Transmission Electron Microscopic Observation of the Martensitic Phase Transformation in Tetragonal ZrO_2

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The present work involves the observation of phase transformation in Y_2O_3-containing tetragonal ZrO_2 polycrystals (Y-TZP) using electron microscopy. The observations indicate that the martensitic phase transformation ususally starts from grain boundaries. Transformation was also observed at the tip of a microcrack and its adjacent region. Our observations also indicate that the tetragonal ZrO_2 grains do not all transform at the same time at the fracture surfaces.

I. Introduction

STUDIES of transformation-toughened ceramics, such as ZrO_2-toughened Al_2O_3[1] and partially stabilized ZrO_2 (PSZ),[2,3] have received widespread attention in the last decade. It is still necessary to understand the mode and process of phase transformation and its effects on toughening. Earlier Bansal and Heuer[4] studied the mechanism of phase transformation in ZrO_2 and the crystallographic orientation relationship between the tetragonal and monoclinic phases. Heuer *et al.*[5] have shown the importance of nucleation in the transformation, and Ma and Rühle[6] have observed some new phenomena in the phase transformation of ZrO_2, for instance, nucleation of the transformation at grain boundaries in Y-TZP. They also observed that some smaller grains start to undergo transformation before larger ones. Other relevant recent work can be found in Refs. 7 to 9. This work represents some of our observations on the phase transformation in 3 mol% Y-TZP using transmission electron microscopy. We have confirmed that the phase transformation usually starts from grain boundaries, and have observed the transformation of ZrO_2 grains in front of a microcrack and its adjacent areas. We also observe that t-ZrO_2 grains at the fracture surface do not all transform at the same time, probably because of the different environments of each grain.

II. Results and Discussion

The samples for this research were prepared from hot-pressed samples. The specimens for TEM observation were obtained by the usual process of thinning the bulk material first and then annealing at 1200°C. The transformation process was observed through electron beam interaction with the specimen.

Figure 1 presents electron micrographs before (*A*) and after (*B*) the specimen was strongly irradiated with a 200-keV electron beam, showing that the in situ tetragonal-to-monoclinic ($t \rightarrow m$) phase transformation usually starts from grain boundaries, which can be regions of higher stress due to thermal expansion and/or elastic anistropy. Initiation at this location is thus to be expected from both the nucleation and the stress relaxation points of view.

Figure 2 shows a region containing a preexisting microcrack before (*A*) and after (*B*) electron beam irradiation, showing transformation at the tip of a microcrack and its adjacent areas, which is another type of stress concentration. We observed that the microcrack did not propagate but slightly widened instead; this may suggest that the relaxation of the stress field of the microcrack makes it more resistant to extension under stress while the phase transformation around the tip of a microcrack does not lead to crack growth and weakening of the material. The transformation is also observed to occur a little removed from a microcrack (arrow in Fig. 2(*B*)). At the front of the microcrack tip, the phase transformation could relax the stress concentration, thus preventing microcrack extension.

In Fig. 3, we have observed the phase transformation of t-ZrO_2 at one side of a crack while the other side has only just started to transform, suggesting that there were different stress levels between the two sides of the microcrack.

Figure 4 shows three grains marked *A*, *B*, and *C*. The diffraction pattern of grain *A* shows the tetragonal phase oriented to a $[013]_t$ zone axis while that of grain *B* shows the tetragonal phase along $[111]_t$. These three tetragonal grains with similar sizes showed different results after being irradiated with a 200-keV electron beam for 3 h; grain *A* showed the most obvious transformation (Fig. 5). The final micrograph (Fig. 6) is a lattice image showing the interface between t- and m-ZrO_2 in a partially transformed grain with a contact plane $(100)_t//(100)_m$. These preliminary studies show the advantages of studying the $t \rightarrow m$ transformation in ZrO_2 using in situ experiments.

