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Microstructure and fracture characteristics of alumina-based prismatic ceramic composites

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Abstract

Al₂O₃/SiC prismatic ceramic composites have been prepared by a structure-controlled process, in which the high-aspect-ratio alumina-based cells with a distinct prismatic texture were separated in three dimensions by thin SiC cell boundaries. The work-of-fracture of the composites has been improved greatly due to the developed paths for crack propagation by the weak cell boundaries, corresponding to longer displacement under reasonable load-carrying condition. The route of crack propagation depended greatly on the interfacial shear strength and boundary thickness. Crack deflecting and delamination are considered as two main contributions at the earlier stage, whereas frictional sliding of fibrous cells becomes more dominant after cracking occurs, especially at lower loading condition. These mechanisms are different from those observed in multilayered monolithic ceramics due to the controlled structures of present materials.

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1. Introduction

Fibrous monolithic ceramics would prove to be candidates for structural applications due to fabricating with commercial powders and showing non-catastrophic fracture behavior. More recently, an extensive effort had been made on the aspects of fabrication and fracture behavior of those fibrous ceramics, and several systems had been developed such as by J.W Holloran [1-5] and T.H. Inoue and co-workers [6-9]. Those ceramics fail non-catastrophically in a similar manner to whisker fiber-reinforced ceramics [10] and multilayered ceramic composites [11,12]. However, it seems to have not been characterized for fracture behavior in fibrous monolithic ceramics. The mechanisms that govern the energy absorption ability of fibrous ceramics are unique, almost referred to that of laminated ceramics or fiber reinforced ceramics, which caused the experimental results not to follow those existing models [1,13–15].

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The objective of the present paper is to evaluate the bending properties such as toughness and fracture energy, and to observe the crack propagation. The main effort is focused to describe the differences in fracture behavior of fibrous ceramics against laminated ceramics and fiber-reinforced ceramics, and fracture mechanism of the prismatic ceramics is to be concluded preliminarily.

2. Experimental procedure

A commercial α -type Al_2O_3 powder with an average particle size of 0.22 μm and specific surface area of 12.3 m^2/g (TM-D, Taimei Chemicals Co Ltd, Japan) was used as a fibrous 'cell'. A fine β -SiC powder (UF-0741, Ibiden Co Ltd) was selected as the interfacial 'cell boundary'.

The prismatic fibrous ceramic was prepared by mold extruding and hot-pressing techniques. The green fibers with a diameter of 0.5 mm were prepared by mold extrusion, and SiC slurry was sprayed on the surface of the arranged green fiber sheets as the thin inter-fiber layers. Then, dozens of sheets were stacked and com-

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pressed into a perform at 523 K under a pressure of 25 MPa, and followed by hot pressing at temperature of 1773 K for 1 h under the same pressure, yielding the prismatic fibrous ceramic.

The bending strength was tested by three-point bending with 0.5 mm/min crosshead speed. The used samples had a polished tensile surface with 1.5 mm thick (in hotpressing direction), 4 mm wide and 40 mm long (in fiber direction). The four-point Single Edge Notched Beam method (SENB) was used for evaluation of fracture toughness and fracture work. The used samples were prepared 3.0 mm thick, 4 mm wide and 40 mm long, and a notch was cut with 1.5 mm in depth and 0.1 mm in tip diameter.

Microstructures and crack propagation of the prismatic ceramic were observed by optical microscope.

3. Results and discussion

3.1. Microstructure

Fig. 1 shows the microstructure in the cross section of prismatic composites with various boundary thicknesses. Flattened hexagonal Al_2O_3 cells about 250 μm in thickness and 550 μm in width are separated by a thin cell boundary (dark), this uniform structure being attributed to the deformation of the green fibers during warm-pressing and hot-pressing. With the increasing of boundary thickness, the cross sections of Al_2O_3 cells showed an ellipse shape. It can be assumed that cells were permitted to move under pressure in the case of thicker cell boundaries.

In practice, the fibrous monolithic ceramic is a special example of laminated or multilayered ceramics. A controlled three-dimensional structure exists in fibrous ceramics, and each fibrous cell is separated completely by a thin cell boundary, although the weaker interphase was introduced just in inter-layers in multilayered ceramics, uniform monolith existed within each layer (separated as two-dimensional). Thus, the difference in the structure results in the variations of mechanical properties and fracture behavior [16].

3.2. Mechanical properties

The mechanical properties of Al₂O₃/SiC prismatic composites with various boundary thicknesses are listed in Table 1. On the basis of the bending data, the toughness energy appears to be improved greatly with little increased toughness but decreased bending strength. It can be considered that the bending strength is decreased due to the reduction of effective load-carrying area by interphase SiC addition, which also results in little increase of fracture toughness. However, the fracture work is improved greatly because the weak interfaces permit the crack propagation with multi-directional routes.

In case of the interphase SiC thickness of $9.3{\text -}15.6$ μm , the fracture work of $1221.4{\text -}1481.6$ J/m² is achieved with good combination of bending strength and toughness. When the thickness is higher than 15.6 μm , the prismatic ceramic is easy to peel/cleave due to weak bonding between the layers, and shear stress along the axial direction is very low, which results from poor sinterability of SiC.

Fig. 2 shows the load-displacement curves of the prismatic ceramics with various SiC interphase. The composites display a non-catastrophic and graceful failure with reasonable load-carrying capability, as described in detail elsewhere [17].

