ESRF	**Experiment title:** Dislocation Structure of VI and VII Ice Determined by X-ray Line profile Analysis	**Experiment number**: HS-2655
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Report:

Pure distilled water has been filled into two different diamond anvil cells (DAC) with different diameters, one of 500 and the other of 200 microns, in order to have the possibility either to work on bigger specimens or to achieve higher stresses. In the case of both phases, Ice VI and Ice VII, single crystal (SX) and polycrystal (PX) specimens have been produced and series of X-ray diffraction rotation images were collected. Typical optical micrographs of the grown specimens are shown in Figs. 1a to 1c.

a) b) c)

Figure 1. The phase contrast optical transmission micrographs of the Ice VI specimens, as they were produced in the DAC. (a) Ice VI single crystal before straining. (b) The Ice VI specimen strained up to the appearence of Ice VII grains on the rim. (c) Multicrystal Ice VI specimen. The specimen diameters are about 200 μm.

In order to achieve high angular resolution independent of sample detector-distances, a 40 keV collimated beam was produced using compound refractive lenses. Series of rotation images were then collected at different detector positions to obtain suitable resolution data for peak shape analysis. A portion of a typical image showing one spot is displayed below (Fig. 2). The pictures in Figure 3 show entire data sets (the sum of

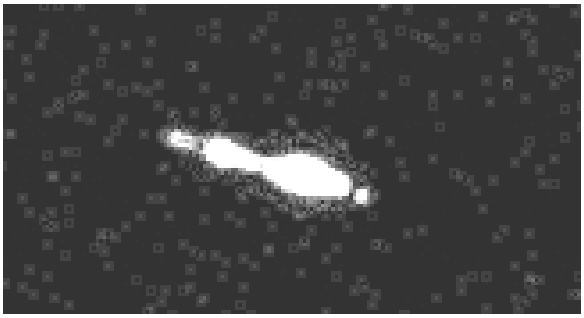
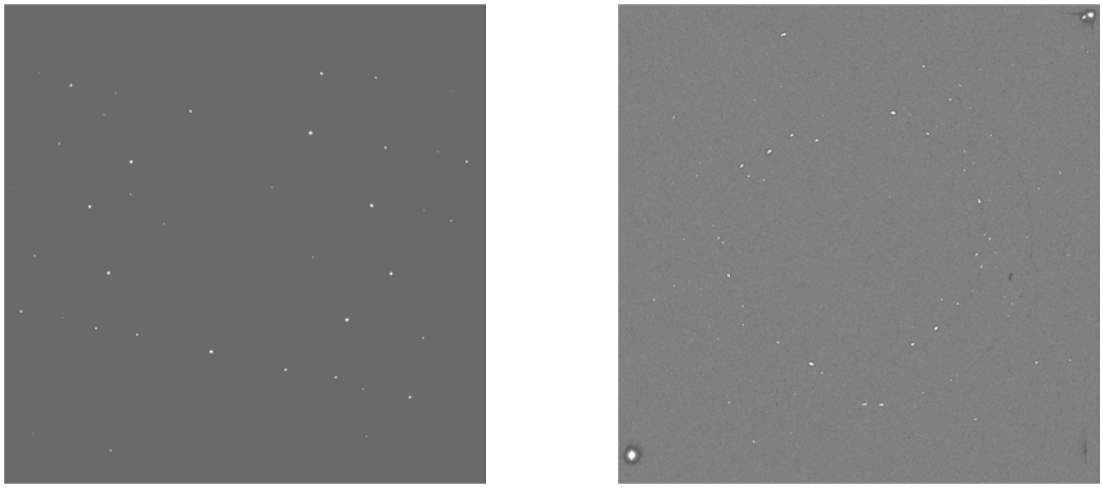


Figure 2. A typical two dimensional image of a diffraction spot corresponding to an individual grain of the strained multicrystal. The data displays strong streaking in the azimuthal direction indicates high sample mosaisity, and peak broadening in the radial direction is due to stain in the sample, most probably due to dislocations.

the rotation images) indicating the completeness of the data. The spots from the series of individual images were then indexed to identify the crystal of origin and Bragg indices. Using this information the integral breadths are plotted versus the absolute value of the diffraction vector in a Williamson-Hall plot, as shown in Fig. 4.



a) b)

Figure 3. Diffraction stacks of the single crystal (a) and multicrystal (b) of the Ice VI specimens. The stacks are the sums of about 90 individual diffraction frames each taken at different ω settings within an angular range of about ± 20º.

The scatter of the breadths indicates strain anisotropy which again is most probably due to the presence of dislocations. For the 200 and 212 reflections the family of reflections is indicated.

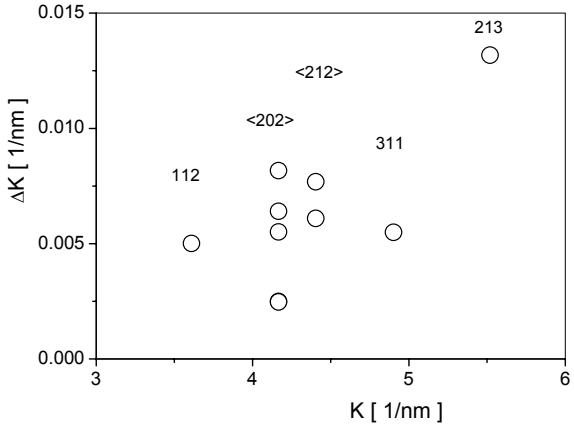


Figure 4. The Williamson-Hall plot of the FWHM of the reflections indicated in the figure corresponding to the strained single crystal of Ice VI. The indices in brackets stand for different families of the reflections.

In the present experiment peak shape analysis has been performed, for the first time, on spots from the individual grains of a polycrystalline sample within a high-pressure cell. Preliminary results indicate that this method will provide plasticity data otherwise unavailable by other methods; detailed analysis is underway.