ESRF	**Experiment title:** Transformation plasticity in quartz and olivine	**Experiment number**: ME 834
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Report:

Mechanical weakening due to solid state transformation of mineral phases has long been proposed to be a significant mechanism for localization of deformation in Earth's lithosphere and the mantle transition zone. Of particular interest are transformation plasticity of quartz, which is supposed to control the mechanical properties of much of Earth's crust, and olivine, which is the most abundant mineral in the upper mantle. So far, experimental observation of transformation plasticity in olivine is lacking. The existence of a minimum in the creep strength of quartz at the displacive □-□ transition was recently proven by Schmidt et al. (2003) using a hydrothermal diamond-anvil cell (HDAC; Bassett et al., 1993). In these experiments, deformation in quartz was achieved by using a single crystal with a synthetic water inclusion and water as the pressure medium, which provided a hydrostatic confining pressure P_c around the sample. The difference between the confining pressure and the pressure in the inclusions generated stress and thus elastic or permanent deformation in the host mineral around the fluid inclusion. Pressure differences between confining and inclusion pressure P_i required for permanent deformation of the quartz around individual fluid inclusions were significantly lower at the phase transition than in either the stability fields of □- or □-quartz. This experimental approach to study deformation at phase transitions is not hampered by the simultaneous discontinuity in thermal expansion at the transition temperature. It can thus potentially be used to quantify the still unknown actual mechanical weakening associated with a phase transition, if the stress around such inclusion can be determined. Therefore, the goals of the experiments reported here were

1. to explore the feasibility of in-situ stress determinations on single quartz crystals at high pressures and temperatures using a HDAC and X-ray micro-diffraction at micrometer resolution (Castelnau et al., 2001; Swamy et al., 2003),
2. to map stress fields in quartz around inclusions at $P_c > P_i$, and

3. to quantify the decrease in the stress required for plastic deformation at the displacive phase transition of quartz relative to that required in the □-quartz stability field.

The experiments were carried out using a monochromatic X-ray beam of 28 keV. The beam was focussed onto the sample in the hydrothermal diamond-anvil cell using a parabolic compound refractive lens. The smallest available spot size was 9.1x2.5 mm^2 (HxV). Single-crystal diffraction patterns were recorded in transmission using a 2-D CCD based diffraction camera. The transmission geometry of the HDAC permitted to record diffraction angles 2□ up to 30° and thus lattice spacings down to 1.7 Å.

The effect of stress on d values of quartz was studied in experiments, in which stress in the sample was generated by compressing a Ti foil between quartz plate and diamond anvil (Fig. 1). Stresses comparable to those expected to occur in the in the host mineral around an inclusion in HDAC experiments (to about 1 GPa) cause a pronounced shift of the d values of the 100 reflection. For this reflection, we obtained a pressure induced shift of -0.03 Å/GPa at 23.5°C based on diffraction patterns of the same, but unstressed, crystal at 0.1 MPa and the pressure along the ice VI melting curve (944 MPa). These experiments support the feasibility of in-situ stress determinations on single crystals at high pressures and temperatures. However, the smallest available beam focus was still larger than the extent of the highly stressed volume in the host quartz (about 1 µm into the quartz around the rim of the inclusions) predicted by finite-element models of the stress distribution. In linescans across inclusion rims, it was occasionally possible (at P_c - P_i > 0.9 GPa) to record a weak signal from the high-stress portion of the sample. Mapping of the stress field around an inclusion will require a much better resolution (a highly focused beam with spot size less than 1µm) and reduction of the fraction of unstressed quartz in the analyzed sample volume by lowering the sample thickness as much as possible, and further improvement of the setup to minimize effects of vibrations and thermal expansion of the apparatus during the scans.

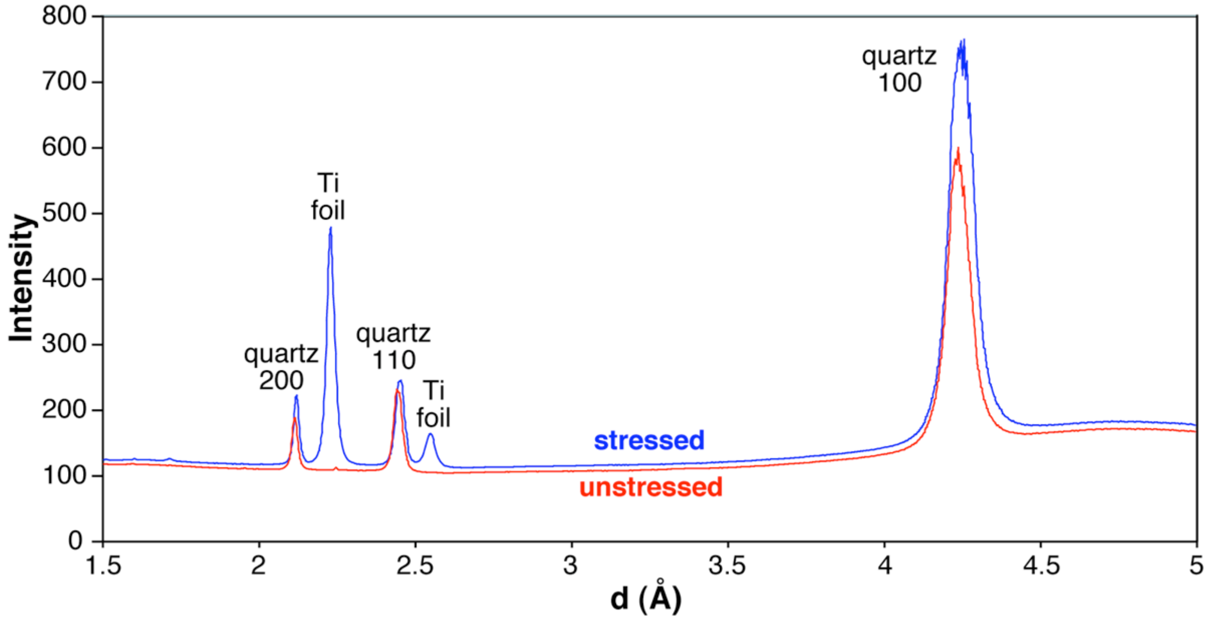

Fig. 1: Single-crystal X-ray diffraction pattern of stressed and unstressed quartz in the sample chamber of a hydrothermal diamond-anvil cell at 23.5 °C and 0.1 MPa. Uniaxial stress was exerted in the direction of the c axis of the sample by compressing a Ti foil between quartz plate and diamond anvil. The d value of the 100 reflection of the stressed quartz shifted by 0.01 Å, which corresponds to an average stress in the sampled quartz volume of approximately 330 MPa. The inhomogeneity of the stress is indicated by the increase in the halfwidth of this reflection (by 11%).

References:

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