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High temperature plastic anisotropy of Y_2O_3 partially stabilized ZrO_2 single crystals

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Abstract

Yttria tetragonal zirconia single crystals 3.4 wt.% (2.0 mol%), have been deformed in compression at high temperature (1400 °C). The compression axis was chosen to activate the primary slip plane ($\{100\}\langle 011\rangle$). In this system there are three primary slip planes, one cubic and two tetragonal one. In each case, the microstructural feature activated during the deformation process has been studied by optical microscopy. The anisotropic behavior of the three primary slip systems has been studied by a suitable choice of the compression axis. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The microstructure and mechanisms of high temperature deformation of Y_2O_3 partially-stabilized ZrO_2 have been broadly studied in the last decades. This is a consequence of the excellent properties of strength and toughness at low temperatures because of the stress-induced martensitic transformation of the tetragonal to the monoclicnic phase. Hannink et al.[1] reported a good review of the phenomena.

A large number of different phases and microstructures can be found in this system. For a 3 mol% Y_2O_3 content, a single tetragonal phase can be stabilized. When polycrystal, a uniform single tetragonal phase is formed with a fine microstructure with grain size below 1 μm. In this 3 mol% yttria tetragonal zirconia polycrystals (3-YTZP), a superplastic behavior was first reported in 1986 by Wakai et al.[2] Since this first publication, many investigations have reported superplastic behavior of fine-grained zirconia polycrystals stabilized with 2–4 mol% Y_2O_3 and several review papers are available now.[3–4]

Recently, Morita et al.[5] have suggested that dislocations can play an important role in the rate-controlling process of a 3-YTZP superplastically-deformed at 1400 °C and for stresses higher than 15 MPa. This hypothesis is being debated nowadays. In this context, the study of the conditions for dislocation activity seems to be essential. This is the framework in which the analysis of the dislocation microstructure in tetragonal single crystals can reveal the accuracy of the hypothesis cited above.

Due to the different slip planes capable to be activated, single crystals were deformed to activate each of them and to check the anisotropic behaviour. As a consequence of the present work, the critical stress for dislocation activity in each case was obtained, and these values have been used to analyze critically the dislocation activity.

2. Experimental procedure

Tetragonal stabilized ZrO_2 single crystals doped with 3.4 wt.% (2.0 mol%) of Y_2O_3 grown by skull-melting (supplied by CERES Corp. Billerica, Mass. USA) were used. The composition was determined by X-ray and PIXE techniques. Samples were oriented by the Laue X-ray back-reflection technique and cut into parallelepipeds

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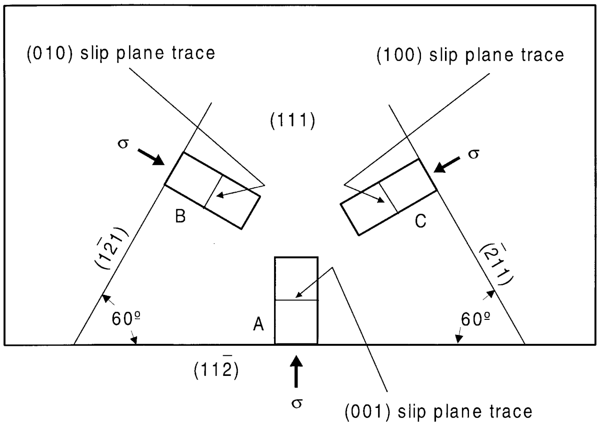

Fig. 1. The three different orientations considered for the compression test are shown. The compression directions and the primary slip plane are displayed for the three types of samples: A, B and C.

with a low-speed diamond saw. Surfaces were polished with a 3 μm diamond paste for optical microscopy observations after deformation. Parallelepipedic specimens, 2.5×2.5×5 mm, were used for compression tests. The samples were cut according to the three different orientations shown in Fig. 1, with the longer (loading) axis along ⟨112⟩ and with the two lateral faces parallel to {111} and {110}. When compressing along the ⟨112⟩ directions only one slip system {100}⟨011⟩ is activated in each case, with Schmid factor equal to 0.47. This slip system is the primary (easy) for cubic stabilized zirconia.[6]

Compression tests were carried out in air at 1400 °C in an Instron universal testing machine model 1185 with a furnace mounted on its frame. The specimens were tested at a constant cross-head speed of 5 μm/min, corresponding to an initial strain rate of 1.5×10^{-5} s^{-1}. SiC pads were used between the alumina pushing rods and the samples to avoid indentation.