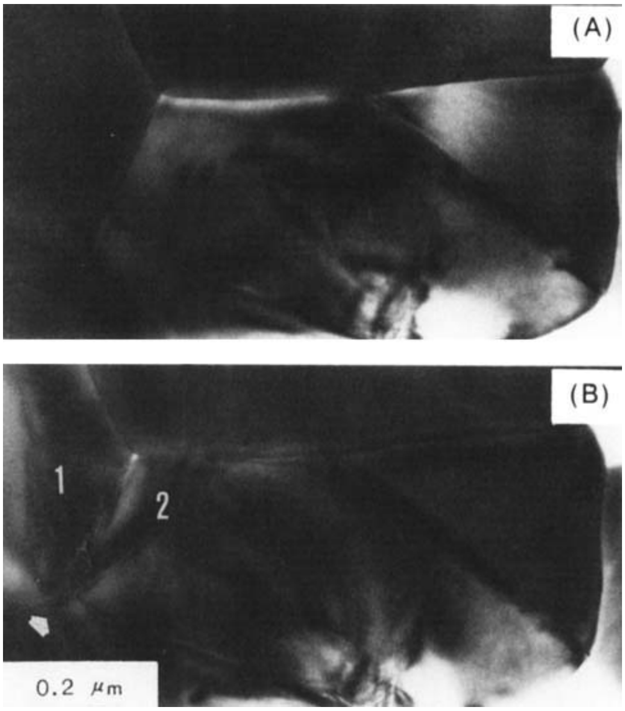

Fig. 1. Electron micrographs of Y-TZP before (*A*) and after (*B*) electron beam radiation with high dosage showing $t \rightarrow m$ phase transformation. Two monoclinic grains (arrow to *1* and *2*) appeared during the process.

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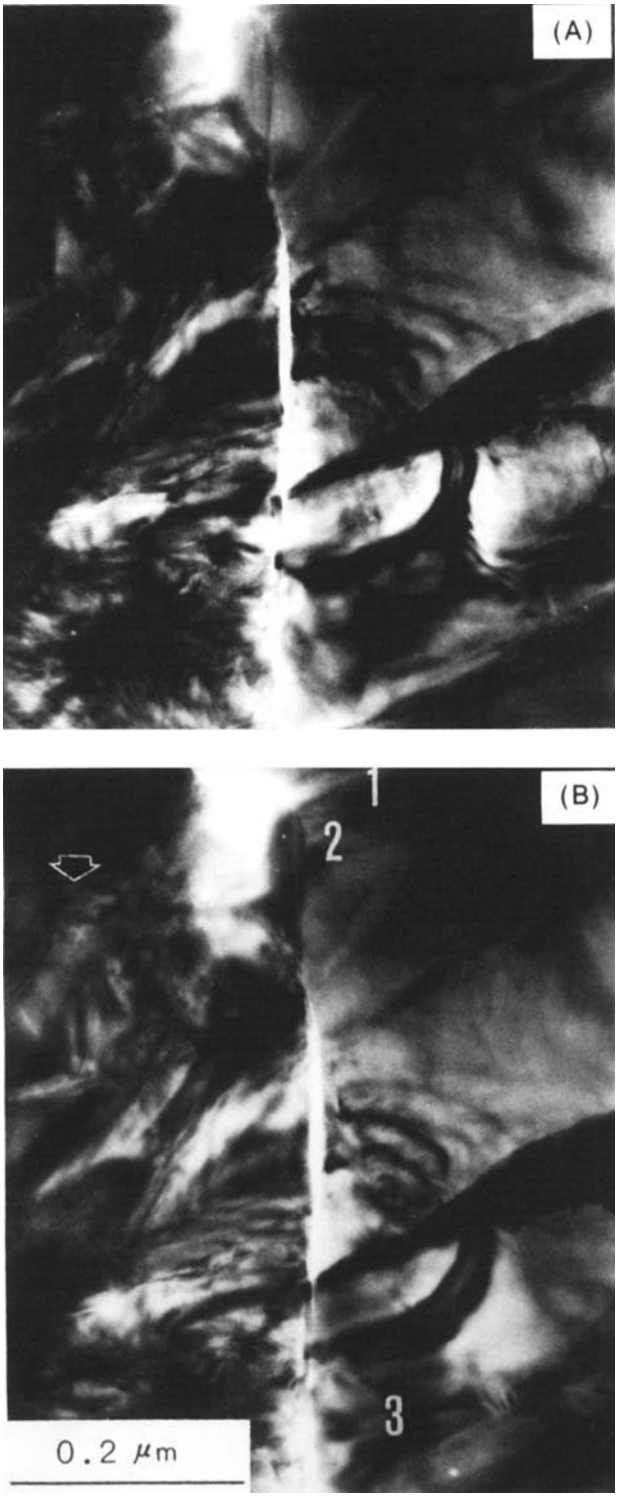

