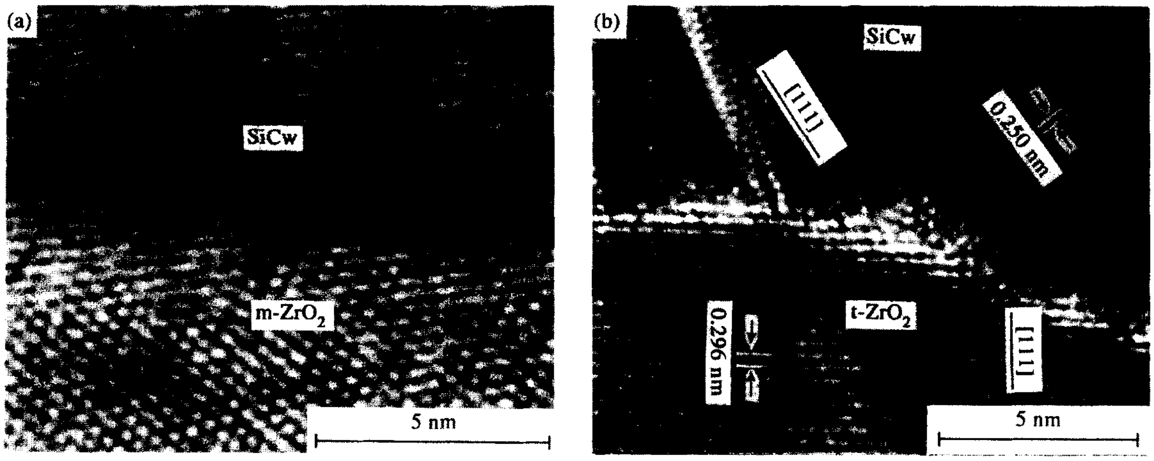

Fig. 3. Microstructure of interface between SiC whisker and ZrO_2 matrix in SiCw/ZrO_2 (with 2 mol% Y_2O_3) composite. (a) No interfacial layer. (b) Thinner disordered atom layer at the interface.

content in the ZrO_2 matrix has promoted the formation of the interface amorphous layer.

3.2 Mechanical properties and fracture mechanisms of SiCw/ZrO_2 (Y_2O_3) composites

The mechanical properties of the SiCw/ZrO_2 (Y_2O_3) composite and its corresponding ZrO_2 matrix are summarized in Table 2. It can be seen that the composite with ZrO_2 (with 2 mol% Y_2O_3) matrix shows an increase of 453 MPa in strength and 2·65 MPa $m^{1/2}$ in toughness compared to the ZrO_2 (2 mol% Y_2O_3) matrix. However, for the ZrO_2 (with 6 mol% Y_2O_3) composite, the increase in strength and toughness is only 17 MPa and 1·16 MPa $m^{1/2}$, respectively, indicating that SiC whiskers incorporated into the ZrO_2 (2 mol% Y_2O_3) matrix provided a better toughening effect than the ZrO_2 (6 mol% Y_2O_3) matrix. Fracture mechanisms of the composites will be discussed in detail below.

The flexure fracture surfaces of ZrO_2 (Y_2O_3) composites are shown in Fig. 5. The fracture surface of the ZrO_2 (2 mol% Y_2O_3) composite is much rougher than that of the ZrO_2 (6 mol% Y_2O_3) composite (Fig. 5(a) and Fig. 5(b)), indicating the strong reaction between the propagating crack and the SiC whiskers during fracture. In addition, pull-out whiskers and residuary holes due to whiskers pull-out could also be observed from the fracture surface of the ZrO_2 (2 mol% Y_2O_3) composite. Crack deflection and whisker pull-out make a contribution to the increase in fracture toughness of ZrO_2 (2 mol% Y_2O_3). In

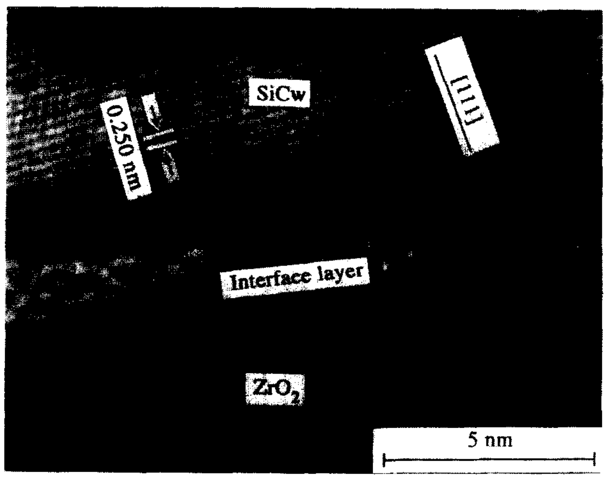

Fig. 4. HREM image of SiCw/ZrO_2 (with 6 mol% Y_2O_3).