The slip plane traces, as well as, the sample shape after deformation have been analyzed by optical microscopy observations of the polished faces in deformed samples using a Leical DMRE microscope.

3. Results

Fig. 2 shows the stress–strain curves obtained when testing along the three ⟨112⟩ orientations for the loading axis (samples labelled A, B and C). An analysis of these plots, as well as the optical microscopy observations will allow to discriminate the deformation mechanisms operating in each case.

At first sight, the testing curves seem to indicate a similar response for samples A and B, as well as a significant difference when compared with the third one. Nevertheless, a careful analysis of the main features in the curves, when correlated to the optical observations allow us to find a great similarity in the deformation mechanisms of the samples B and C. These mechanisms turn to be more simple in the case of the type-A samples.

Deformation of type-A samples takes place by means of dislocation motion along the primary slip plane (100), as deduced from the step lines found on the sample faces (Fig. 3a). The step discontinuities in the curves, measured when deformation is higher than 5%, are linked to the formation of very narrow Lüders bands parallel to the primary slip plane (Fig. 3b and c).

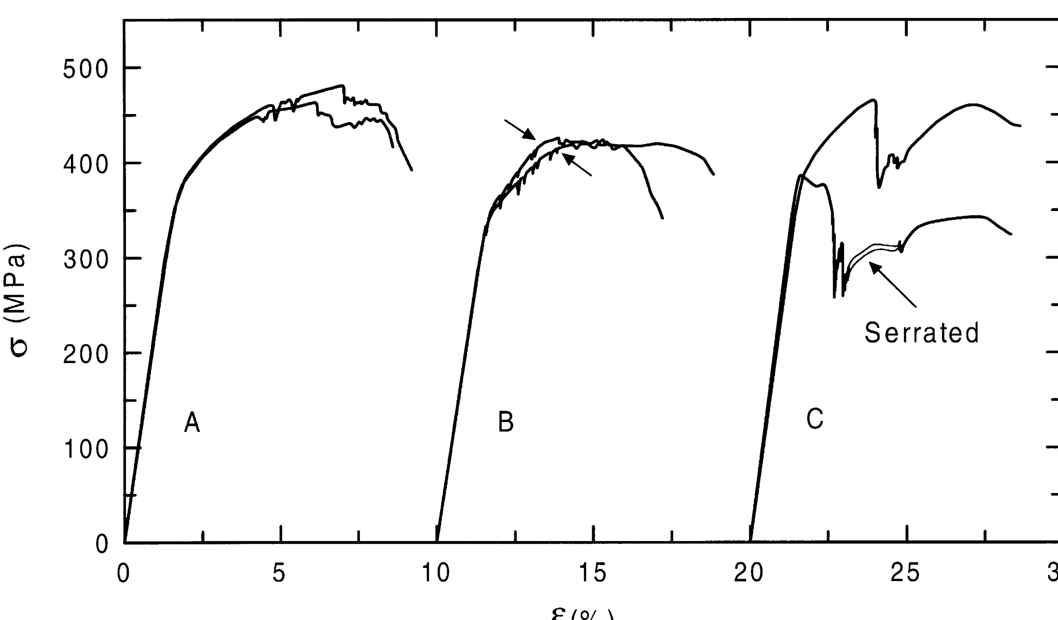

Fig. 2. Stress–strain curves at 1400 °C for the three types of samples used in this work. The arrows in B indicate the beginning of the activation of dislocations. The two lines, in type C samples, indicate the amplitude of the stress discontinuities.

