

## Ferroelastic and Plastic Deformation of $t'$ -Zirconia Single Crystals

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Polydomain tetragonal ( $t'$ ) zirconia was deformed in compression along a  $\langle 100 \rangle$  orientation at various temperatures between 500° and 1400°C. The stress-strain curves showed a plateau corresponding to ferroelastic deformation, followed by plastic deformation at a higher yield stress level. In both ranges, the strain-rate sensitivity of the stress was measured by stress-relaxation tests. The microstructure of the tetragonal domains after ferroelastic deformation and the dislocation substructure were studied by transmission electron microscopy in a high-voltage electron microscope. As expected, ferroelastic deformation suppressed the tetragonal variant with its  $c$ -axis parallel to the loading direction. The dislocation structure consisted of intersecting dislocations on different slip systems with strongly bowed-out segments. The microprocesses of deformation are discussed here by comparing the deformation data with those of cubic zirconia deformed in the same orientation and based on the observed microstructure. The particular microstructure of  $t'$  zirconia seems to prevent recovery, so that the high flow stress of  $\sim 700$  MPa is preserved up to 1400°C.

### I. Introduction

MATERIALS containing tetragonal zirconia are promising for structural applications, because of their stress-assisted tetragonal-to-monoclinic transformation near room temperature, which enables the design of zirconia-toughened ceramics, and also because of their potential for ferroelastic domain switching at high temperatures. Ferroelastic domain switching was first observed in so-called polydomain tetragonal zirconia, or  $t'$  zirconia.<sup>1</sup> The role of  $t'$  zirconia in toughening ceramics is reviewed elsewhere.<sup>2</sup> This zirconia is a metastable tetragonal phase of low stabilizer content, obtained by a diffusionless displacive transformation during quenching from the cubic phase field;<sup>3</sup> it consists of tetragonal domains or twin variants, which are arranged in a complicated domain and colony structure, described in more detail in Section III of this paper. The ferroelastic deformation consists of the reorientation of the tetragonal domains in the field of the applied stress. Some details of this process have been studied in numerous papers.<sup>1,4–10</sup> Recently, ferroelastic domain switching has been

observed directly by *in situ* straining experiments inside a high-voltage electron microscope (HVEM).<sup>11</sup> In the course of the ferroelastic tensile deformation, only one of the three variants survived, forming a tetragonal single crystal. This crystal contained residual defects. In compression, two of the three variants may remain.

Ferroelastic domain switching in zirconia is not restricted to the  $t'$  phase. *In situ* straining experiments in the HVEM<sup>12</sup> and, later, X-ray diffractometry<sup>13,14</sup> have shown that it may also occur in the tetragonal precipitates in partially stabilized zirconia. Domain switching can have a remarkable effect on the plastic deformation or via dislocation motion. The model of precipitation hardening in partially stabilized zirconia<sup>15</sup> is based on the friction caused by the formation of antiphase boundary-like defects, which the dislocations have to produce in at least one of the variants composing the colonies. Ferroelastic deformation preceding plastic deformation changes the domain structure of the precipitates and, therefore, influences the motion of dislocations in the tetragonal regions of the specimen.<sup>16</sup>

Despite numerous studies on the ferroelastic deformation of zirconia, knowledge of the temperature and strain-rate dependence of the coercive stress, as well as of the plastic flow stress of tetragonal zirconia, is still insufficient. The present paper aims to fill this gap for compression experiments on  $t'$  zirconia, to compare the data with that on the plastic deformation of cubic zirconia, and to yield some information on the domain and dislocation structures formed by ferroelastic and plastic deformation.

### II. Experimental Procedure

Specimens of  $ZrO_2$ -3 mol%  $Y_2O_3$  measuring  $\sim 2$  mm  $\times$  2 mm  $\times$  8 mm were cut along faces perpendicular to the  $\langle 100 \rangle_c$  axes by a wire saw for uniaxial compression experiments. The indexing characterized by the subscript “c” relates to a pseudocubic system fixed with the specimen. The  $t'$  phase was obtained by annealing the specimens in air at 2150°C in the cubic phase field for 10 min, in a standard furnace containing a zirconia tube that had been resistance-heated to reach the high temperature. Afterward, the specimens were quenched, inside the furnace, to 1400°C within 3 min, by switching off the heating current of the zirconia tube, and then furnace-cooled to room temperature within 60 min, to prevent additional crack nucleation. Below 1400°C, diffusion is slow enough to not destroy the metastable  $t'$  structure. The whole crystal volume, in contrast to that of partially stabilized zirconia, should consist of  $t'$  phase containing a uniform concentration of the yttria stabilizer. Because the starting material was inhomogeneous and had many internal cracks, specimens of a suitable quality were difficult to prepare. Therefore, the number of experiments was quite restricted.

The compression specimens were next ground and carefully polished. Deformation tests were performed along the long  $[010]_c$  axis, in a single-screw testing machine, in air, at a strain rate of  $6.6 \times 10^{-6} s^{-1}$ . Stress-relaxation tests were conducted to determine the strain-rate sensitivity of the stress,  $r = d\sigma/d \ln \dot{\epsilon}$ , where  $\sigma$  is the stress and  $\dot{\epsilon}$  the ferroelastic or plastic strain rate. The stress-relaxation curves were plotted as stress,  $\sigma$ , versus the

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