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PLASTICITY OF CUBIC ZIRCONIA BETWEEN 700°C AND 1150°C OBSERVED BY MACROSCOPIC COMPRESSION AND BY *IN SITU* TENSILE STRAINING TESTS

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ABSTRACT

Plastic deformation and dislocations microstructure of the of Y_2O_3-stabilized cubic ZrO_2 has been studied by means of macroscopic deformation tests in air at temperatures between 700°C and 1150°C and subsequent transmission electron microscopy as well as *in situ* straining experiments in a high voltage transmission electron microscope at 1150°C. Compression in the $[1\bar{1}2]$ direction invokes the $(001)[1\bar{1}0]$ slip system, that already has been confirmed at higher temperatures. At lower temperatures the dislocation microstructure is determined by screw dislocations of zig-zag shape in narrow slip bands. The *in situ* experiments at 1150°C demonstrated the instantaneous creation of a complex dislocation microstructure consisting of slip bands of parallel edge dislocations accompanied by areas with dislocation loops and dislocations with screw components.

INTRODUCTION

For conventional compression tests the brittle-ductile transition temperature was generally assumed to be 1000°C, or higher [1] but so far plastic deformation has not been observed below 1100°C. At low temperatures plastic deformation in fully stabilized cubic ZrO_2 until now was only investigated using a high hydrostatic confining pressure [2] or by means of micro hardness indentation. Under confining hydrostatic pressure plasticity at temperatures as low as 250°C was observed [3]. Recently, measurements of the dislocation velocity have been performed on cubic ZrO_2 by means of the double etching technique combined with indentation and four point bending in the temperature range between 1108°C and 1435°C [4]. These results impelled conventional compression tests at temperatures even lower than 1200°C. To observe the dynamics of the dislocation motion directly *in situ* tensile straining experiments in a HVEM at 1150°C were performed.

SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURE

Single crystals of 11 mol% Y_2O_3-stabilized cubic ZrO_2 were used in this study. Compression samples of parallelepipeds of 2·2·8 mm^3 were prepared by standard means. The direction of deformation for all crystals is $[1\bar{1}2]$, which activates the primary $(001)[1\bar{1}0]$ slip system at higher temperatures. From deformed samples transmission electron specimen and from undeformed samples *in situ* specimen have been prepared with a foil normal parallel to $[\bar{1}11]$. The tensile axis of the *in situ* specimen is $[1\bar{1}2]$. Details about the *in situ* specimen preparation can be found elsewhere [5].

The macroscopic compression experiments were carried out in a single-screw testing machine in air at different temperatures. It is possible to run the machine in closed-loop mode under strain control as well as under load control. In the strain control mode one can determine the activation volume by means of stress relaxation experiments at constant strain (R) or strain rate changes (SRC), or in load control mode by means of incremental load changes. The strain rate usually was 10^{-6} 1/s. Strain rate cycling experiments were performed by changing the strain rate from 10^{-6} 1/s to $5 \cdot 10^{-6}$ 1/s.

In order to compare our results with the mechanical behaviour at higher temperatures one sample was first deformed at 1400°C and afterwards at 1150°C. In this experiment different strain rates have been used.

The activation energy was determined by a incremental temperature change experiment at about 700°C.

The high-temperature tensile straining stage for *in situ* experiments is described in [6]. The tests were performed in a high voltage transmission electron microscope operating at an acceleration voltage of 1 MV. The temperature of the sample was 1150°C.

RESULTS OF COMPRESSION TESTS

Figure 1 a shows the stress-strain curve of one sample first deformed at 1400°C and afterwards at 1150°C. The strain rates are: $\dot{\varepsilon}_1 = 10^{-6} 1/s$, $\dot{\varepsilon}_2 = 3.35 \cdot \dot{\varepsilon}_1$, $\dot{\varepsilon}_3 = 5 \cdot \dot{\varepsilon}_1$, $\dot{\varepsilon}_4 = 33.5 \cdot \dot{\varepsilon}_1$, $\dot{\varepsilon}_5 = 50 \cdot \dot{\varepsilon}_1$. In figure 1 b results are plotted for different samples deformed at 700°C, 800°C, 900°C and 1150°C. At 1400°C the flow stress is quite sensitive to the strain rate and during stress relaxation experiments the stress drops to small values, as already reported in [8]. At 1150°C the same tests hardly result in any change in flow stress. The strain rate sensitivity

$$I = \frac{\Delta\sigma}{\Delta \ln\left(\dot{\varepsilon}_{\text{plast}}\right)} \qquad \{1\}$$

can be received from stress relaxation experiments, strain rate changes or incremental load changes, respectively. At 1400°C and after some strain typical magnitudes of I are in the order of 30 MPa [8]. However, at 1150°C the strain rate sensitivity I is nearly zero [7] and increases again with decreasing temperature up to I = 7.8 MPa at 700°C [7]. With decreasing temperature an increasing yield drop effect can be observed after strain rate changes, stress relaxation experiments and partial unloading.

