A new approach to experimental determination of orientation relationships in pressure-induced phase transformations applied to CdS

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Cadmium sulfide is a semiconducting material used in photometers. At ambient pressure CdS crystallizes in the wurtzite-type structure with the space group \( P6_{3}mc \). At about 2.5 GPa it transforms into the rocksalt type with the space group \( Fm-\overline{3}m \) and the coordination number of the atoms increases from 4 to 6 [1]. At the phase transition a single crystal turns into a polycrystal with a highly preferred orientation [2]. In order to get information about the transition mechanism, the orientation relations between the low- and the high-pressure phase should be determined.

A single CdS crystal was loaded into a diamond anvil pressure cell (DAC) with an opening angle of 90° [3]. The crystal size was 220 x 180 x 40 \( \mu \text{m}^3 \). The indentation of the stainless steel gasket had an initial thickness of 70 \( \mu \text{m} \) and the diameter of the sample hole was 270 \( \mu \text{m} \). A 4:1 mixture of methanol and ethanol was used as pressure transmitting medium. The pressure was measured using the ruby fluorescence method [4]. The synchrotron experiments were performed at beamline P02.1 with a beam energy of 60 keV (\( \lambda = 0.20713 \text{ Å} \)). The data were collected with a Perkin Elmer 1621 detector. The measurement was carried out over an \( \omega \)-range from -42° to 42° with an exposure time of 30 s/step. First, the single crystal was measured with a step width of \( \Delta \omega = 0.15° \). After that, the pressure was increased until the crystal was completely transformed to the high-pressure phase. This state was measured with \( \Delta \omega = 0.5° \). The pressure was subsequently decreased until the high-pressure phase completely disappeared and a third measurement was taken. To get better coverage of reciprocal space, each measurement was done twice with the DAC rotated by 90° around the beam axis.

From the 2D detector images the reflections of the sample were indexed. With the program Fit2D [4] the inner and outer radius of each powder ring were fixed to get a girdle which contains just intensities belonging to a given set of lattice planes hkl. Due to the strong texture of the material these rings show sharp knots and the usual texture refinement and pole figure calculation with the program MAUD [6] failed. Therefore, the intensities were read out directly and combined to incomplete experimental pole figures (see Fig. 1).

From the pole figures the Orientation Distribution Function (ODF) was calculated using the harmonic method [7]. The ODF enables the determination of complete pole figures that cannot be measured and so widens the range that can be compared in search for orientation relations between phases. It can also be plotted directly to show the components of the orientation in Eulerian space (\( \phi_1, \Phi, \phi_2 \)). Fig. 2 shows the \( \phi_2 = 0° \) slices of Eulerian space with indexed main orientation components \( g = (hkl)[uvw] \).

As can be seen from the pole figures and the ODF plots, at the transformation from the low-pressure phase to the high-pressure phase the orientation changes and leads to a complex orientation distribution. Nevertheless, when transformed back to the low-pressure phase the sample reassumes the orientation of the original single crystal. This leads to the assumption that the transformation process might be martensitic.

The next step will be the calculation of the orientation correlation from the orientation distributions of both phases. The orientation correlation will give clues to the actual transformation mechanism.
Figure 1: Experimental pole figures of the 002 and 100 reflections of the low pressure phase and the 111 and 200 reflections of the high pressure phase of CdS.

Figure 2: a) ODF of CdS single crystal, LP phase, hexagonal, $g = (110)[001]$, b) ODF of CdS polycrystal, HP phase, cubic, with complex orientation distribution, c) ODF of CdS polycrystal, LP phase, hexagonal, $g = (110)[001]$.

References