ESRF	**Experiment title:** X-ray investigation of dislocations in high pressure forms of SiO2	**Experiment number**: HS-3553
Beamline: ID 11	**Date of experiment**: from: 8 May 2008 to: 13 May 2008	**Date of report**: 28 August 2008 *Received at ESRF:*
Shifts: 15	**Local contact(s)**: Gavin Vaughan, ID11	

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Report:

The goal of the experiment was to investigate the nature of dislocations that can develop in phases of SiO_2 plastically deformed, in-situ, under the pressures of the Earth mantle using micro-diffraction and peak broadening analysis techniques. To do so, individual grains have to be found in the diffraction pattern, and for each peak corresponding to those individual grains, we need to obtain a high resolution diffraction pattern for peak profile analysis.

Upon arrival, we had several samples of SiO_2 in the stishovite phase that had been prepared from SiO_2 glass, mixed with platinum, and laser heating, in-situ in the diamond anvil cell. Upon investigation on the beamline, we found that all grains sizes were too low to be observed with the technique.

Therefore, we prepared a new sample starting from SiO_2 in the stishovite phase that had been synthesized in a multi-anvil press prior our arrival to ESRF. This sample was appropriate and was studied at 27 GPa, 39 GPa, 50 GPa, 50 GPa after relaxing stresses in the sample using laser heating, and 50 GPa after inducing high density of defects by cycling the pressure up and down.

For all measurements, the cell was mounted on a single axis goniometer for ω turning. The high resolution CCD coupled detector was used in a close (130 mm) and a far (580 mm) distance from the specimen.

In the close detector position, the diffraction patterns were usually recorded in steps of $\Delta\omega = 0.2$ deg. with 15s counting period over a range of -110 to -65 degrees one side of the sample and 70 to 115 degrees on the other side. These measurements were then used to determine the orientation matrices of the scattering grains and preliminary analysis performed on the beamline indicates that individual grains can indeed be observed within the polycrystal in the diamond anvil cell.

The detector was then moved in the far position. In the far mode, we recored patterns with the detector in a 3x3 matrix position. For each detector position, we recorded patterns with the cell covering the same ω range than in the close detector mode in steps of $\Delta\omega = 0.6$ deg.

Typical image obtained for tor the close detector position is shown in Figure 1. It is then processed for background and powder pattern filtering (Figure 2) from which individual peaks are then extracted and can be processed for determining the orientation matrix of the scattering grains. Typical high-resolution image for a single spot from the SiO_2 is shown in Figure 3. Peak broadening in the radial direction is due to strain in the sample, most probably due to dislocations.

Further data processing is under way. C. Nisr, who joined for the experiment as part of her Masters degree, will start a PhD thesis fully dedicated to this subject in September 2008.

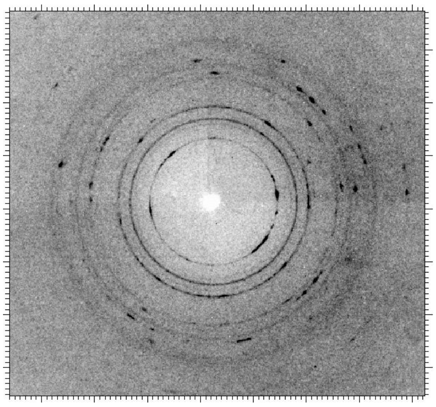

Figure 1: Typical diffraction image in close detector position at one ω value for SiO_2 stishovite at 27 GPa.

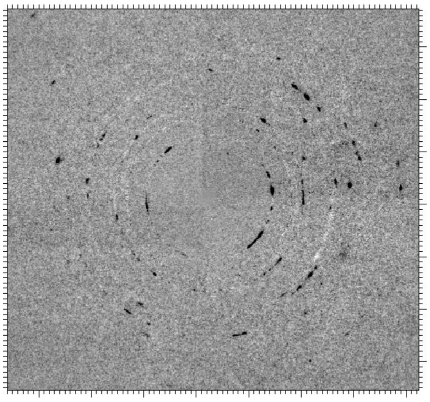

Figure 2: Single grains diffraction extracted from the image in Figure 1 after background and powder pattern filtering.

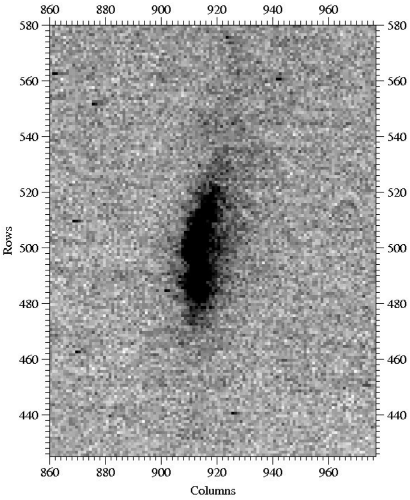

Figure 3: Typical high resolution image of a 110 diffraction spot of SiO_2 stishovite at 27 GPa corresponding to a single grain in the polycrystal. Peak broadening in the radial direction is due to strain in the sample, most probably induced by dislocations.