Slip traces on the $(\bar{1}11)$ face and the orientation of the birefringence patterns of deformed samples viewed in crossed polarizers in [110] direction proofed a (001) main slip plane at low temperatures in accordance with experimental results at high temperatures.

TEM OBSERVATIONS

Figure 2 shows transmission electron micrographs of a sample deformed to $\varepsilon_{\text{plast}}$=1.4% at 700°C. The dislocations are concentrated in localized bands being separated by dislocation free regions as displayed in figure 2 a. All dislocations have the same Burgers vector $\vec{b}$ along the $[\bar{1}10]$ direction. Compression in $[1\bar{1}2]$ direction activates the same primary $(001)[\bar{1}10]$ slip system at low temperatures as already observed at high temperatures [9].

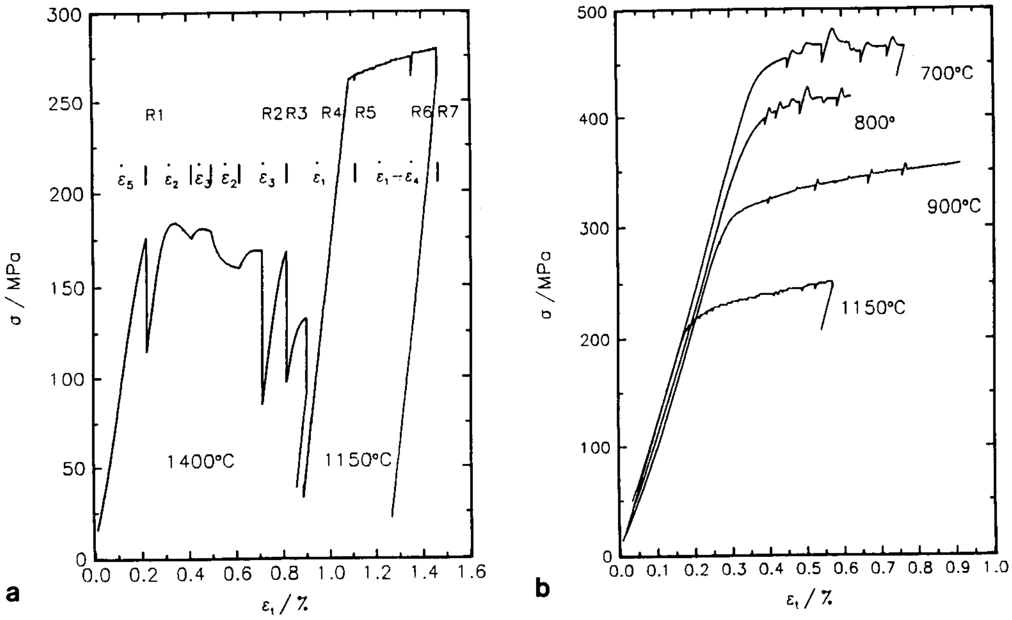

Figure 1 Stress-strain curves of c-ZrO$_2$ with stress relaxation tests (R) and strain rate changes. a) One sample deformed first at 1400°C and afterwards at 1150°C. b) Different samples deformed at different temperatures.