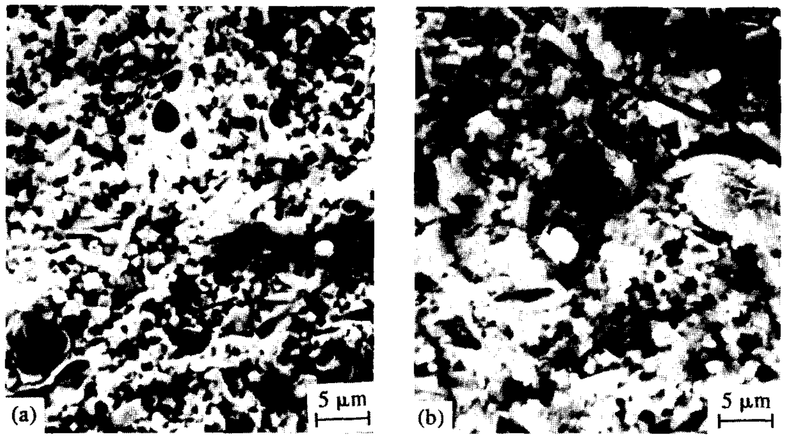

Fig. 5. SEM photographs of the fracture surface of SiCw/ZrO_2 (Y_2O_3) composites. (a) SiCw/ZrO_2 (with 2 mol% Y_2O_3) composite. (b) SiCw/ZrO_2 (with 6 mol% Y_2O_3) composite.

contrast, for the ZrO_2 (6 mol% Y_2O_3) composite, the trace of whisker pull-out could not be found, indicating the strong interfacial bonding stress between whisker and matrix, which could be explained by the results of the HREM observations stated above. For the ZrO_2 (6 mol% Y_2O_3) composite, the whisker and matrix are separated by a thin layer of an amorphous phase. It increases the bonding strength of the whisker–matrix interface and thereby prevents the whiskers pulling out from the matrix. In addition, the lower strength and toughness of the ZrO_2 (6 mol% Y_2O_3) matrix itself makes the crack progress at a high speed and hence causes a larger stress field at the tip of the crack, which makes the whisker fracture before pull-out. The relationship between the crack path and the microstructure of the composites can be demonstrated more clearly by examining the crack propagation produced by Vickers indentation, as shown in Fig. 3.

The results if XRD indicate that the content of t→m transformation is 29·2% during the fracture process in the ZrO_2 (2 mol% Y_2O_3) composite, which also makes a contribution to the improvement of fracture toughness. However, in the ZrO_2 (6 mol% Y_2O_3) composite, there is no dynamic t→m transformation during the fracture process due to the high Y_2O_3 content.

In a word, the combination effects of the transformation stress field of ZrO_2, the thermal mismatch stress between matrix and SiC whisker make the fracture surface of the ZrO_2 (2 mol% Y_2O_3) composite much rougher than that of the ZrO_2 (6 mol% Y_2O_3) composite.

4 CONCLUSIONS

1. The main toughening mechanisms in SiCw/ZrO_2 (with 2 mol% Y_2O_3) composites are crack deflection, crack bridging, whisker pull-out and dynamic t→m transformation. Crack deflection is the main toughening mechanism in SiCw/ZrO_2 (with 6 mol% Y_2O_3) composites.
2. The mechanical properties of SiCw/ZrO_2 (with 2 mol% Y_2O_3) composites are higher than those of SiCw/ZrO_2 (with 6 mol% Y_2O_3) composites,

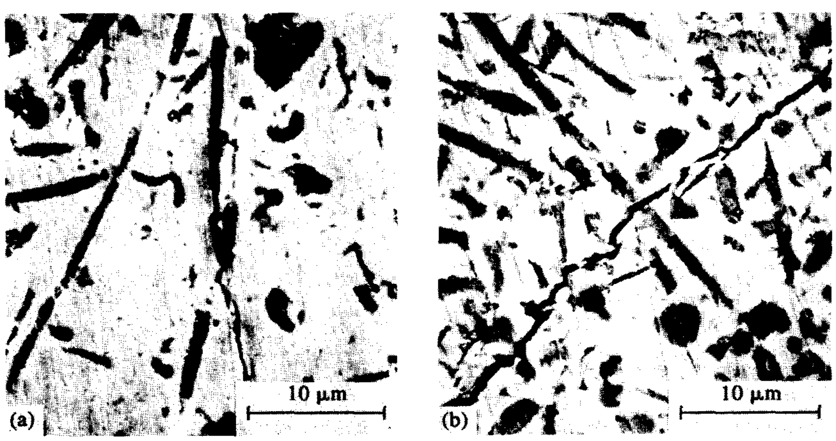

Fig. 6. SEM photographs of indentation crack propagation paths in SiCw/ZrO_2 composites. (a) SiCw/ZrO_2 (2Y) composites. (b) SiCw/ZrO_2 (6Y) composites.

indicating that the Y_2O_3 content has a marked influence on the mechanical properties of $SiCw/ZrO_2$ (Y_2O_3) composites.

3. The whiskers in ZrO_2 (2 mol% Y_2O_3) composites are directly bonded with the ZrO_2 matrix without the presence of an interface layer. In contrast, for ZrO_2 (6 mol% Y_2O_3) composites, there is a thin, uniform layer of amorphous phase at the interface between SiC whisker and ZrO_2 matrix, indicating that a high Y_2O_3 content promotes the formation of an interfacial layer.
4. The presence of an interface amorphous layer between whisker and matrix restrains whisker pull-out during the fracture process.

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