The work-of-fracture of the prismatic ceramic is composed of two parts, earlier stage and later stage, corresponding to the displacement of lower or larger than about 0.5 mm, respectively. During the initial stage with relatively high load-carrying capacity, the deflecting, delaminating and tensile fracturing occur alternatively, yielding a saw-teeth curve. Crack deflection and delamination are two main contributions to the improved fracture energy. During the later stage of bending test, the load-carrying capacity becomes very low, which means the ending of tensile fracturing and the starting of fiber sliding. The sliding friction among fibers becomes a dominant factor for the improved work-of-fracture in the later stage of bending test.

The total displacements are up to 1.0 mm and similar to those of fiber reinforced ceramic composites [10]. However, relatively short displacement occurs for the

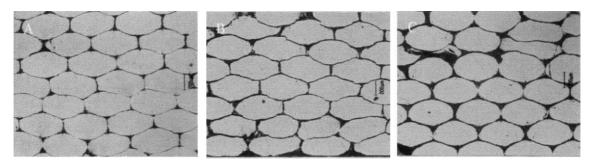


Fig. 1. Cross-section observation of the alumina-based prismatic ceramics with SiC boundaries in various thickness of (A) 4.4 μ m, (B) 9.3 μ m, and (C) 15.6 μ m.

Table 1 Properties evaluation of the Al₂O₃/SiC prismatic composites

Interlayer thickness (μm)	Fracture work (Jm ⁻²)	Fracture toughness (MPa m ^{1/2})	Bending strength (MPa)
4.4	794.6	2.70	164.4
9.3	1221.4	6.85	253.1
15.6	1481.6	5.02	210.3

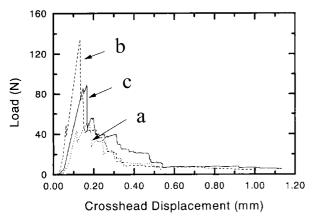


Fig. 2. Load–displacement curves of of the alumina-based prismatic ceramics with SiC boundaries in various thickness of (a) 4.4 μ m, (b) 9.3 μ m, and (c) 15.6 μ m.

alumina-based multilayered ceramics, in which displacement showed value of 0.4–0.6 mm, similar to those of the prismatic ceramics in the earlier stage [11,12,14–16].

Generally, two factors govern the fracture properties of the composites: the fracture resistance of the cell and cell boundary (load-carrying capacity in Fig. 2), and weaker cell boundaries in comparison to the cells (developed crack routes or displacement in Fig. 2).

3.3. Fracture characteristics

As analyzed above, the controlled structure of the cellular ceramics is closely related to their mechanical properties, including fracture features. Fig. 3 shows the crack propagating paths on the side-surface of a sample after the notched bending test. The main route of crack

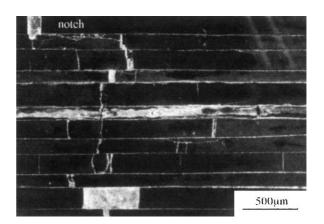
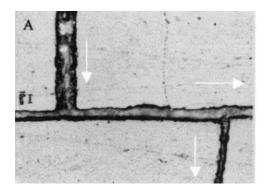


Fig. 3. Crack propagation route on the side surface of the prismatic ceramics with SiC boundaries in thickness of $9.3~\mu m$.

propagation exists near the notch tip, the cracks deflect but do not cause a longer delamination. However, cracks occur also quickly on the multi-portions or else over the entire section, near or far from the main route, which plays an important role in the further improvement for crack path. Then, those cracks with small-scale deflection result in long-distance delamination by interacting with the main route in a bridging manner, some delaminations stretching up to two outer loading points of the tested sample.

A more detailed observation is seen in Fig. 4, in which several typical manners of crack growth can be analyzed. While going across a fibrous layer, a crack is deflected into a weak interface layer, causing a delamination. However, some delaminations grow only a finite distance and deflect in the next fibrous layer or deflecting and delaminating occur at same time (Fig. 4A). The



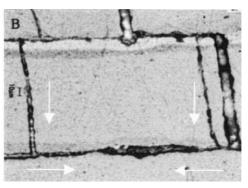


Fig. 4. Propagating manners of cracks nearby the interlayer showing deflecting and delaminating (A) and bridging (B).

interlocking of delamination is attributed to the heterogeneous SiC interlayer [12]. During the middle and later stages of the bending test, the interaction occurs among various propagating cracks, and the bridging is a dominant mechanism as in Fig. 4B. Finally, the sliding of adjacent cells or/and cellular layers lasts a longer displacement at lower load-carrying. Thus, the fracture behavior of the prismatic ceramics lies between multilayered ceramics and fiber reinforced ceramic composites.

4. Conclusion

By means of structure-controlled processing, prismatic ceramic composites of the Al₂O₃/SiC system have been prepared with a distinct prismatic texture of alumina-based cells, which are separated in three dimensions by thin SiC cell boundaries.

The fracture toughness of the composites was improved significantly. However, the bending strength was decreased due to the reduced effective cross-section by weaker boundaries. When the thickness of SiC interphase is 9.3–15.6 μ m, a fracture work of 1221.4–1481.6 J/m² was obtained with good combination of bending strength and toughness.

Generally, the composites fractured in a non-brittle manner, and crack deflecting and delimitating are considered as two main contributions for the improved fracture energy, and frictional sliding of adjacent fibrous cells becomes more dominant after cracking occurs, especially at lower load condition. Fracture characteristics of the prismatic ceramics lies between those of the multilayered ceramics and fiber reinforced ceramic composites due to three-dimensional controlled structure.

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