ESRF	In situ observations of dislocation-grain boundary interaction in ice during compression tests	**Experiment number**: ME-1247
Beamline: ID-19	**Date of experiment**: from: 26/08/2005 at 8:00 to 29/08/2005 at 8:00 from: 12/10/2005 at 8:00 to 13/10/2005 at 8:00	**Date of report**: 25 February 2006
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Report:

The aim of this project is to better understand the viscoplastic deformation of ice. Previous works [Nakaya, 1958] have shown that the single crystal of hexagonal ice mainly deforms by basal dislocation glide leading to a very strong anisotropic behaviour. However, there is still little information on the physical mechanisms involved in the accommodation of strain incompatibility between anisotropic grains of polycrystalline ice.
Because we are interested in the microstructural evolution of ice during its deformation, in-situ X-ray topographic observations of compression tests on ice tri-crystals have been performed. The micro press that was especially designed to fit in the cold cell of the ID 19 beam line, was used (ESRF ME-306, ME-579 and HS-2249). It allows following the deformation, under X-ray exposure, of a thin section of ice (20x17x1 mm^3) undergoing plane strain compression, without removing the sample from the goniometer. Polychromatic light was used because the white beam provides reflections on different crystallographic planes for the same grain, the comparison of which allows obtaining more information on the 3D crystallographic defaults. Also, under special configurations of the relative crystallographic orientations of the grains, the white beam may allow studying the central grain and its neighbours simultaneously, which is particularly interesting for grain boundary and triple junction studies.
Ice multicrystals were grown by controlling the microstructure (size and crystallographic orientation of the grains). Several ice samples with the same microstructure were cut from the same ice batches in order to compare in-situ X-ray results with observations made during compression tests performed in our laboratory cold room with the same loading device placed between crossed polarizers. Polarized light allows observations, such as crystallographic orientation, grain boundary and triple junction evolutions, at the millimetre scale.

First results

Each multicrystal experiment takes a long time (about 10 hours, to determine the initial crystallographic structure, then to record, on photographic films, the microstructural evolution of each grain of the multicrystal). Thus, only a few experiments were performed during the allocated time at ESRF.

First, the quality of our crystals was analyzed by topography: the dislocation density was pretty good, about 10^6 m/m^3 , thus allowing the observation of individual dislocations. During compression, it was possible to follow the displacement of individual dislocations. Their velocity was found to be between 0.5 and 1 µm/s at -10°C under a compression stress of 0.3MPa. Since the resolved shear stress on the basal plane is less than the applied compression stress (see Fig. 1), this is in agreement with the order of magnitude of the mobility of dislocations in ice found in the literature (about 10 µm s^{-1} MPa^{-1} at -10°C [Okada et al., 1999]). During loading the dislocation density increases and it was possible to map the dislocation density evolution in grains. Grain boundaries are the place of stress concentration that appear as black areas limited by white loops in the three topographs corresponding to each tri-crystal grain shown on Figure 2. At the beginning of loading, these black regions seem to be related to the internal elastic strain (or elastic distortion) since they disappear when the external load is removed. However, after the specimen has been loaded for some time, then unloaded, the topographs still exhibit black areas at the grain boundaries: these must be related to the energy stored in the dislocations that accumulate due to the strain incompatibility between grains. When two grains are compatible, as grains 1 and 2 in the tri-crystal presented on Figures 1 and 2, the Burgers vectors of both grains are parallel and less distortion is observed. The observations made during compression tests performed under polarized light (LGGE cold room) are in agreement with that obtained under X-ray : Figure 1 shows that slip lines pass directly through the grain boundary from grain 1 to grain 2, whereas for the other grain boundary the Burgers vectors of grains 1 and 3 are not compatible so the energy stored at the boundary needs to be released by recrystallization.

References

Nakaya U., 1958. Mechanical properties of single crystals of ice. U.S. Snow, Ice and Permafrost Research Establishment. Research report 28, 1-46.

Okada, Y., Hondoh, T. and S. Mae. 1999. Basal glide of dislocations in ice observed by synchrotron radiation topography. *Phil. Mag. A, 97 (11), 2853-2868.*

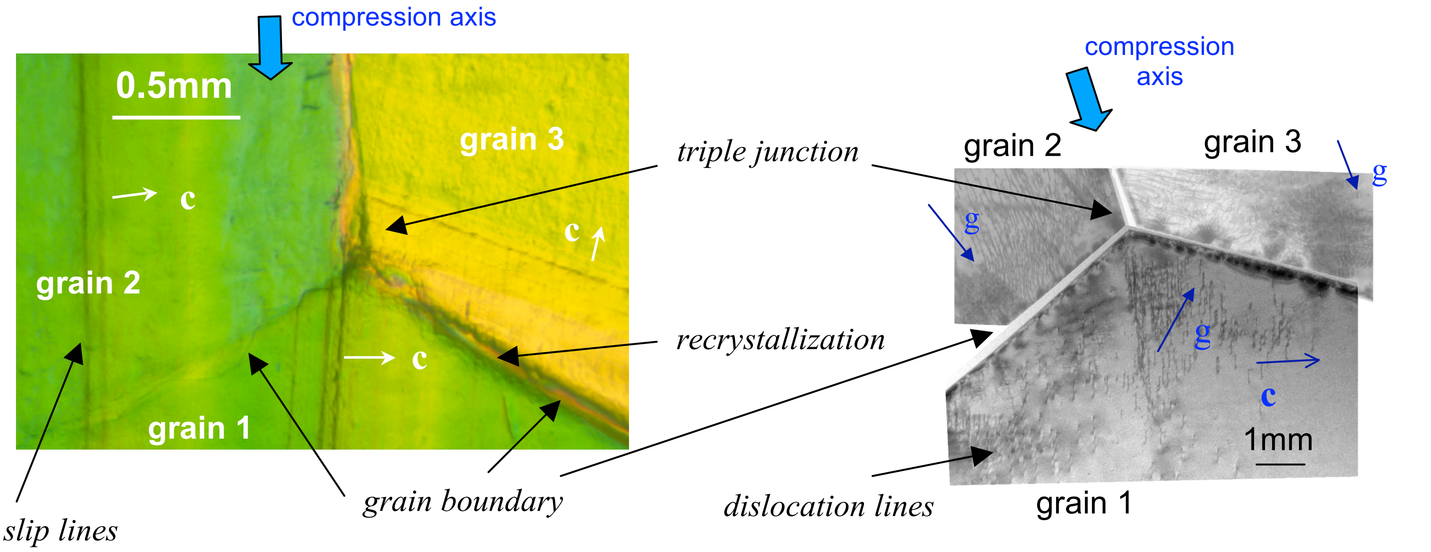

Figure 1: Photograph under polarized light of a tri-crystal undergoing a compression of σ = 0.9 MPa during 10h30mn. The misorientation between grains are given in the table below:

Angles between grains (misorientation)	1-2	1-3	2-3
c axis ($c_i.c_j$)	45°	10°	55°
a axes ($a_i.a_j$)	0°	20°	50°

Figure 2: Topographs of the (1,0,-1,2) planes of the 3 grains of the tri-crystal with the same microstructure as that of the tri-crystal shown on Figure 1. The compression stress is 0.3 MPa, during 15mn.