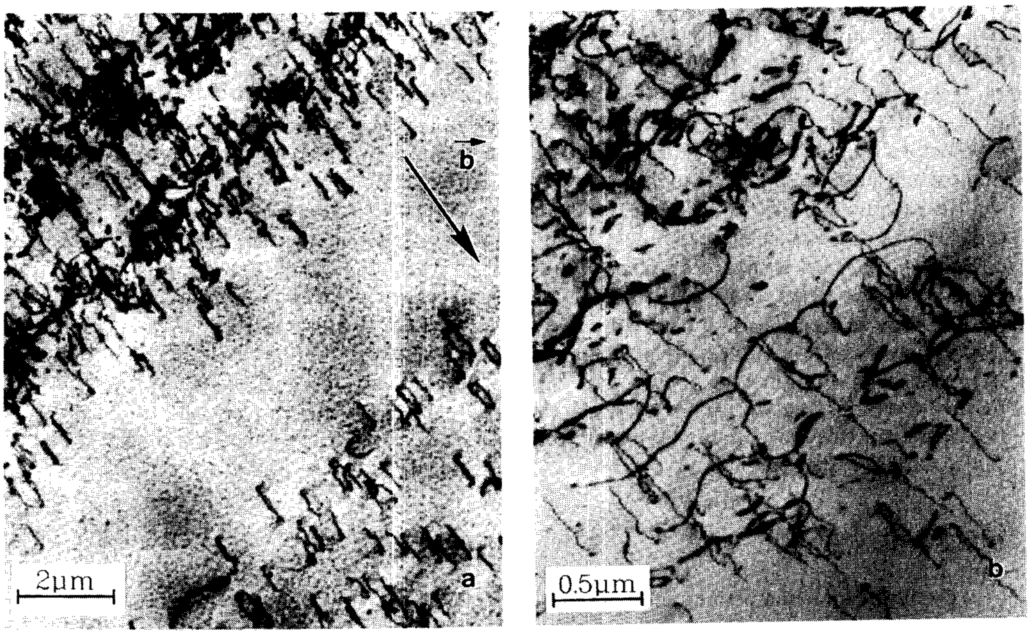

Figure 2 Transmission electron micrographs near the [$\bar{1}$ 11] pole of a sample deformed at 700°C to ε_{plast}=1.4%.

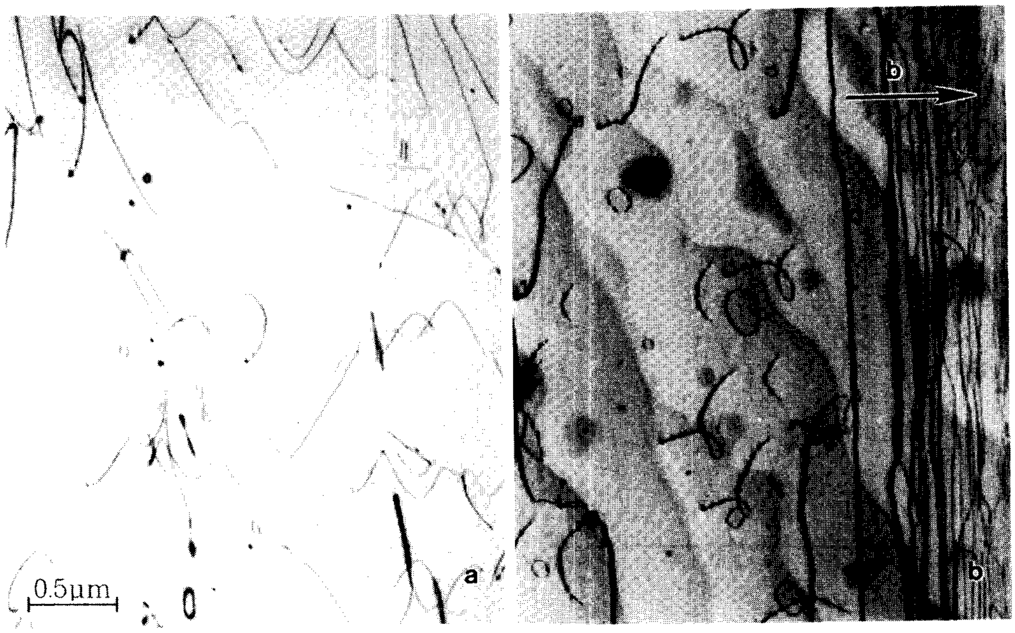

Figure 3 High voltage transmission electron micrographs of c-ZrO_2 deformed at 1150°C a) macroscopically in compression and b) tensile *in situ*.

Screw dislocations are dominating but edge dislocations, loops and debris can be found too. The screw dislocations are heavily jogged and bow out between pinning points, whereas the edge dislocations are rather smooth (2 b).

Figure 3 a is a transmission electron micrograph taken near the $[\bar{1}11]$ pole of a sample deformed to $\varepsilon_{plast}=1.2\%$ at 1150°C. Contrary to the results at lower temperatures (Figure 2) the screw dislocations are not dominating. The dislocation lines are smooth and again oriented along <100>. Some dislocations are jogged and dislocation loops are present.

To examine the dislocation processes directly *in situ* tensile straining experiments are a powerful tool. The as received cubic material is free of dislocations. During straining at 1150°C suddenly a complex dislocation structure develops. As shown in figure 3 b the characteristic microstructure consists of dislocation loops and dislocations with screw components as well as of slip bands of parallel edge dislocations. All dislocations have the same $[\bar{1}10]$ Burgers vector. According to the instantaneous creation of this microstructure the velocity of the dislocations seems to be large. It was only possible to observe the appearance or disappearance of dislocations. As demonstrated in figure 4 the loops, marked by the arrows in the left figure, and the screw dislocation S disappear in time less than one video frame (approximately 0.04 s). Similarly the sudden appearance of loops, signed with the arrows in the right figure, or the screw dislocation D is observed.

