EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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 original report should be sent, together with 5 reduced (A4) copies, to the User Office.

In addition, please send a copy of your file as an e-mail attachment to reports@esrf.fr**,** using the number of your experiment to name your file. This will enable us to process your report for the ESRF Annual Report.

Reports accompanying requests for additional beam time

If your report is to support **a new proposal**, the orginal report form should be sent with the new proposal form, and a copy of your report should be attached to each copy of your proposal. 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.
- bear in mind that the report will be reduced to 71% of its original size. A type-face such as "Times", 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

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ESRF	**Experiment title:** Liquid Ordering at the Liquid-Brushite Interface	**Experiment number**: SI-779
Beamline: ID-03	**Date of experiment**: from: 14-jun-02 to: 22-jun-02	**Date of report**: 22. 08. 2002
Shifts: 21	**Local contact(s)**: Dr. Christopher Walker	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

J. Arsic* ,D. Kaminski*, P. Poodt* and E. Vlieg*

Department of Solid State Chemistry, University of Nijmegen, The Netherlands

Report:

Here we repport the interface structure of the brushite (010) face in the contact with it's growth solution.

Brushite, $CaHPO_4*2(H_2O)$, is considered as the model system for biomineralisation. It grows easily from solution in a platelike morphology and contains water layers in its structure. The surface is highly hydrated.

Our interest in brushite arises from our study of solid –liquid interfaces. The structure at the solid-liquid interface determines the growth shapes and velocities of a crystal and the effects of impurities and surfactants. Due to the ordered water already incorporated in the crystal structure of brushite, liquid ordering at the interface is expected to be quite strong. Brushite crystals were mounted on the sample holder in the growth chamber[1], covered with a drop of saturated growth solution and a Mylar foil. Data were collected at X-ray

energy of 10 KeV. Data collection was very difficult because we have found out quickly that the powerful synchrotron radiation X-ray beam completely destroyed the surface after a few seconds, if measured in air. The only way of measuring was to put 0.5mm Al into the beam and to measure in situ. In this way it seems that equilibrium is established in which the proces of the beam damage and healing of crystal were equally fast.

Under these conditions we were able to measure in total 158 non-equivalent reflections consisting of the (20) and (2-2) rods and specular reflectivity,with the agreement factor of 8% when averaged over all measured conditions. Figure 1 and 2 shows the measured structure factor amplitude along 20 rod and specular reflectivity, respectively.

Our data shows that the surface of brushite terminates with a single water layer while in the specular rod the presence of the second ordered water layer is visible. We have seen a similar behavior in our earlier work on KDP[1].

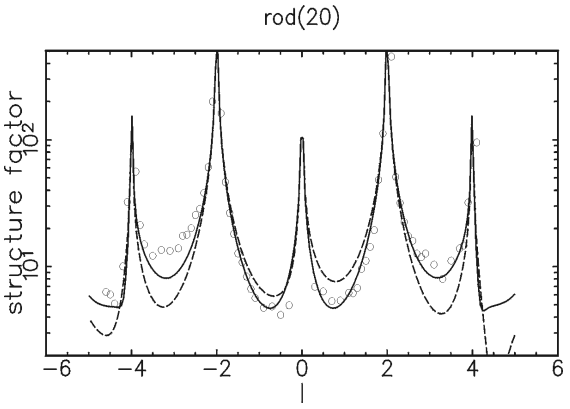

Fig 1. *Structuure factor amplitude along the (20) rod of brushite. Circles represent data points. The solid line represents the best fit for the termination with single water layer. The dashed line is the best fit assuming two water layers on the top.*

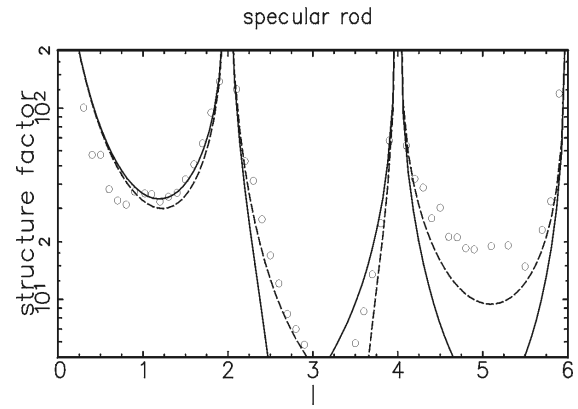

Fig. 2 *The structure factor amplitude along the specular rod, sensitive to out-of-plane ordering of the liquid. Circles represents the data points, solid line a model with one water layer on the top and the dashed line best fit for a model with two ordered water layers at the interface.*

A publication on these results is in preparation.

References:

[1] M.F. Reedijk, J. Arsic, F. F. A. Hollander, S. A. de Vries and E. Vlieg- submitted