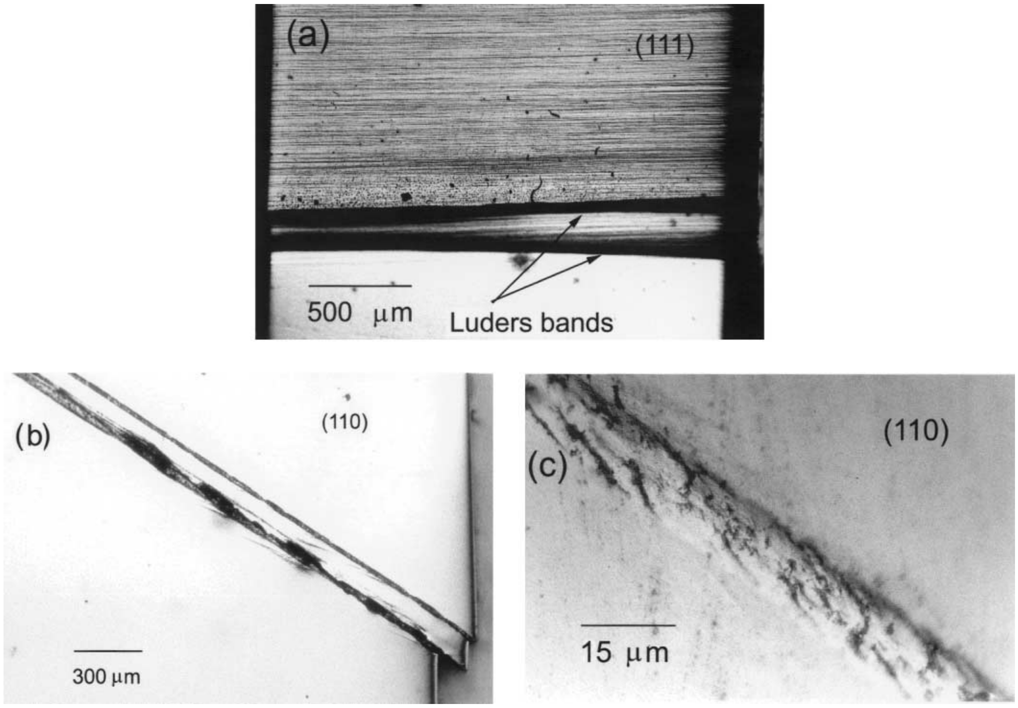

Fig. 3. Optical image of the polished faces in the type-A samples, showing: (a) slip plane traces and Lüders bands in one (111) face, (b) Lüders bands in one (110) face and (c) detail of Lüders band in one (110) face.

The deformation mechanisms involved in the type-B samples are more complicated. Although the deviation from the elastic behaviour in the stress–strain plots is measured approximately at the same stress level as the type A samples, the origin of that deviation is different. It is not a continuous smooth process, but a discontinuous one, as a result of small stress discontinuities. It is related to twin formation before the dislocations are activated, as previously was reported.[7] Fig. 4 shows the traces left by the twins on face $(1\bar{1}0)$. These traces form an angle of $\sim 20^\circ$ with the $\langle 112 \rangle$ compression axis, which corresponds to the theoretical one that the twin planes $(10\bar{1})$ would form when they cut the lateral face.

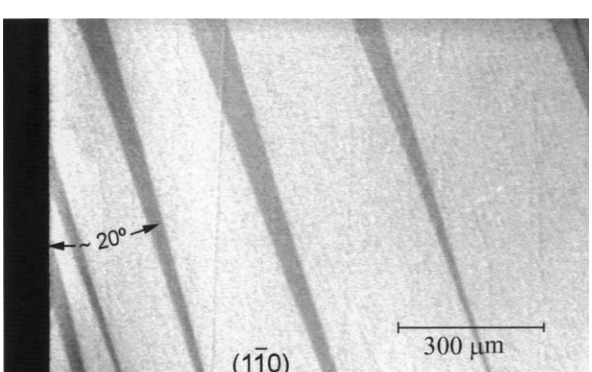

Fig. 4. Aspect of the twins formed during the first stage of deformation in the type-B samples.