DISCUSSION

The dislocation microstructure of samples deformed at relatively low temperatures indicates that the screw dislocations interact with some kind of obstacles. Under stress the dislocations bow out between these pinning points. This interaction becomes weaker with increasing temperature up to

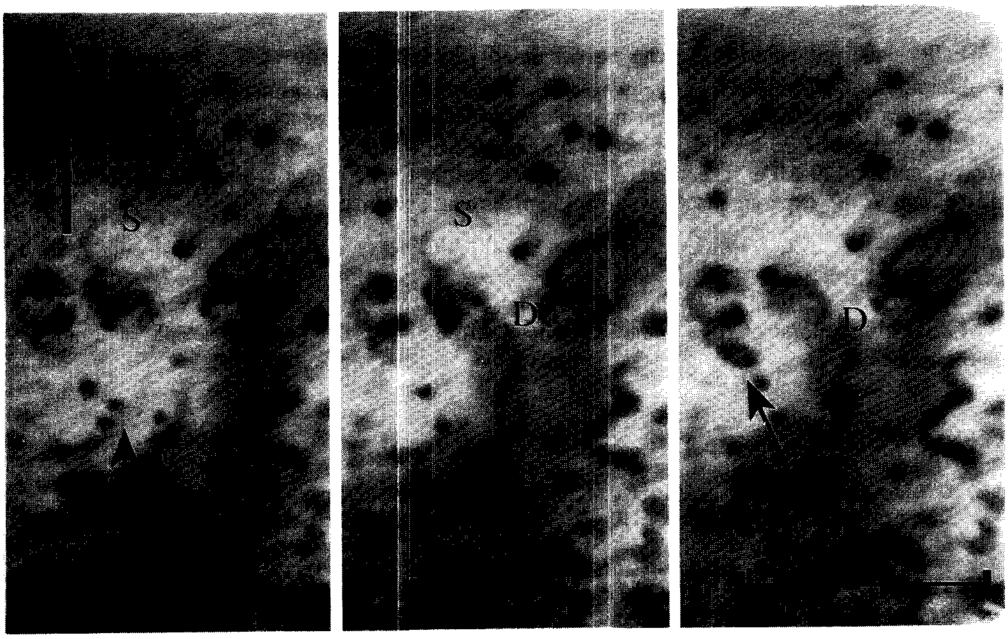

Figure 4 Time sequence of an *in situ* tensile straining experiment at 1150°C. The appearing and disappearing loops are indicated by arrows and the screw dislocations by S or D respectively.

1150°C. The nature of the obstacles are not known yet. They may be point obstacles, small precipitates or jogs.

Jog dragging can explain that the glide velocity is lower for screw dislocation than for edge dislocations. It should be mentioned that cross slip of screw dislocations is possible for example on the {111} slip plane supported by the external stress. A break-up of dislocation dipoles than results in the observed debris.

The activation volume

$$V = \frac{kT}{\Phi I} \qquad \{2\}$$

with the Boltzmann constant k, temperature T and the Schmid factor Φ, is inverse proportional to the strain rate sensitivity I. At 700°C the calculated strain rate sensitivity I would result in an activation volume $V = 76\ b^3$. Assuming that the distance between two pinning points in figure 2 represents the true obstacle distance an activation distance of about 0.4 b can be calculated.

From incremental temperature change experiments an activation enthalpy $\Delta H(700°C) = 7.2$ eV and a Gibbs free energy of activation $\Delta G(700°C) = 3.3$ eV can be estimated [7].

The yield point effect after strain rate cycling, stress relaxation experiments and partial unloading can be explained with the induced Snoek effect, the Cottrell effect or changes of the mobile dislocation density. Usually the first two mechanisms cause a sharp yield point which has not been observed in the material investigated. Taking into account the instantaneous dislocation creation and the extreme high dislocation velocity during *in situ* experiments therefore the change of mobile dislocation density seems to be a good description of the yield point effect.

CONCLUSION

Fully stabilized cubic ZrO_2 can be deformed plastically already at 700°C by conventional compression tests. The $(001)[1\bar{1}0]$ slip system can be activated on deformation along $[1\bar{1}2]$. At low temperatures the dislocations are mainly screw dislocations and concentrated in slip bands. The activation volume is 76 b^3 and the activation energy ΔG about 3.3 eV at 700°C and a total flow stress of about 460 MPa. The obstacles observed at low temperatures are not active anymore at 1150°C and *in situ* experiments showed very high dislocation velocities.

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