ESRF	**Experiment title:** Synchroton X-Ray topography study of dislocation-grain boundary interaction in ice	**Experiment number**: HS-2249
Beamline: ID-19	**Date of experiment**: from: 14 December. 2003 7:00 to: 18 December 2003 7:00	**Date of report**: 16 March 2004
Shifts: 9	**Local contact(s)**: **Jürgen Härtwig**	*Received at ESRF:*

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Report:

Viscoplastic deformation of hexagonal ice (Ih) derives mainly from dislocation glide on the basal plane. This particularity results in a very strong viscoplastic anisotropy that induces strong strain incompatibilities between grains and high stress concentrations in the vicinity of grain boundaries in polycrystalline ice. These lead to a heterogeneous intra-granular strain field, then to a heterogeneous crystallographic distortion field, in each grain of the polycrystal. In order to understand the evolution of the ice microstructure resulting from the distortion and the dislocation organization, in-situ compression tests have been performed at ID 19 beam line, using a specially designed micro press (see ESRF report ME-306 and ME-579 for the description of the device).

Three types of ice specimens have been studied : single crystals, tri-crystals and multi crystals in order to enlighten the role of grain-boundaries and triple junctions. The specimens were thin sections ($21x17x1\ mm^3$) made from ice grown in our laboratory. These thin sections were inserted between two plastic plates and compressed in their plane (2D-plane strain state; see report ME-306 and ME-579). Because each experiment takes quite a long time (about 10 hours) for the X-Ray determination of the initial microstructure and for the recording of its evolution during the compression test (the mobility of dislocations in ice is very low about $5\mu m\ s^{-1}\ MPa^{-1}$ at –15°C, i.e. $T=0.95\ T_m$), only a few tests could be performed in the nine allotted shifts. Consequently we chose, for all the compression tests, to have one of the crystals with the same crystallographic orientation, namely the hexagonal symmetry axis lying in the plane of the specimen, at about 10° of the compression axis. This allows the effect of the grain boundary misorientation to be studied, starting from a single crystal to a grain completely surrounded by other grains.

Polychromatic light was essentially used because it provides topographs on different crystallographic planes for the same grain, and may also give simultaneous topographs for neighbouring grains (if the misorientation is well chosen). Then, the 3D crystallographic distortion in a grain as a function of the distortion of the other grains can be studied. In order to limit crystal defect superimposition on topographs, the thickness of the ice specimens was limited to 1 mm.

The in-situ X-ray compression tests have been accompanied by compression experiments performed in our laboratory with the same configuration as that used at ESRF, namely the micro-press and the ice specimens with nearly the same microstructure (thin sections cut from the same ice batch). The surface observations were done using cross polarized light, which allows to analyse the evolutions of the crystallographic orientations, grain boundaries and triple junctions (see figure 1). The experiments with cross polarized light have the advantage to be simpler to set up, while only a few experiments can be done at the highly demanded ID19 beam line.

Preliminary analysis of these few experiments, show that

- A good agreement between X-ray and polarized light experiments is observed in the zones of high heterogeneous deformation and high crystallographic distortions (see figures 1 and 2).

- Owing to the stress concentrations near grain-boundary, multi-crystals deform easier than tri-crystals that deform easier than a single crystal.

- The deformation leading to crystallographic distortions takes place first near grain boundaries. The distortion intensities depend on the crystallographic orientations of the neighbouring grains relative to the axis of compression and relative to each other.

- For some observed grains dislocations organize themselves in bands, and glide from the grain boundary into the grains, roughly perpendicular to the basal plane of each grain. The local stress tensor must be determined (by numerical finite element simulations) in order to analyse the appearance of these dislocation features.

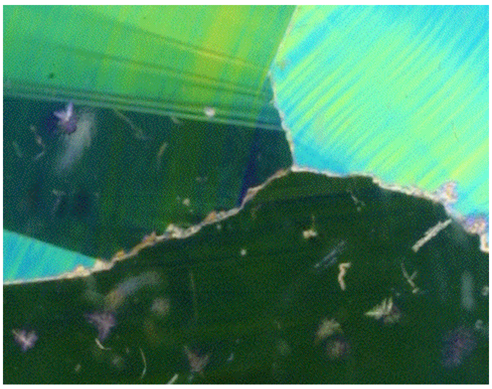

Figure 1 : Photograph of a tri-crystal undergoing a compression of 0.1 MPa in our laboratory cold room (cross polarized light; same device as that used at ID19).

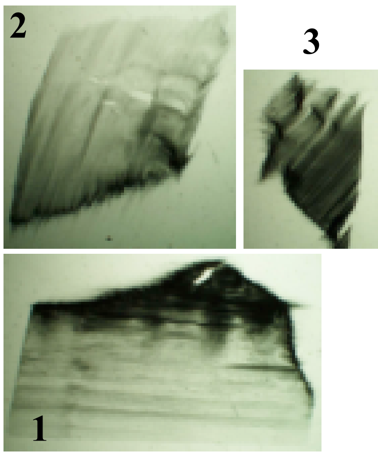

Figure 2 : Topographs of the three grains of the tri-crystal corresponding to that shown on Figure 1 (specimen cut from the same batch), undergoing a in-situ compression of 0.1 MPa : **1)** Prismatic plane **[$\bar{1}$100]**; **2)** Prismatic plane **[$\bar{1}$2$\bar{1}$0]**; **3)** Prismatic plane **[$\bar{1}$100]**.