6. Microstructure of deformed cubic zirconia single crystals

In this chapter, the microstructures of some of the deformed ZrO\(_2\) samples are presented. The microstructures were investigated by optical interference and birefringence microscopy as well as by transmission electron microscopy in the HVEM as described in Chapter 4.2. The Burgers-vectors were determined by taking micrographs of the same specimen area at different \(\mathbf{g}\)-vectors and using contrast extinctions according to \(\mathbf{g} \cdot \mathbf{b} = 0\). That the slip planes engaged in dislocation glide could be identified, wide-tilting experiments in the transmission electron microscope were performed.

6.1. Optical microscopy of cubic zirconia

Fig. 6.1.1. demonstrates the stress-birefringence patterns of ZrO\(_2\)-10mol\%Y\(_2\)O\(_3\) deformed in the \(\langle100\rangle\) compression direction at 1100°C, i.e. in the intermediate temperature range of low strain rate sensitivities.

![Fig. 6.1.1. Stress-birefringence patterns of ZrO\(_2\)-10mol\%Y\(_2\)O\(_3\) deformed along \(\langle100\rangle\) at 1100°C with \(\dot{\varepsilon} = 10^{-5}\) s\(^{-1}\) till \(\varepsilon = 2.5\%\); \((010)\) and \((001)\) observation planes, respectively](image)

The images correspond to the (010) and (001) side faces. On both faces, well defined slip bands run in the \(\langle110\rangle\) directions. Surface steps are only visible in \(\langle100\rangle\)}
directions. This is consistent with the activation of slip on the four equivalent sets of planes of \( \{110\} \) type with non-zero orientation factor.

Fig. 6.1.2. displays the stress-birefringence patterns of the same material deformed in the same compression direction at 1400°C. Unlike intermediate temperatures, the slip bands are now much finer and more homogeneously distributed. In the particular case, slip is concentrated on two sets of orthogonal \( \{110\} \) planes, as no contrast appears on the face in Fig. 6.1.2b.

In the following, results are presented on the ZrO\(_2\)-15mol%Y\(_2\)O\(_3\) material deformed in the \( \langle 112 \rangle \) compression direction. At low temperatures in the range of stable deformation, only single Lüders bands develop near the ends of the sample. These bands grow to a remarkable width so that the specimens has a kinked shape. According to Fig. 6.1.3. taken from a specimen deformed at 1000°C, i.e. in the range of unstable deformation, slip is now localized in several narrow Lüders bands still at the ends of the compression sample. The orientation of the bands at an angle of about 54.74° with respect to the compression direction on the \( \{110\} \) face and of 90° on the \( \{111\} \) face corresponds to the easy slip system on \( \{100\} \) glide planes.
6.1. Optical microscopy of cubic zirconia

Fig. 6.1.3. Stress-birefringence patterns of ZrO$_2$-15mol\%Y$_2$O$_3$ deformed in $\langle 112 \rangle$ at 1000°C with $\dot{\epsilon} = 10^{-5}$ s$^{-1}$ till $\epsilon = 1.5\%$; (110) and (111) observation planes, respectively

Fig. 6.1.4. displays the stress-birefringence patterns of the same material deformed at 1200°C.

Fig. 6.1.4. Stress-birefringence patterns of ZrO$_2$-15mol\%Y$_2$O$_3$ deformed in $\langle 112 \rangle$ at 1200°C with $\dot{\epsilon} = 10^{-5}$ s$^{-1}$ till $\epsilon = 1.8\%$; (110) and (111) observation planes, respectively

The dislocation bands on parallel $\{100\}$ planes propagate now over the entire length of the sample. The distance between the Lüders bands decreases with increasing temperature so that the slip becomes quite homogeneous at 1400°C as shown in Fig. 6.1.5.
6.2. Transmission electron microscopy in the HVEM

The following series of figures presents the dislocation structure of ZrO$_2$-10mol%Y$_2$O$_3$ crystals deformed along $\langle 100 \rangle$ at different temperatures. In the micrographs, the compression direction is indicated by the vector $\mathbf{d}$. All micrographs were taken near [001] poles perpendicular to the {001} side faces. It was tried to identify the activated slip planes by the following criteria. In the projection used, two sets of orthogonal slip planes run in $\langle 110 \rangle$ directions and are oriented edge-on. Dislocations on these planes should therefore be imaged as straight lines. The other two sets of orthogonal {110} planes intersect the specimen surface along the [010] direction and are inclined with respect to the surface by 45°, so that the dislocations may show their curved shape. {111} planes intersect the specimen surface always along $\langle 110 \rangle$ directions and are also inclined, so that the respective dislocations may show a curved shape, too. In addition, the directions of the Burgers vectors were used to back up the identification of the slip planes.

At low temperatures, slip is concentrated in slip bands, as shown by optical microscopy for the intermediate temperature range in Fig. 6.1.1. The following figures
show crystal regions within these bands. Fig. 6.2.1. is a micrograph of a sample deformed at 700°C.

The Burgers vectors of the dislocations are \(1/2[110]\) or \(1/2[\bar{1}00]\). The straight dislocations in the lower part of the figure run parallel to these directions and belong therefore to \{110\} slip planes. The bowed-out dislocations in the upper part of the figure extend on \{111\} planes. The bowing of short dislocation segments results from pinning of the dislocations by localized obstacles.

