Micro-Indentation Fracture Behavior of Al_2O_3 -24 vol% $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ Composites Studied by Transmission Electron Microscopy

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The micro-indentation fracture behavior of pressureless-sintered Al_2O_3 -24 vol% ZrO_2 composites containing different Y_2O_3 contents are studied by transmission electron microscopy (TEM) in Vickers indented specimens at room temperature. The hardnesses of those composites decrease with increasing Y_2O_3 , whereas the crack lengths increase. In Al_2O_3 - ZrO_2 (0% Y_2O_3), most of the cracks propagate along Al_2O_3 / ZrO_2 interfaces and Al_2O_3 grain boundaries, while a few grains of monoclinic ZrO_2 (m- ZrO_2) fracture transgranularly. The fractured grains appear to be heavily deformed and to result in a marked reduction in the driving force of indentation cracks. In the Al_2O_3 - ZrO_2 (3% Y_2O_3), the main fracture mode is a mixed type with inter- and transgranular, and tetragonal-monoclinic (t-m) phase transformation in t- ZrO_2 grains partially occur by the indentation. In the Al_2O_3 - ZrO_2 (8% Y_2O_3), the fracture is mainly transgranular, but serious plastic deformation is not observed in the transgranularly fractured cubic ZrO_2 (c- ZrO_2) grains.

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I. Introduction

There have been many reports on the development of high strength and high fracture toughness of structural ceramic composites in order to improve their reliability. One of the composites is an Al_2O_3 - ZrO_2 system which is known to show the phase transformation toughening⁽¹⁾⁻⁽³⁾. The Al_2O_3 - ZrO_2 composites have been produced to apply to fields of wear parts and tool materials. Of these composites, many studies on the fabrication process, microstructures and properties have been carried out since 1980⁽⁴⁾⁻⁽⁷⁾. Recently Al_2O_3 and ZrO_2 powders with high purity and fine grain sizes have been produced. Using these powders, the Al_2O_3 - ZrO_2 composites, which have high density and superior mechanical properties, have been fabricated by pressureless-sintering at a comparatively low sintering temperature of 1450°C. Although the mechanical properties of these composites have been reported⁽⁴⁾⁽⁸⁾⁽⁹⁾, the relationship between microstructure and fracture behavior has been little investigated.

Micro-indentation fracture (MIF) technique is widely used not only for measuring the hardness and fracture toughness, but also for characterizing the deformation and fracture behavior in brittle materials⁽¹⁰⁾⁻⁽¹³⁾. Particularly, by the combination of a Vickers hardness test and TEM, more detailed microstructural studies to identify the mechanical behavior have become possible. In the previous papers⁽¹⁴⁾⁽¹⁵⁾, the microstructure, crack propagation and deformation behavior of Vickers indented specimens from room temperature to 1300°C in the unstabilized Al_2O_3 - ZrO_2 composite were studied by high-resolution electron microscopy (HREM). The purpose of

the present work is to study the crack propagation and fracture toughness behavior of the Al_2O_3 - ZrO_2 composites containing various Y_2O_3 contents.

II. Experimental Procedures

Starting powders with a nominal composition of 76 vol.% Al_2O_3 and 24 vol.% $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ were mixed by wet ball milling in a plastic jar. The mixture was then dried in vacuum, preformed by uniaxial pressing with 10 kg/mm² and sintered in air for 2 h at 1450°C. The sintered body was cut into thin slices using a diamond cutter. Samples for hardness indentation were prepared to the size of 50 × 50 × 70 mm³ and polished to a mirror plane. Vickers indentations were made with a load of 10 kg applied for 30 s. Scanning electron microscopy (SEM) was used for examining the fracture morphology over a wider scale after the three-point bending test. TEM samples were prepared from thin slices, which were mechanically polished to a thickness of about 150 μm and punched out to a diameter of 3 mm by an ultrasonic cutter. After polishing, indents were made with a load of 50 g for 15 s by a Vickers indenter. The back side of the indented plane was polished further up to about 30 μm in thickness by a dimple grinder and finally milled with Ar ions at an accelerating voltage of 3 kV with a glancing angle of 25 degrees. Coating by evaporated carbon was performed to prevent charge-up effects during TEM observations. TEM observations were performed with a 400 kV electron microscope (JEM-4000EX) with a resolution of 0.17 nm.

