

Ferroelasticity of t' -Zirconia: I, High-Voltage Electron Microscopy Studies of the Microstructure in Polydomain Tetragonal Zirconia

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The microstructure of polydomain tetragonal zirconia (t' -ZrO₂), i.e., a ZrO₂ modification exhibiting ferroelastic behavior, is studied by high-voltage electron microscopy. This material consists of three domain variants of the tetragonal phase with their c -axes nearly orthogonal to each other. Always two variants of these platelike domains are alternately arranged, forming elongated regular colonies. Hence, in both variants the common habit plane of the domains is a {110} twin plane. The colonies are of columnar shape with a <111> longitudinal axis. They are bound by {110} planes, too, which are twin planes for the domains in the contiguous colonies. Owing to their particular structure and the helical arrangement of the adjoining colonies, the material remains coherent and pseudocubic over large macroscopic regions, although it is formed by different tetragonal domains.

I. Introduction

IN MANY zirconia-based ceramic materials, transformation toughening is the dominant toughening mechanism, based on the martensitic transformation of the tetragonal phase into the monoclinic one. High strength and fracture toughness of tetragonal polydomain zirconia (t' -zirconia) with an yttria content of 3 mol%, however, are attained by a ferroelastic transformation.¹ At 1100°C the strength is twice as high as in fully stabilized (cubic) zirconia, although this temperature is well above the equilibrium transition temperature between the tetragonal and monoclinic phases. Accordingly, no monoclinic phases had been found in the stressed samples.^{2,3}

The ferroelastic transformation process differs from the stress-induced martensitic transformation in the fact that there is no change in the crystal structure, but a reorientation of the ferroelastic domains. Ferroelastic properties of tetragonal zirconia arise from a symmetry-lowering ferroic phase transition of the cubic parent phase, resulting in three energetically equivalent orientation states, also called twin variants or domains. These domains, mutually in nearly orthogonal arrangement, form pseudocubic crystals. Under stress, domains can reorient their c -axes to accommodate the strain.⁴ The details of this

effect were investigated by several authors.¹⁻⁶ They depend on the direction and sign of the stress with respect to the crystal orientation and equal domain switching in ferroelectric and ferromagnetic materials. Uniaxial compression tests on t' -zirconia revealed the stress-strain hysteresis, a characteristic feature of ferroelastic materials.⁷ Moreover, the stress-strain behavior was measured as a function of strain rate and temperature.⁸

As one component of the microstructure of this material, the nature of the domain walls was analyzed by high-resolution electron microscopy.⁹ The individual domains form colonies which themselves are frequently arranged in very regular structures, sometimes described as "herringbone" domain structures.³ As in t' -zirconia, the tetragonal domains fill the whole volume, their spatial arrangement has to be highly symmetrical in order to minimize the coherency strain energy. It may therefore be expected that the three-dimensional arrangement of the colonies differs from that in partially stabilized zirconia, in which the colonies of tetragonal domains are embedded in a uniform matrix (e.g., Ref. 10). There is almost no information available on this regular arrangement of the colonies in t' -zirconia. It can be gained by applying high-voltage electron microscopy (HVEM) to relatively thick specimens, facilitating the reconstruction of the real three-dimensional structure. This is the subject of Part I of the present paper. Similarly, the microprocess of domain switching has never been observed directly. High-temperature *in situ* straining experiments were therefore performed inside the HVEM. They are described in Part II.

II. Experimental Procedure

ZrO₂ single crystals of 3 mol% Y₂O₃ (Ceres, North Billerica, MA) were prepared by the skull melting technique. From these crystals, 2 × 2 × 8 mm³ specimens were cut in the shape of parallelepipeds with faces orthogonal to the <100> pseudocubic axes. The t' -phase was obtained by heating the samples within the cubic phase field at 2150°C for 10 min. Subsequently, the samples were furnace-cooled to room temperature within 60 min to prevent crack nucleation. In this temperature range, diffusion effects are negligible, owing to the sluggish cation diffusion. TEM samples for structure investigations were prepared by standard methods, dimpling and ion milling down to a final thickness of about 0.5 μm. The microscope used for diffraction contrast experiments was a JEOL HVEM, operating at an accelerating voltage of 1000 kV and equipped with a 45° double-tilting goniometer stage.

III. Results and Discussion

It is well known that in tetragonal zirconia three variants of tetragonal domains occur with their c -axes nearly parallel to the cube axes of the parent phase. They are denoted by t_1 , t_2 , and t_3 according to their respective c -axes being parallel to the [100],

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