	Experiment title: Strain gradients and geometrically necessary dislocations : a study in deformed ice single crystals.	**Experiment number**: ME 793
Beamline: ID 19	**Date of experiment**: from: 30 june 2004 to: 02 july 2004	**Date of report**: 16 march 2005
Shifts: 6	**Local contact(s)**: Jurgen Härtwig	*Received at ESRF:*

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Report:

There has great interest in developing mechanism-based theories of strain gradient plasticity including an internal length scale to describe the deformation of crystalline materials on the micron scale (Fleck et al., 1994; Fleck and Hutchinson, 1997; Nix and Gao, 1998). In torsion, strength increases with decreasing wire diameter. These results are interpreted in terms of geometrically necessary dislocations (GND) accommodating plastic strain gradients. Strengthening effects arising from strain gradients are generally observed on *the micron scale*.

Due to the very large viscoplastic anisotropy of the crystal, ice appears to be a material for which the density of GND is much higher than that of statistically distributed dislocations *on a length scale higher than one centimetre.* Strain gradients associated with the storage of GND appear a general feature of the deformation microstructure of ice. The lattice distortion of glacier ice crystals determined by hard X-ray diffraction is related to the bending of the basal plane and the torsion around the c-axis (Montagnat et al., 2003a). This lattice distortion is shown to be compatible with the basal dislocations generally observed in ice. The large difference in strain rate between crystals oriented for basal slip (soft grains) and hard grains is at the origin of the build up of a non-uniform internal stress field in the polycrystal (Duval et al., 1983), which is partially relieved by the formation of strain gradients.

In this context, several experiments were carried out on ice single crystals deformed in torsion. First hard X ray diffraction experiments performed on such samples at the Institute Laue Langevin (Hamelin and Bastie, 2002) have shown a clear relationship between the density of GND and strain gradients (Montagnat et al., 2003b). The torsion strain appears to be totally accommodated by geometrically necessary basal dislocations. The variation of the calculated density of GND with the diameter of samples can be considered as a real size effect, which supports the assumption on a very large internal length scale in ice. Basal screw dislocations are invoked to accommodate strain gradients in torsion since the bending of basal planes is not required in such experiments. But the former technique does not allow reaching such a microscopic scale.

So in a next step, we used the X-ray topography technique on the ID 19 beamline to check assumptions on the dislocations accommodating the torsion strain. An obvious difficulty was the relatively high dislocation density, which was between $4x10^9/m^2$ and $2x10^{10}/m^2$ in the experiments carried out by Montagnat et al. (2003b). To overcome this obstacle, we stopped torsion tests at different low levels of strain compatible with topographic observations. The torsion experiments were performed at -15° C on ice single crystals grown in the laboratory. The initial dislocation density was expected to be less than $1x10^8/m^2$ (Higashi, 1988). Cylindrical samples with the c-axis along the torsion axis were deformed for strain varying between 0.1 and 5 %. With such a configuration, basal plane is parallel to the permanent shear plane. Basal slip is therefore the only deformation mode. Thin sections of these deformed samples were analysed by both the topography technique and the Laue hard X-ray technique.

Fig. 1.

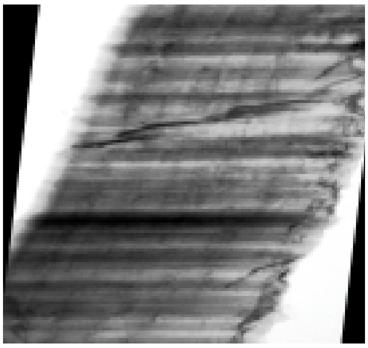
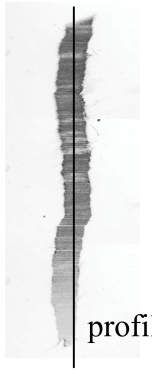


Fig. 2.



The figure 1 shows the white beam topography of one of the Laue spots corresponding to a prismatic reflection obtained on a slightly deformed sample. The torsion of the lattice planes around the c-axis is given by the deviation of the edge of the crystal from the vertical and by comparison with the macroscopic deformation curve allows to calculate the density of GND. The observation in deformed samples of slip lines parallel to the basal plane shows the localisation of the dislocations. Such a localisation is the signature of co-operative movements of the dislocations. Fourier analysis were done on intensity profiles taken along prismatic reflection patterns limited by a thin slit (figure 2) (this allows to reduce the surface artifacts). The distribution of the dislocation arrangements presents a scaling invariance indicating long-range interactions (at the scale of the sample). Therefore the internal stress field at large scale imposes the arrangement of dislocations, at the sample scale and down to the local scale, as proven by the anti-persistent nature of the signal (intensity profile). This “anti-persistency” is the signature of locally anti-correlated dislocation density gradients. A paper is under preparation to present these results (Montagnat et al.).

These analyses deduced from the X-ray diffraction study associated with dislocation dynamic simulations (master work of Juliette Chevy, LGGE) provided a better understanding of the interaction between dislocation glide systems during deformation of ice single crystal.

The obtained results are essentials for the understanding of internal stresses impact on the arrangement of geometrically necessary dislocations when ice is deformed under imposed plastic strain gradients.

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