III. Results and Discussion

1. Hardness and fracture toughness

Figure 1 shows the hardness and crack length of monolithic Al_2O_3 and Al_2O_3 - ZrO_2 composites with different Y_2O_3 contents. In this figure, AZ0Y, AZ3Y and AZ8Y indicate the Al_2O_3 - ZrO_2 (0%mol Y_2O_3), Al_2O_3 - ZrO_2 (3%mol Y_2O_3) and Al_2O_3 - ZrO_2 (8%mol Y_2O_3) composites, respectively. As can be seen in this figure, the hardness (H_v) of the monolithic sintered Al_2O_3 body shows a maximum value of about 1800 as an average value, but the Al_2O_3 - $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ composites show a gently decreasing value with increasing Y_2O_3 content. The crack length made by the Vickers indentation is a minimum value at the Y_2O_3 -free Al_2O_3 - ZrO_2 composite, showing the highest fracture toughness. The crack length becomes gradually longer with increasing Y_2O_3 content, and the Al_2O_3 - ZrO_2 (8% Y_2O_3) composite shows the maximum value, which is larger than that of the monolithic Al_2O_3 .

2. Fracture morphology by SEM

Figure 2 shows SEM images of typical fracture surfaces of the monolithic Al_2O_3 and Al_2O_3 - $\text{ZrO}_2(\text{Y}_2\text{O}_3)$

Fig. 1 Y_2O_3 content dependence of hardness and crack length.

composites, after the three point bending test. Although sintering process was carried out at the same condition, the average grain size of Al_2O_3 is about 2–3 μm which is extremely larger than that of Al_2O_3 (0.5 μm) and ZrO_2 (0.3 μm) in the Al_2O_3 - $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ composites independent of Y_2O_3 content. Thus, the grain growth of Al_2O_3 is restrained by the dispersed ZrO_2 . As clearly seen in Fig. 2(a), the fracture mode of the sintered monolithic Al_2O_3

Fig. 2 SEM fracture morphologies of Al_2O_3 (a), Al_2O_3 - ZrO_2 (0% Y_2O_3) (b), Al_2O_3 - ZrO_2 (3% Y_2O_3) (c) and Al_2O_3 - ZrO_2 (8% Y_2O_3) composites (d) after 3-point bending test.

Fig. 3 TEM image (a) showing crack propagation in the $\text{Al}_2\text{O}_3\text{-ZrO}_2(0\%\text{Y}_2\text{O}_3)$ composite. The mark, I, indicates the indentation center. The HREM image (b) shows transgranular fracture of the m- ZrO_2 grain indicated with a dark circle in (a).

Fig. 4 (a) TEM image showing crack propagation in the Al_2O_3 - $\text{ZrO}_2(3\%\text{Y}_2\text{O}_3)$ composite. An insetted electron diffraction pattern was taken from the region of a dark circle. (b) HREM image showing transgranular fracture of a ZrO_2 grain in the Al_2O_3 - $\text{ZrO}_2(3\%\text{Y}_2\text{O}_3)$ composite.

Fig. 5 TEM image showing crack propagation in the $\text{Al}_2\text{O}_3\text{-ZrO}_2(8\%\text{Y}_2\text{O}_3)$ composite.

Fig. 6 HREM image (a) showing transgranular fracture of Al_2O_3 and c-ZrO_2 grains in the $\text{Al}_2\text{O}_3\text{-ZrO}_2(8\%\text{Y}_2\text{O}_3)$ composite. (b) and (c): enlarge micrograph and an electron diffraction, respectively, of the region indicated with a rectangle in (a).

body shows mixed type failure; that is, the intergranular fracture showing sharp grain shapes and a transgranular fracture with flat cleavage planes. On the other hand, Figs. 2(b), (c), (d) show a tendency for transgranular fracture to increase and for number of pores indicated by arrows, to increase with increasing Y_2O_3 contents, although Al_2O_3 and ZrO_2 grains are not clearly distinguished in these SEM fracture images. In order to characterize the details of the fracture behavior, we carried out TEM observations of crack propagation.

3. Crack propagation by TEM

Figure 3(a) is a TEM image showing the crack propagation originating at the corner of a Vickers indentation. The mark I indicates an indentation site. ZrO_2 grains, which are seen as relatively dark regions with fine contrast, are homogeneously distributed in the Al_2O_3 matrix. Main fracture mode is clearly seen as an intergranular fracture; that is, cracks propagate along the Al_2O_3 grain boundaries and $\text{Al}_2\text{O}_3/\text{ZrO}_2$ interfaces. As indicated by arrows, the direction of crack propagation is deflected by the existence of strong strain fields and microcracks around m- ZrO_2 particles, which were introduced by the phase transformation during the sintering process⁽¹⁴⁾. However, sometimes transgranular fractures were also observed in m- ZrO_2 grains, as indicated with a dark circle. Figure 3(b) shows a HREM image of the m- ZrO_2 showing a transgranular fracture, which is an enlarged image of the dark-circled area in Fig. 3(a). In this image, one can see the existence of heavily deformed lattice fringes, showing plastic deformation. This observation suggests that the cracks, when propagating into the m- ZrO_2 grains, lose a large amount of their driving force due to the plastic deformation.

