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.

	Experiment title: Observation of dislocations in ice multi-crystals	**Experiment number:** **ME-306**
Beamline: ID19	**Date of experiment**: from: 14 Dec 2001 7:00 to:16 Dec 2001 7:00 from: 17 May 2002 7:00 to:20 May 2002 7:00	**Date of report**: **22 August 2002** *Received at ESRF:*
Shifts:	**Local contact(s)**: **José Baruchel, Jürgen Härtwig**	

Names and affiliations of applicants (* indicates experimentalists):

Armelle Philip
Alexis Demongeot
Jacques Meyssonnier

Laboratoire de Glaciologie et de Géophysique de l'Environnement, CNRS
Domaine Universitaire, BP 96
38402 Saint-Martin-d'Hères, cedex (France)

Report:

The aim of this project was to acquire the basic information necessary to prepare further Synchroton X-ray in-situ creep experiments aiming at the observation of dislocation processes acting in the deformation of ice. (for example dislocation-grain boundary interactions such as dislocation sources, pile-ups formation, or obstacles to dislocation glide). This will be very important to understand how the viscoplastic deformation in ice is accommodated, and then to build ice behaviour models to be used in the simulations of polar ice sheets.

Different ice samples were tested in transmission with white and/or monochromatic beam conditions. During its study, each ice specimen was placed in the microtomography cryostat of the ID19 line. The temperature of the cold cell cryostat is fixed at -60¡C.

The observations are still being analysed, however, the main results are as follows:

- Two methods for preparing the ice specimens, in the shape of thin sections with appropriate dimensions were tested: one by using a microtome and the other by using a milling machine. Milling seems to reduce work hardening in ice (fewer crystallographic defaults were observed).

- Different thicknesses of the ice thin sections were looked at. We concluded that the ice samples must be less than 1mm thick in order to observe individual dislocations (see photo˚1) ; however under 1mm in thickness great difficulties are encountered for machining the specimens.

- Sublimation of ice occurs in the cold cell in which a flux of nitrogen at approximately -20¡C is circulated while the temperature of the cryostat (located at the bottom of the cell) is -60¡C. Of course sublimation of the upper part of the thin sections (far from the copper holder) was especially worrying for samples l mm thick. For the observation of thinner ice sections, we tried to coat them with silicon grease, which does not interact with X-rays. Another possibility is also to insert the ice in an adjusted plastic case.

- The glue used to fix the ice sections on the cryostat copper holder was thermally conductive grease. However the temperature gradient between the copper holder and the cell nitrogen atmosphere leads to frost deposition on the copper holder and the lower part of the specimen in contact with it. Using conductive grease enhances this phenomenon. Furthermore, since the handling of such small ice specimens is not easy, it is difficult to control the shape of the glue joint, which can eventually spread too much onto the ice surface. Then the frost film interacting with X-Rays deteriorates the resolution.

These preliminary results obtained in December 2001 allowed us to fix the design of a compression device to study the beginning of plastic deformation in ice. This set up has an upper moving blade loading a thin plate of ice. To avoid buckling and sublimation, the ice thin section is inserted between two plastic plates transparent to X-Rays (Macrolon). The ice section dimensions are 1x15x20 mm^3. This device can fit in with the cold cell of the ID˚19 line, slightly modified to increase the cell volume.

In order to test this apparatus, a few single crystals and bi-crystals were deformed under different load levels in May 2002. It was possible to observe the evolution of the ice microstructure during loading. For example, a single crystal with the c-axis at 75¡ from the compression axis developed a ribbon like feature approximately 1mm thick, almost perpendicular to the basal slip lines, which propagated in the direction of the basal lines at a velocity compatible with that expected for basal dislocations under the applied temperature and loading conditions (see fig. 2).

These first results on the observation of individual dislocations and of dislocation displacements are very encouraging and future experiments must be performed to progress in the understanding of dislocations movement and of dislocations interaction with grain boundaries in relation to the viscoplastic deformation of ice.

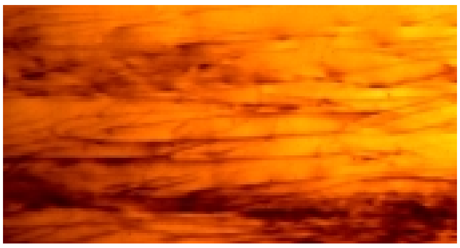

Figure 1: Dislocations in a prismatic diffraction plane observed in a 1mm thick ice thin section (monochromatic beam, λ=0.69).

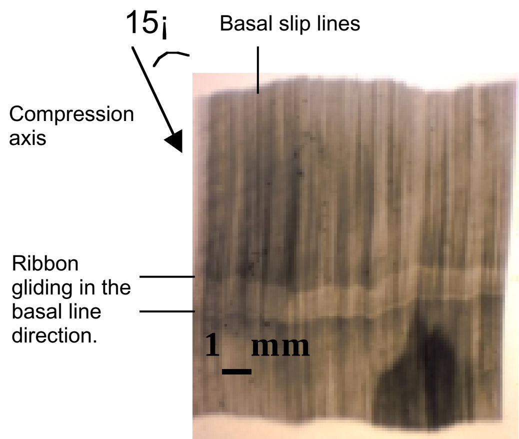

Figure 2: Microstructural features in a prismatic plane (white beam).