Fig. 6.2.2. displays the dislocation structure of a sample deformed at 800°C. In Fig. 6.2.2.a., dislocations of all possible slip systems with non-zero orientation factors are imaged. In Fig. 6.2.2.b., a few dislocations of \(1/2[101]\) or \(1/2[\bar{1}01]\) Burgers vectors are extinguished. In Fig. 6.2.2.c., dislocations of \(1/2[110]\) Burgers vectors and in Fig.
6.2.2. Microstructure of ZrO$_2$-10mol%Y$_2$O$_3$ deformed along $\langle 100 \rangle$ at 800°C till $\varepsilon = 1.1\%$, shown with different diffraction vectors at the [001] zone axis. a: $\mathbf{g} = [200]$, b: $\mathbf{g} = [020]$, c: $\mathbf{g} = [220]$, d: $\mathbf{g} = [220]$.

6.2.2.d. those of $1/2[1 \overline{1} 0]$ Burgers vectors are not visible. These dislocations belong to $\{111\}$ slip planes rather than to $\{110\}$ ones. The angle between the $\{111\}$ slip planes and $\{001\}$ observation plane is $54.74^\circ$. If the dislocations belonged to $\{110\}$ slip planes, they would appear as straight lines at the [001] zone axis, which they do not do. The dislocations have the bowed-out shape again.

Fig. 6.2.3. displays the microstructure of a specimen deformed at 900°C. In this figure, dislocations are located in two bands, separated by a region of a lower dislocation density. Since many dislocations are straight and oriented parallel to the
6.2. Transmission electron microscopy in the HVEM

Fig. 6.2.3. Dislocation structure of ZrO$_2$-10mol%Y$_2$O$_3$

deaformed along $\langle 100 \rangle$ at 900°C till $\varepsilon = 2.2 \%$, shown with
the [220] $\mathbf{g}$ -vector at [001] zone axis
direction of the diffraction vector [2 2 0], they should belong to the (110)1/2[1 T 0] slip
system. Besides, there is a number of curved dislocations that are randomly oriented.
In accordance with the Burgers vector analysis, these glide on the {101} planes.

6.2.4. The microstructure of ZrO$_2$-10mol%Y$_2$O$_3$ deformed along $\langle 100 \rangle$ at 1050°C
till $\varepsilon = 2.1 \%$, shown with two different diffraction vectors with the [100] pole. a:
$\mathbf{g} = [200]$, b: $\mathbf{g} = [2\overline{2}0]$. 
Fig. 6.2.4. presents the dislocation structure of the sample deformed at 1050°C. With the \( \mathbf{g} \) vector in Fig. 6.2.4.a, all dislocations of slip systems with non-zero orientation factors are visible. Many of the dislocations have \( 1/2[110] \) or \( 1/2[1\overline{1}0] \) Burgers vectors as shown for the latter set in Fig. 6.2.4b. Since they are quite straight, slip occurs on \{110\} planes rather than on \{111\} planes. Some dislocations are curved, too.

Fig. 6.2.5. of the specimens deformed at the high temperatures of 1300°C (a) and 1400°C (b) were taken with a \( \mathbf{g} \) vector showing all relevant dislocations. Using the above criteria, the dislocations may belong again to both \{111\} and \{110\} slip planes. It is characteristic of the high temperatures that the dislocations are distributed homogeneously, in contrast to low and intermediate temperatures. Besides, the dislocations do not bow out between localized obstacles anymore.

6.2.5. The microstructure of ZrO\(_2\)-10mol\%Y\(_2\)O\(_3\) deformed along \( \langle 100 \rangle \) at 1300°C and 1400°C till \( \varepsilon = 2.8\% \), shown with \( \mathbf{g} = [200] \)

Summarizing the qualitative observations, it may be stated that both \{110\} and \{111\} slip planes are activated at all temperatures during deformation along \( \langle 100 \rangle \), where the easy slip systems with \{100\} planes are out of stress. Slip is localized at low temperatures and becomes homogeneous at high temperatures. At low temperatures, the dislocations bow out between localized obstacles. This mechanism ceases above about 1050°C.
In order to obtain quantitative data from the microstructure, the dislocation density $\rho$ was measured from about four selected micrographs for each temperature by counting the numbers of intersections of the dislocation lines $N_1$ and $N_2$ with two orthogonal grids of straight lines of lengths $P_1$ and $P_2$ according to $\rho = \frac{N_1/P_1 + N_2/P_2}{t}$. The specimen thickness $t$ was estimated from the projected length of dislocations crossing the specimen on a known slip plane. As demonstrated by Fig. 6.2.6, the dislocation
density is constant up to 1250°C and decreases rapidly above this temperature.

Furthermore, the average length of bowed-out dislocation segments was determined at different temperatures. For this, micrographs were selected showing dislocations with easily visible cusps at obstacles along their lines like Fig. 6.2.2. The cusps at the obstacles are marked by either a reduced or an increased electron microscopy contrast. The average segment length was taken as evaluated dislocation line length \( l_d \) per number of cusps \( m \): \( l_s = l_d / m \). The segment length increases from about 70 nm at 700°C to 150 nm at 800°C to 900°C (Fig. 6.2.7.). At high temperatures, the dislocations are not pinned anymore, as stated above.

A few micrographs were also taken from a ZrO\(_2\)-15mol%Y\(_2\)O\(_3\) crystal deformed along \(<112>\) at 1200°C. Fig. 6.2.8 gives such an example. Dislocations on the \{100\} easy slip plane are imaged edge-on and appear in very narrow slip bands indicating planar slip. The broader band contains dislocations that belong to the \{111\} slip planes.

> 6.2.8. The microstructure of ZrO\(_2\)-15mol%Y\(_2\)O\(_3\) deformed along \(<100>\) at 1400°C till \( \varepsilon = 1.8\% \), shown with \( \vec{g} = [200] \)