Twins as described above had been also observed after deformation in 3 mol% yttria doped zirconia.[8] In this system, three variants of the tetragonal phase, whose *c*-axis are perpendicular to each other, are present in a domains structure. During the deformation those two domain variants with their *c*-axes ($c/a = 1.01$) perpendicular to the [010] tensile direction were transformed into the third one with its *c*-axis parallel to the tensile direction, giving rise to an unique domain. In a similar way, reorientation of some variants can be induced by compression along the *c*-axis of the chosen one.[9] This transformation is called ''ferroelastic switching''. It is a continuous process generating twins of the transformed material. Twins tend to grow when the volume of the transformed region increases.

After twin formation in type-B samples, dislocations are activated (arrows of B in Fig. 2); and in consequence deformation continues by dislocation glide along the primary slip planes, and formation of Lüder bands. A second slip system is activated simultaneously, with the same character; i.e. $\{100\}\langle 011 \rangle$, but a lower Schmid factor (Fig. 5a).

Deformation in the type-C samples have similar features as the type-B ones. However, besides twin formation and dislocation motion in two slip systems, an inhomogeneous deformation band is created (Fig. 6). The formation of this band produces a sharp decrease of stress (of approximately 100 MPa) in the stress-strain curves, followed by other small jumps of stress in form of serrations.[7] This inhomogeneous deformation or

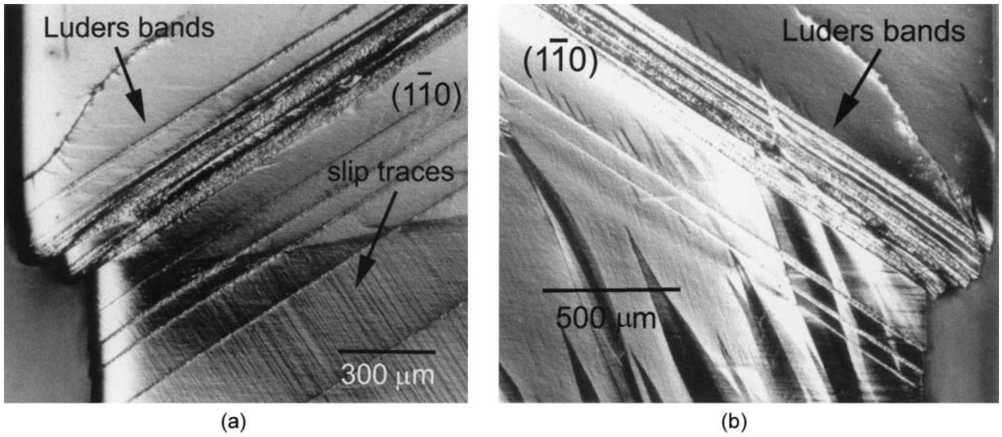

Fig. 5. Optical micrographs showing Lüders bands, slip plane traces of the secondary slip system and twins in (1$\bar{1}$0) face of the type-B samples.

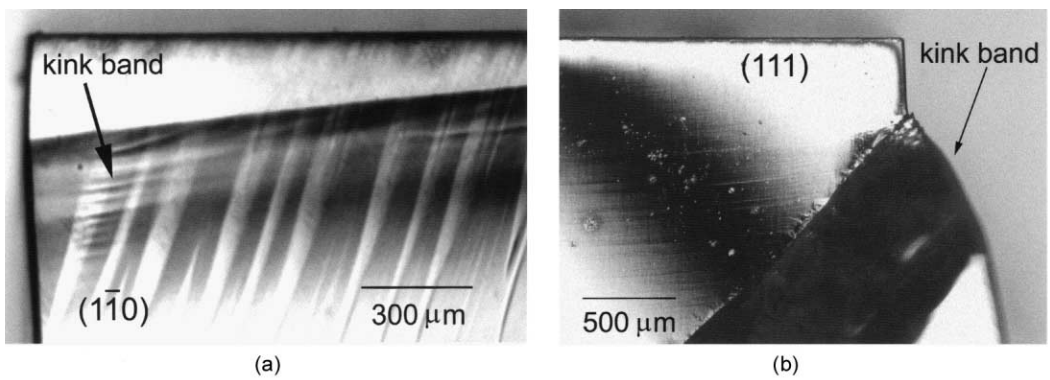

Fig. 6. Optical image of (1$\bar{1}$0) and (111) faces showing kink band formed during deformation in type-C samples.

