	Experiment title: Synchroton X-Ray topographic study of dislocation-grain boundary interaction in ice	**Experiment number**: ME-579
Beamline: ID-19	**Date of experiment**: from: 1 August. 2003 7:00 to: 4 August 2003 7:00	**Date of report**:
Shifts: 9	**Local contact(s)**: **Jürgen Härtwig**	*Received at ESRF:*

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Report:

The experiments that were performed at ESRF in August 2003 are part of a project aiming at a better understanding of the mechanisms involved in the viscoplastic deformation of ice. The ice crystal exhibits a very strong viscoplastic anisotropy which induces strain incompatibilities between grains in the polycristal of ice. These incompatibilities lead to strain heterogeneities inside each grain. Grain boundaries actually play an important role as effective sources of dislocations and as strong obstacles to their motion [Liu, 1995], and also in the nucleation of new grains (i.e. recrystallization) [Mansuy, 2001].

Since the problem of heterogeneous deformation is too complex to be hopefully addressed during a single 3 or 4 days period, we focused on two main topics:

- first, the characterization of laboratory grown ice crystals in terms of dislocation density,
- second, the evolution of the density of dislocations and their repartition inside the grains during compression tests.

The preliminary results obtained during previous experiments done at the ID 19 beam line (report ME 306) allowed us to fix the experimental setting. The crystal specimens are thin plates of dimension $1x17x21mm^3$, made by using a milling machine. These thin sections are tested in compression with a micro press installed inside the ID19 cold cell. To avoid buckling and sublimation during deformation, the ice thin sections were inserted between two plastic plates transparent to X-rays (Makrolon).

1. Ice single crystal experiments

Since our previous experiments showed very large variation in dislocation densities in different undeformed laboratory grown crystals, several growth conditions were tested in order to identify a growth protocol that results in a low dislocation density. Two main techniques were studied :

- Method 1: the ice single crystals were cut from one grain in a large grained polycristal of columnar ice grown from a seed of the same type of ice: during growth in a vertical temperature gradient the grains with a horizontal c axis increase in size (at the expense of their neighbours) so that it is possible to obtain big column-shaped grains about 50 mm in diameter and more than 10 cm long.
- Method 2: the ice single crystal is grown from a mono-crystalline seed stuck on the bottom of a cylindrical container with a latex wall. This expansible wall is expected to decrease the residual stresses that might result from the increase in volume during the water – ice phase change.

For both methods, de-ionized water was used and a vertical temperature gradient was applied for the growth.

Each undeformed ice crystal was placed in the ID19 line cold cell then was analysed in transmission with white and/or monochromatic beam conditions. The results obtained so far are not conclusive : Method 1 generates grains with an extremely variable dislocation density while the only crystal analysed that was grown with Method 2 shows a dislocation density that is too high to allow observation of individual dislocations.

2. Tri-crystal experiments

Compression tests were performed on tri-crystals in order to study the effect of grain boundaries and triple junctions on the viscoplastic deformation of polycrystalline ice.

A compression device specially designed to fit in the ID 19 cryogenic cell (slightly modified to increase the cell volume) was used.

Only a few ice specimens were analysed because each tri-crystal test lasted at least 10 hours. This included the determination of the initial crystallographic structure, then the recording of the evolution of the tri-crystal microstructure. The applied compressive stress was in the range from 0.025 MPa to 0.1 MPa. Since the mobility of dislocations in ice is very low, compared to that of metals even at a temperature near melting point (about $5\mu m\ s^{-1}\ MPa^{-1}$ at –15°C, i.e. $T=0.95\ T_m$), and we wanted to go far enough in the deformation (as far as 10%) so as to observe marked changes in the microstructure, each mechanical test took a very long time.

Only the polychromatic light was used for these crystals because

- the white radiation gives reflections on different crystallographic planes for the same grain, the comparison of which allows to obtain more information on the observed dislocation features;
- moreover, in the present case of a tri-crystal, the white beam can allow to study the two or three neighbouring grains simultaneously under Bragg condition, which is particularly interesting for grain boundary and triple junction studies.

These first experiments on tri-crystals show that

- the grain boundary appears to be the place of strong crystallographic distortions that depend on the crystallographic orientations of the neighbouring grains relative to the axis of compression and relative to each other ;
- dislocation bands (organized structures) start at the grain boundary and propagate inside the grains roughly perpendicular to the basal plane of each grain.

Liu F. , Baker I. and Dudley M. (1995), *Phil.Mag.A*, vol.71, No.1, 15-42.

Mansuy Ph., Philip A. and Meyssonnier J. (2001), J.Phys.IV, vol.11, EDP Sciences Les Ulis, pp. Pr4-267,Pr4-274.