Fig. 2. Electron micrographs of the same microcrack before (*A*) and after (*B*) electron beam radiation with high dosage showing that there are $t \rightarrow m$ phase transformations at and around the tip point of the microcrack (*1*, *2*, and *3* monoclinic grains appeared). There are also phase transformation which occurred far from the microcrack. After all these changes the microcrack is widened.

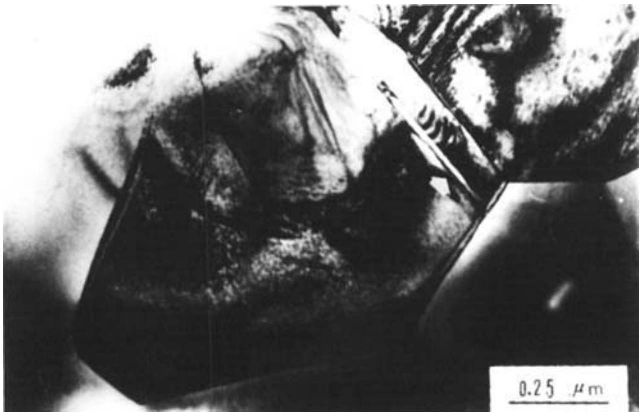

Fig. 3. Electron micrograph showing phase transformation occurred first on one side of the crack indicating probably different stress levels on the two sides of the crack.

Fig. 4. Electron micrograph of Y-TZP showing three tetragonal grains, *A*, B, and *C*, and the diffraction patterns of grain *A* with $[013]_t$ of *t*-phase and $[111]_t$ of *t*-phase of grain *B*.

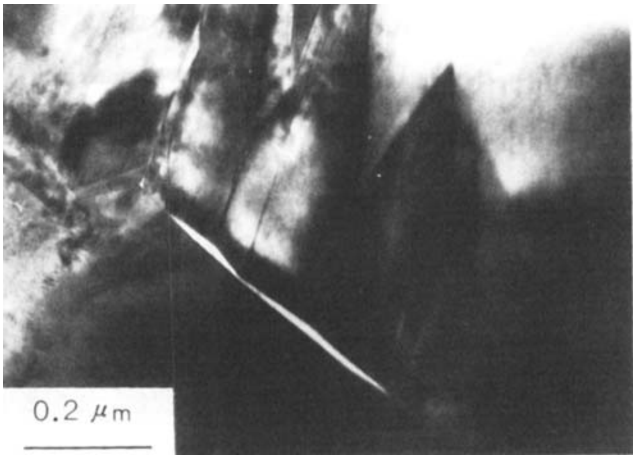

Fig. 5. Electron micrographs of the grain *A* in Fig. 4 after electron beam radiation for 3 h indicates the transformation occurrence as shown with arrow.

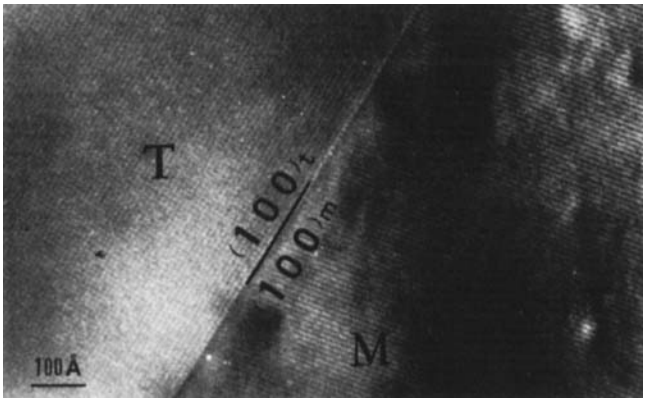

Fig. 6. High-resolution electron micrograph showing contact plane between tetragonal phase and monoclinic phase.

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