kink band was found in zinc in association with mechanical twinning.[10] In that material, it relieves bending stresses induced by twinning. The following serrated yielding corresponds to the successive bend planes. These produce a thickening of the inhomogeneity as observed when comparing the kink bands in samples deformed at different strains. These kink bands have also been found in f.c.c. metals deformed by single slip.[11,12]

4. Discussion

The differences found in the mechanisms of deformation operating in the several types of samples indicates that an anisotropy should really exist at the macroscopic scale. These differences can be explained considering that in our samples, at the tests temperature (1400 °C), the tetragonal phase is formed with a single domain. This is in contradiction with what happens with the t precipitate when Y_2O_3 content is higher than 2 mol%. In this case the tetragonal particle is formed by the three possible t variants corresponding to the three possible orientations, perpendicular to each other. In consequence, the material should exhibit an isotropic behaviour at the macroscopic scale.

When the compression axis is the $\langle 11\bar{2}\rangle$ (type-A samples), the c-axis of the tetragonal cell forms 35° with the loading one, and the two a-axes 66° with that one. The primary slip plane is (001), with a fourfold-symmetry (cubic plane), in which dislocation motion can be easily activated. Perfect dislocations can glide along this plane. The dislocation density generated while deforming gives rise to an internal stress. This one is responsible for inhibition of twins formation, as reported in other systems as sapphire.[13,14] Ferroelastic switching is avoided. This is the case of the type-A samples (Fig. 3).

When the compression axis is either $\langle 1\bar{2}1\rangle$ or $\langle \bar{2}11\rangle$ (type-B and type-C samples), the easy slip planes are either (010) or (100). Both of them have a twofold-symmetry (tetragonal ones). Then, dislocation motion activation turns to be very difficult. Dislocation can be activated only by generation of partial ones, and this is an energetically unfavouble case. Consequently, twin formation by ferroelastic switching is an easy process compared with dislocation activation one. These twins (dark areas in Fig. 4) have their origin in a change of orientation of the tetragonal axis during deformation and they are formed by the material whose *c*-axis has been reoriented. The dislocations appear only in later stages of deformation. In these stages, in addition to the primary slip plane, the (001) plane is also activated. Although this is not the plane with the biggest Schmid factor, it is the one in which dislocations motion is easy due to the fourfold-symmetry. In these cases, both twins and dislocations should be observed. This is in agreement with the experimental results (Fig. 5).

With respect to the differences found between the type-B and type-C samples, i.e. the kink band formation in the C samples, it might be related to the elastic anisotropy in these single crystals. This elastic anisotropy is reported in literature,[15] with relatively low values of the anisotropic factor ($A \cong 0.3$) even for cubic single crystals. This anisotropy can be explained because the internal stresses can be relaxed more easily for some crystallographic orientations through a kink band process.

It is important to emphasize that the critical resolved stress for dislocations motion in all the cases is higher than 150 MPa. This is different than the stress measured by Morita in YTZP deformed in the superplastic regime ($\sigma > 15$ MPa).

5. Conclusions

The analyses of high temperature compression tests in 3.4 wt.% (2.0 mol%) yttria-doped zirconia with different crystallographic orientations show an anisotropic behaviour of the different $\{001\}\langle 110\rangle$ activated slip systems.

This anisotropic behaviour is based on the fact that the material, at the tests temperature (1400 °C), present a monolithic structure with an only domain corresponding to the stable tetragonal phase of zirconia.

The resolved stress needed to activate dislocation motion is as high as 150 MPa at 1400 °C in any case.

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