Figure 4(a) is a TEM image of the Al_2O_3 - ZrO_2 (3mol% Y_2O_3) composite, showing the crack propagation. Although the main fracture mode is an intergranular fracture, as can be seen in this image, transgranular fractures were frequently observed in Al_2O_3 and ZrO_2 grains. This result is consistent with the fracture morphology observed in Fig. 2(c). The crack is more straight and longer than that of the Al_2O_3 - ZrO_2 (0 mol% Y_2O_3) composite (Fig. 3(a)). Near the crack, microcracking, which has been expected⁽⁴⁾⁽⁹⁾ by the stress-induced phase transformation of ZrO_2 grains in the process zone, was not observed. The electron diffraction pattern, which is insetted in this image, was taken from the region of a dark circle, where a crack propagated transgranularly. From the electron diffraction pattern, one can see that two phases of tetragonal and monoclinic structures coexist in the ZrO_2 grain. This means that the t-m transformation partially occurred by the stress-induced martensitic transformation during the crack propagation. Figure 4(b) shows a high-resolution image of the crack tip zone in ZrO_2 . In this image, one can see twins of (111) and (021) planes at the tip of the crack. This type of microtwins were previously observed, in spherical-typed m- ZrO_2 grains in the Al_2O_3 - ZrO_2 (0 mol% Y_2O_3) composite⁽¹⁴⁾. As indicated with dark circles,

a few of edge dislocations were observed between microtwins for the accommodation of stress. It can be noticed that these microtwins exist only near the main crack formed by indentation.

Figure 5 is a TEM image showing crack propagation in the Al_2O_3 - ZrO_2 (8 mol% Y_2O_3) composite. Although intergranular fractures are frequently observed, a main fracture mode in this composite is transgranular fracture, in which cracks propagate into Al_2O_3 and ZrO_2 grains. So that, the crack propagation type is more simple than those of other composites; that is, cracks propagate along more straight line with few deflections. Figure 6(a) shows a portion of the crack propagation in the Al_2O_3 - ZrO_2 (8 mol% Y_2O_3) composite. The HREM image in Fig. 6(b) is an enlarged image of the rectangular area in Fig. 6(a) and a corresponding electron diffraction pattern was insetted in (c). As can be clearly seen in this image, transgranular fractures mostly occurred in Al_2O_3 and ZrO_2 grains. Furthermore, in contrast with the Al_2O_3 - ZrO_2 (0 and 3 mol% Y_2O_3) composites, no plastic deformation and no micro-twinning in ZrO_2 were observed in the transgranularly fractured c- ZrO_2 . The remarkably decreased hardness and increased crack length in Fig. 1 are considered to be due to the increasing of pores and the absence of remarkable crack shielding mechanisms in the c- ZrO_2 grains.

IV. Conclusions

The microstructure of the Al_2O_3 - $\text{ZrO}_2(\text{Y}_2\text{O}_3)$ composites, prepared by pressureless-sintering at 1450°C using high purity and fine raw powders, are characterized as follows.

(1) The hardness decreases by the dispersion of ZrO_2 compared with the monolithic Al_2O_3 and also its value gradually decreases with increasing Y_2O_3 content. On the other hand, the crack length increases with increasing Y_2O_3 content.

(2) The main fracture mode in Al_2O_3 - ZrO_2 (0% Y_2O_3) is intergranular, but sometimes transgranular fractures are also observed in m- ZrO_2 grains. The main toughening effect in this composite is due to crack deflection along boundaries of fine grains, which have strain fields and microcracks, and due to the plastic deformation in the m- ZrO_2 grains.

(3) The fracture mode in Al_2O_3 - ZrO_2 (3% Y_2O_3) is a mixed type of intergranular and transgranular fractures. The phase transformation in t- ZrO_2 grains partially occurred out, it can not give conspicuous contributions on toughening.

(4) In Al_2O_3 - ZrO_2 (8% Y_2O_3), cracks propagate in Al_2O_3 and ZrO_2 grains. However, the plastic deformation of c- ZrO_2 grains is not observed.

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