ESRF	**Experiment title:** **X-ray topographic investigations of quartz single crystals around the quartz-cristobalite transition point**	**Experiment number:** HS 559
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Report: The aim of the experiment was to study the behaviour of monocrvstalline quartz at high temperatures, up to the region of the quartz-cristobalite-transformation. To our knowledge no such investigations had been carried out before, using quartz single crystals up to such high temperatures. Earlier investigations of the quartz crystal structure in dependence of the temperature mainly concentrated on the a-a-phase transition around 573°C. Their objective was the determination of the structure and the physical properties of an incommensurate intermediate phase, which appears during the phase transition. Among others, these observations show changes in the real structure during the phase transition, e.g. the creation of Dauphine-twins. This was the reason to choose X-ray diffraction topographic methods to study in real time experiments the interaction of the different phases with the defect structure (phase nucleation, creation and annihilation of defects, . . .) at higher temperatures. We were interested to obtain results to following points :

- Information about the influence of as-grown structurally defects on the transition behaviour and the creation of defects during the temperature treatment, i.e. during the heating-up or cooling down phase.
- Information about the mechanical behaviour of the crystal samples, especially a possible creation of cracks around the transition points.
- Information about the structural behaviour of very perfect quartz samples in dependence on temperatures close to the quartz-cristabolite transition range. We also hoped to find the temperatures at which the reconstructive transition begins and how the structure would change locally within the crystal volume. It is known from powder diffraction, that quartz transforms into cristabolite very slowly. β-Quartz is still observable after several hours even at considerable higher temperatures than the transition temperature.

For the experiments white beam topography in transmission geometry was used. At the ESRF ID19 beamline this technique enables a high geometrical resolution at large crystal-detector distances, necessary for using a large high temperature furnace. The heater of the furnace were two 1000W halogen lamps, whose light was focused on the sample. The temperature was measured with a type B thermocouple, situated lmm from the sample, so that we expected a considerable offset of the absolute value of temperature. The furnace operated with an EUROTHERM-controller, which on its part was controlled by the ESRF SPEC control system to enable a variable temperature-time management. The samples were cut from a nearly dislocation-free crystal (approximately 5x5 mm^2, lmm thick, HF-etched to remove surface defects). Therefore the samples contained only few single dislocations (Fig.1). The quartz plate was mounted on an Al_2O_3-ceramic holder as stress-free as possible. This was very important to minimize the formation of temperature stresses during the heating. The in situ recording chain was used, with Kodak-films (exposure time ≈0.1s) and several cameras (in situ registration of several reflections, with different image field and resolution) connected to a video recorder.

In accordance with well-known observations the creation of Dauphine-twins takes place slightly below the a-ß-phase transition temperature. This can be observed by means of reflections (e.g. 30-31), which are sensible to changes of the structure factor due to twin-law, and thus showing structure factor contrast. In all experiments we also observed the formation of cracks due to temperature stresses although we worked with a very low temperature-time-gradient. Fortunately the sample did not break down and so we could obtain topographs up to 1180°C. The twins and temperature stresses disappeared above the α-β-phase transition temperature. Up to 1180°C (maximum temperature in the experiments) neither changes of the defect structure nor a creation of new defects were observed (Fig.2). Dislocations were observed similar to those at room temperature. A beginning of a reconstructive phase transition could not be observed within a time interval of 2h at 1180°C. The a-ßphase transition did not show any changes in the defect structure during cooling down. During the high-low transition only a creation of Dauphine-twins took place and a deformation of the crystal due to temperature stresses; the number of dislocations, however, did not increase.

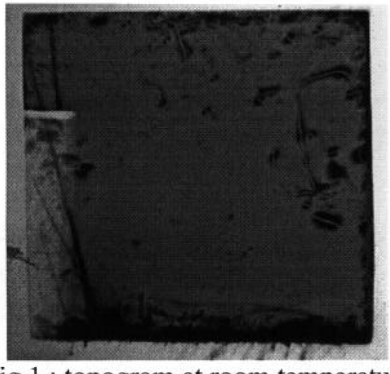

Fig.1 : topogram at room temperature

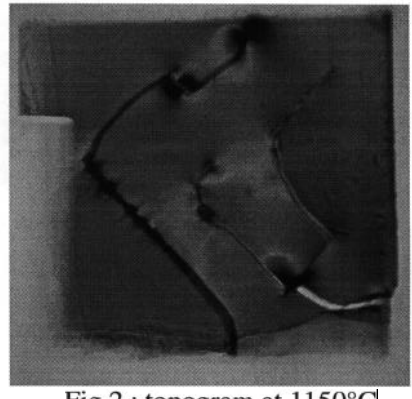

Fig.2 : topogram at 1150°C