EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: SXRD investigation of the alpha-quartz (0001) surface structure at room temperature and across the alpha-beta phase**	**Experiment number**: SI 1764
Beamline: BM25B	**Date of experiment**: from: 11/02/09 to: 17/02/09	**Date of report**: 02/04/09
Shifts: 18	**Local contact(s)**: Dr. Pilar FERRER	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Wolfram Steurer*, Anton Tamtögl*, Michael Mayrhofer-Reinhartshuber*

Institute of Experimental Physics, Graz University of Technology, Petersgasse 16, 8010 Graz, Austria

Gilles Renaud*, Tobias Schulli*

Institut Nanosciences et Cryogenie/SP2M/NRS, 17 rue des Martyrs, 38054 Grenoble CEDEX 9, France

Report:

The goal of our experiment was to determine the surface structure of α-quartz (0001) by surface x-ray diffraction (SXRD). Despite the importance of quartz surfaces in molecular biology and micro technologies, and the fact that quartz is the second most common mineral in Earth's crust, surprisingly little fundamental work on the surface structure has been carried out up till now. Investigations by atomic force microscopy [1], low-energy electron diffraction [2] and helium atom scattering [1] have been performed the last years, however, no high-resolution picture of the surface structure could be obtained so far. Present experimental evidences of the surface structure of α-quartz (0001) are limited to the observation of an hexagonal (1x1) surface reconstruction and indications of steps on the surface of one-third of the unit cell height [1].

In the experiment reported here, we have measured crystal truncation rods (CTR) at room temperature, shortly below the α–β phase transition temperature (at ~500 K), and above the phase transition temperature (at ~900 K). Measured CTRs – see Fig. 1 for an exemplary CTR measured at G-30 – are of high quality and show distinct features[1]. We have also observed small reconstruction peaks (indicating either a 2x1 or 2x2 reconstruction) in scans performed along the main orientations in reciprocal space; a survey of observed reconstruction peaks is depicted in Fig. 2. Interestingly, the magnitude of these peaks is greatly enhanced in the room temperature scans compared to measurements at elevated temperature. We did not have time to investigate these reconstruction peaks (and their evolution with temperature) in detail however.

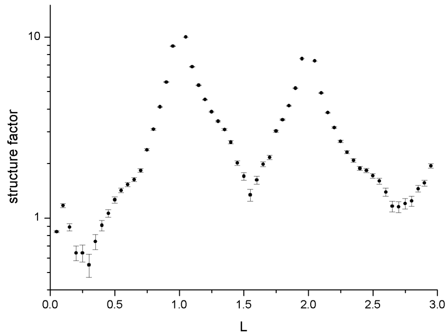

Fig. 1: Exemplary CTR measured at G-30.

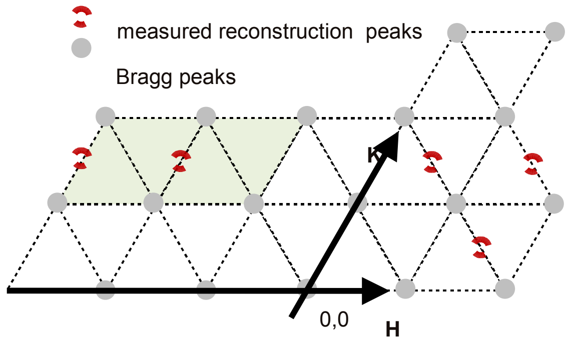

Fig. 2: Survey of observed reconstruction peaks. The shaded area indicates the region of a performed mesh scan.

Our experimental observation of a temperature dependence of the magnitude of the reconstruction peaks is very intriguing with respect to a very recent publication by Y.W. Chen et al. [3]. On the basis of DFT simulations the authors predict five reconstruction patterns (three with a 2x1 pattern, two with a 1x1 pattern) for the quartz surface that are energetically almost identical. Moreover, the authors show that transitions from one reconstruction to another occur without breaking bonds and that the energetically most favorable structure is a 2x1 pattern. Y.W. Chen et al. also suggest that several patterns coexist on a larger surface. Therefore, the observed temperature dependence in our measurements could indicate an almost continuous transformation from a 1x1 pattern to the energetically more stable 2x1 pattern.

To conclude, the obtained results look promising but a more extensive investigation of the α-quartz (0001) surface by SXRD (using a faster goniometer like at BM32 and with thicker quartz samples) is needed to clarify the above outlined hypothesis and to determine the surface structure unambiguously.

[1 W. Steurer et al., *The structure of the α–quartz (0001) surface investigated using helium atom scattering and atomic force microscopy*, Surface Science **601**, 4407-4411 (2007)
[2] F. Bart et al., *LEED study of the (0001) α–quartz surface reconstruction*, Surface Science **311**, L671-L676 (1994)
[3] Y-W. Chen et al., *Finding stable alpha-quartz (0001) surface structures via simulations*, Applied Physics Letters **93**, 181911-3 (2008)

1 Due to technical problems with one of the stepper motors and initial stray reflections from the sample holder, we could measure only a few CTRs at each temperature. The latter problem was mainly caused by a rather complicated and narrowed mounting of the 10x10x1 mm^3 quartz samples. We would also like to note that an appreciable amount of beam time was consumed by the slow goniometer at BM25B.