| ESRF | **Experiment title:** Quartz transition: structure of the interface between alpha and incommensurate phases | **Experiment number**: MA 206 |
|---|---|---|
| **Beamline**: ID 19 | **Date of experiment**: from: 8/11/2006 to: 11/11/2006 | **Date of report**: 28/02/2007 |
| **Shifts:** 12 | **Local contact(s)**: Jurgen Haertwig | *Received at ESRF:* |


**Names and affiliations of applicants :** (all experimentalists)

G. Dolino, P. Bastie

Laboratoire de Spectrometrie Physique ; Universite Joseph Fourier - Grenoble 1, France

B. Capelle

Laboratoire de Mineralogie et Cristallographie ; Universite Pierre et Marie Curie, Paris, France

V. Chamard

TECSEN, Faculte des Sciences et Techniques Saint Jerome, Marseille, France

P. L. Guzzo

Universidade Federal de Pernambuco Cidade Universitaria, Recife, Brazil


## Report:

Since its discovery in 1889, the $\alpha$–$\beta$ transition of quartz at 847 K, has been the subject of a continuous interest, and it is often presented as the generic example of a first order transition in a mineral. However for some times, puzzling incertitude remains on the exact transition behaviour, in particular after the discovery in 1956 of the transitional opalescence of quartz. Around 1984, an intermediate incommensurate (inc) phase, was observed in a temperature range of 1.5 K between the classical $\alpha$ and $\beta$ phases by neutron diffraction at ILL. The presence of this new phase explains most of the puzzling variations observed previously, as this has been critically discussed by two of us [1]. In February 2004, we have performed a successful experiment on the origin of the opalescence at ESRF on ID 19, with the main results [2] summarised below:

- The opalescence is observed only during the coexistence state between $\alpha$ and inc phases, close to a well defined interface between the two phases, characterised by a small discontinuous jump of the inc wavevector $q$, of 0.002 a* upon cooling.

- This phase interface separates two different light scattering regions. On the $\alpha$ phase side, there is the large angle scattering, probably due to Dauphiné microtwins. On the high temperature side, there is a small angle opalescence, which is due to the presence of the rotation domains of the usual inc phase (in a non-equilibrium state).

The aim of our new experiment was to perform new x-ray measurements, with optimized experimental conditions, to answer the new questions raised by this previous experiment.

Accurate white beam diffraction can easily be performed in the coexistence region of quartz, using our furnace with a temperature control of a few mK. In the new experiment, the experimental conditions were optimised (larger detector distance, often with two exposition times, short for lattice spots, long for satellites). We combine recording with a photographic plate, for high resolution and large field, and with the FRELON x-ray camera, for real time measurements. We use two natural quartz samples with faces perpendicular to the

X,Y, Z axis of quartz, with different size along these axis: a bar Nx (7.5x17x7.5), a cube Nc (10x10x10 mm3) both with X-ray beam close to X axis

We have performed a complete investigation of the inc phase between Ti, the high transition temperature with the $\beta$ phase to Tc the low transition temperature with the $\alpha$ phase. For both samples, we obtained new evidence for the presence of the 1q inc phase in a small range of a few 0.01 K around Ti. We obtained an accurate measurements of the thermal variation of the wave vector modulus and rotation in the pure inc phase between Ti and Tc.

Upon heating, there is nucleation of the inc phase with a single phase boundary which moves across the whole sample while the temperature increases slowly. However with a little cooling, the phase boundary can be stabilised in the middle of the sample as shown in Fig. 1. When the lattice spots is sensitive to the presence of Dauphiné twins, we get well contrasted pictures of Dauphiné twins in the $\alpha$ phase region (on the right side of the Laue spots). An interesting result is that there is a well defined boundary limiting a central region with an intermediate grey contrast and with diffuse X-ray scattering; this is in good agreement with the hypothesis of Dauphiné microtwins. Then on the left comes the inc phase, characterised by the presence of 12 satellites streaks. Under the effect of coexistence stresses, the lattice spots exhibit striking elastic deformations, which have been measured in a great number of conditions.

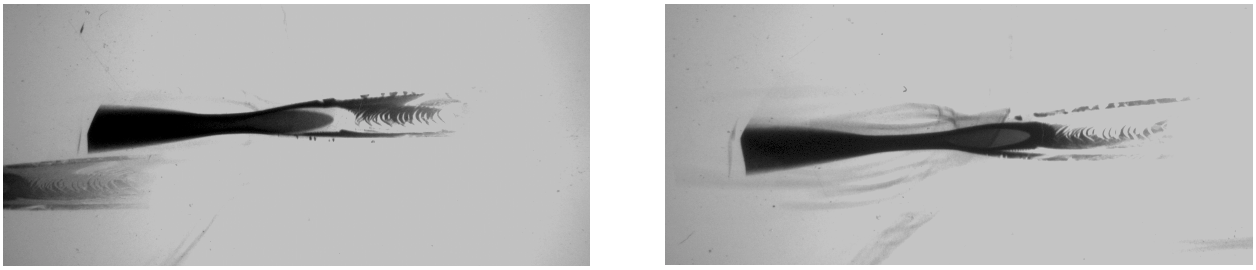

Fig 1 : Two Laue spots visualising the intermediate region responsible of the wide angle light scattering (in the middle) and showing inverted contrast for the Dauphine twins (on the right)

Under finite temperature gradients, the nucleation of the $\alpha$ phase can occurs nearly simultaneously on the two lateral sample surfaces, in front of the colder optical windows, producing a new geometry of the phase coexistence with the presence of two interacting phase boundaries in the sample, allowing measurements under new conditions.

Finally the cubic sample was positioned with one of its face (perpendicular to the Y axis) in front of the x-ray and optical windows, allowing the observation of the inc-$\alpha$ transition on this surface, which was at the lower temperature in the sample. Then upon cooling from the inc phase: the final decrease of the inc satellites can be followed as well as the nucleation of the $\alpha$ phase.

This work gives much information on the complex behaviour occurring at the lock-in transition of the inc phase of quartz.

**References**

[1] Dolino G and Bastie P, J. Phys. Condens. Matter 13, 11485 (2001).
[2] Dolino G, Bastie P, Capelle B, Chamard V, Härtwig J, and Guzzo P, Phys. Rev. Lett. 94, 155701 (2005).