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Ferroelasticity of t'-Zirconia: II, *In situ* Straining in a High-Voltage Electron Microscope

Bernd Baufeld,† Dietmar Baither,‡ Ulrich Messerschmidt,§ and Martin Bartsch

Max Planck Institute of Microstructure Physics, Halle (Saale), D-06120, Germany

Andreas H. Foitzik* and Manfred Rühle*

Max Planck Institute of Metals Research, Stuttgart, D-70174, Germany

The ferroelastic deformation of t'-ZrO_2, the microstructure of which was described in detail in Part I, was investigated by *in situ* deformation experiments in the high-voltage electron microscope at 1150°C. During the experiments those two domain variants with their c-axes perpendicular to the [010] tensile direction were transformed into the third one with its c-axis parallel to the tensile direction. The subsequent 'switching' of the domains inside the colonies proceeds much faster than the penetration of the transformation front into a neighboring colony. Therefore, the transformed region, exhibiting a unique tetragonal structure and containing residual defects, preferentially expands into the longitudinal directions of the colonies. The transformation of single domains proceeds instantaneously within the time resolution of the video tape recording.

I. Introduction

In the metastable material t'-zirconia, tetragonal domains fill the entire crystal volume. The microstructure is described in detail in Part I of this paper.[1] Under load, t'-ZrO_2 shows ferroelastic behavior. Very few data are available on the temperature and strain rate dependence of the coercive stress.[2–4] These data, together with a few results of the present authors, are discussed in the review paper.[5] According to this, the coercive stress strongly depends on temperature. It is probably higher than the stress for single slip on the $\langle 110 \rangle \{001\}$ slip system of cubic zirconia, but lower than the flow stress of partially stabilized zirconia (PSZ).

Ferroelastic domain switching was also observed in partially stabilized zirconia in which colonies of tetragonal domains are embedded in a homogeneous matrix.[6] According to the coercive stress and yield stress data just mentioned, ferroelastic deformation precedes dislocation plasticity, the basic mechanisms of which in untransformed PSZ are described in Refs. 7–10. If ferroelastic deformation takes place, the dislocations must move not through the original microstructure of partially stabilized zirconia, but through a microstructure changed by the ferroelastic deformation. Although the transformed areas may represent a tetragonal single crystal, they contain residual defects. The consequences of this situation are outlined in Ref. 5. In this sense, t'-zirconia may be considered a model substance to study ferroelastic deformation occurring also in other zirconia materials.

The ferroelastic deformation takes place by reorientation or switching of the tetragonal domains under the action of a suitably oriented uniaxial stress. This process proceeds by the movement of domain walls.[11] Contrary to the domain walls in ferromagnetic materials, in ferroelastic ones they are relatively thin, viz, about 5 nm in t'-ZrO_2.[12] Owing to these thin walls, domain switching should not be achieved by a simple sidewise motion of the walls as in ferromagnetics. Instead, new domains are assumed to be nucleated at the domain walls which grow two-dimensionally along the wall, with the whole process repeating until the domain is completely switched.[11,13]

To our knowledge, this process has not directly been observed up to now. Therefore, *in situ* deformation experiments in a transmission electron microscope should provide a better insight into the process of domain switching. In contrast to the uniaxial compression applied in macroscopic experiments, the *in situ* tensile deformation should result in the formation of a single domain orientation, if the tensile direction is chosen along a cube axis. The present paper describes such experiments on t'-ZrO_2.

II. Experimental Procedure

ZrO_2 single crystal specimens of 3 mol% Y_2O_3, prepared from the same material as analyzed in Part I of this paper, were used for the *in situ* experiments. The high-temperature straining stage requires microtensile samples 8 mm in length and about 2 mm in width. They are fixed to the grips of the stage by two tungsten pins in two holes of the samples. The holes are 0.5 mm in diameter and separated by 5 mm.

At first, a wire saw was used to cut slices about 0.3 mm thick from a parallelepiped with all faces orthogonal to the $\langle 100 \rangle$ pseudocubic axes. Holes were drilled by an ultrasonic drilling machine. Then, the samples were ground to a thickness of 0.1 mm on a brass plate, using a 3 μm diamond suspension. The same suspension on a polishing cloth was used for polishing. For further thinning the central region, a dimple grinder was used with a copper grinding wheel and the same diamond suspension. This dimpling is especially necessary to reduce the cross section to enable deformation by the limited force of the straining stage (15 N). To avoid fracture before ferroelastic or plastic deformation of these brittle materials, all faces of the specimens must be polished most carefully, which here was done with an alumina suspension using a cloth wheel before the samples were further thinned down using an argon ion mill at 0.3 mA gun current and 5 kV gun voltage. The final thickness of the region transparent in the electron microscope is about 0.5 μm. For *in situ* tensile straining experiments, perforation of the specimens should in any case be avoided by carefully controlling the thickness via optical thickness fringes.

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*Member, American Ceramic Society.

†Present address: Japan Science and Technology Corporation (JFT), Nagoya 456, Japan.

‡Present address: Max Planck Institute of Metals Research, Stuttgart, D-70174, Germany.

§Author to whom correspondence is to be addressed.