ESRF

Experiment title: PRESSURE-INDUCED AMORPHIZATION OF QUARTZ AND BERLINITE

Experiment number: CH16

Beamline: BL3

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Report:

Pressure-induced amorphisation of quartz (α-SiO_2)

Pressure-induced amorphization occurs in a great variety of crystalline solids including ice, molecular crystals and minerals. Quartz (α-SiO_2) and its analogs (berlinite α-$AlPO_4$ and germane α-GeO_2) represent the archetypal and the most studied minerals undergoing this crystal to amorphous phase transformation upon pressure increase. The onset of amorphization is accompanied by the growth in single crystals of birefringent lamellae a few µm thick. They have been interpreted as amorphous lamellae which coarsen and coalesce and account for bulk amorphization. They may also represent a highly disordered crystalline phase which amorphizes upon decompression. In this project we have started the in-situ study of pressure-induced amorphization of quartz by using for the first time X-ray topography with monochromatic and white beams as well as Microdiffraction.

Results of preliminary runs at ESRF

Six allocated shifts at ESRF have permitted to set-up this experiment and to chek up its feasibility on BL3. Single crystals of α-SiO_2 were compressed hydrostatically in a diamond anvil cell (DAC).and optically monitored Up to the appearance of the birefringent larnellae at 17 GPa. The first X-ray topographs of the whole sample *(200x200* µm) were obtained at 3.3 GPa and 10 GPa with a monochromatic beam (E=27 kev, λ=0.46 Å). Figure 1 a (3.3 GPa) shows classical contrast without spot deformation. Figure lb (at 10 GPa) displays a strong anisotropic deformation of the spot as outlined by the oriented diffuse contrasts. The difficulties to find Bragg reflections preclude further experiments using monochromatic beam.

Similar data were obtained with a white beam at 5.2 and 17.5 GPa . In that case the adjustments were easier. On the same Laue pattern several tomographs can be observed corresponding to different diffraction vectors. In Figure 2, the Bragg spot appears with two small "satellites" presenting a stripped structure. The form of the central spot is similar to the shape of the sample seen by optical microscopy. These observations are unexpected since no transforation is repoted in that pressure range for quartz. Further experiments are thus needed to confirm and interpret these observations. At 17.5 GPa the compressed single crystal is invaded by birefringent lamellae, but some peripheric regions remain free of these features. The few tomographs we could record in the allowed beam time have not yet permitted the observation of these Iamellae by X-ray. However, other numerous modifications are observed in the X-ray pattern. Well-defined spots are observed as well as elongated stripes radially disposed. Some stripes present a quadratic symmetry. Some diffuse elongation is also observed for the small spots (Fig. 3). For understanding the origin of the stripes in connection to the appearance of the birefringent lamellae, we have collected every. 10 µm on the sample more than 50 Laue diffraction patterns with a white beam collimated down to 5x5 µm2 in the DAC. Out of the lamellae, the patterns are characteristic of a well-ordered crystal. In the lamellae the diffraction patterns only show the previously observed long stripes. The stripping is thus related to the lamellae. This stripping phenomenon is still unclear but is related to a transition of α-SiO_2 to another crystalline phase highly-disordered or stressed by the existence of disoriented domains. At higher pressures (25 GPa) the sample is no longer diffracting the X-rays as expected for an amorphous phase. The previous preliminary data have shown that X-ray Microdiffraction and X-ray topography images can be obtained at high pressures in a DAC using the high brillance of ESRF BL3 which permits to collimate the beam to very small sizes .

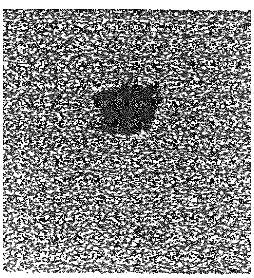

Figure l a

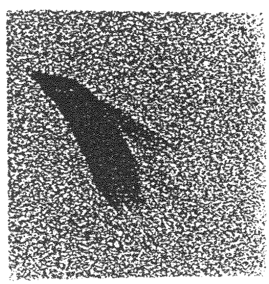

Figure 1 b

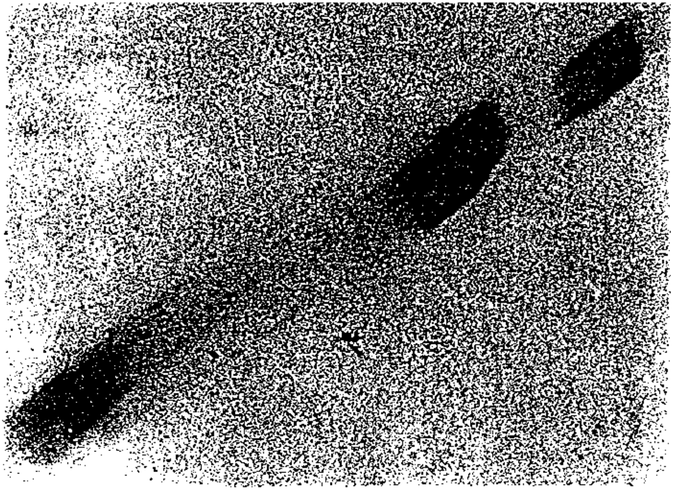

Figure 2

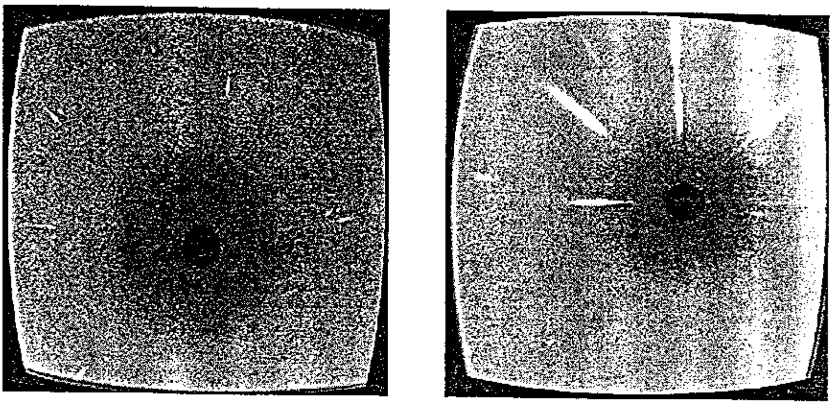

Figure 3. In situ diffraction of quartz at 17.5 GPa with a 5x5 μm spot.
Left Laue pattern: outside the lamellae, right Laue pattern: inside the lamellae