ESRF	Dislocation density evolution during heterogeneous deformation of an ice multicrystal	**Experiment number**: HS-2931
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Report:

The objective of this work was to quantify the evolution of the dislocation density during the heterogeneous deformation of an ice multicrystal. During the experiment multicrystals of ice were deformed by in-situ compression under X-rays. The extreme anisotropy of the ice crystal viscoplastic behaviour, due to the fact that dislocations glide essentially on the basal planes, induces strain incompatibilities between grains, leading to intragranular heterogeneity. In this study, we were specially interested in the evolution of the heterogeneous crystallographic distortions that develop inside the ice crystal and in interpreting these distortions in terms of the geometrically necessary dislocations needed to bent or twist the crystal lattice.

The tricrystals were cut from batches of columnar ice (lake ice type with column shaped grains) grown by freezing deionized water in a vertical temperature gradient. The specimen was placed in a compression device specially design to fit in the cold cell of the ID-19 beam line. First the initial microstructure of the ice tricrystal was analysed with polychromatic X-rays. Polychromatic light is required to acquire simultaneously the topographs of at least two diffracting planes per grain, as well as the two diffracted images of the 3 grains of the tri-crystal. This is essential for studying the grain to grain interactions at the grain boundaries and triple junction. The reticulography method was used which consists in using an absorbent grid placed in front of the specimen in order to split the incident beam in a number of sub-beams. Each diffraction spot is thus split into sub-regions and it is possible to calculate the lattice orientation at the grid scale in the grains of a multicrystal. The grid dimension was 1mm x1mm. From the lattice distortion it was then possible to calculate the density of geometrically necessary dislocations at each grid point [L.Capolo, 2007].

The crystal quality was very good, since the dislocation density (GND) was less than $10^7 m/m^3$ before the compression tests. During the deformation, the lattice distortion evolves and so does the dislocation density distribution. One example of topographs obtained with a split x-ray beam is shown in Figure 1 for a grain of a tricrystal before compression and after one hour under 0.6 MPa. Figure 2 shows the distribution of the

GNDs density calculated from the mapping of the crystallographic orientations obtained from the topographs on Fig. 1. These results, as well as the others that were obtained with different specimens (5 multi-crystals and 3 single-crystals have been tested during the alloted time) show the heterogeneous evolution of the distortions that can be quantified by a density of geometrically necessary dislocations. Due to the deformation incompatibility between the grains of a multicrystal, the GNDs density is higher at the vicinity of the grain boundaries and at the triple junction. The current study has shown the feasibility of the X-ray topography reticulography method. However other multicrystal specimen must be studied to obtain more information on the influence of crystal orientation and of the misorientation between grains on the dislocation density evolution in polycrystalline ice.

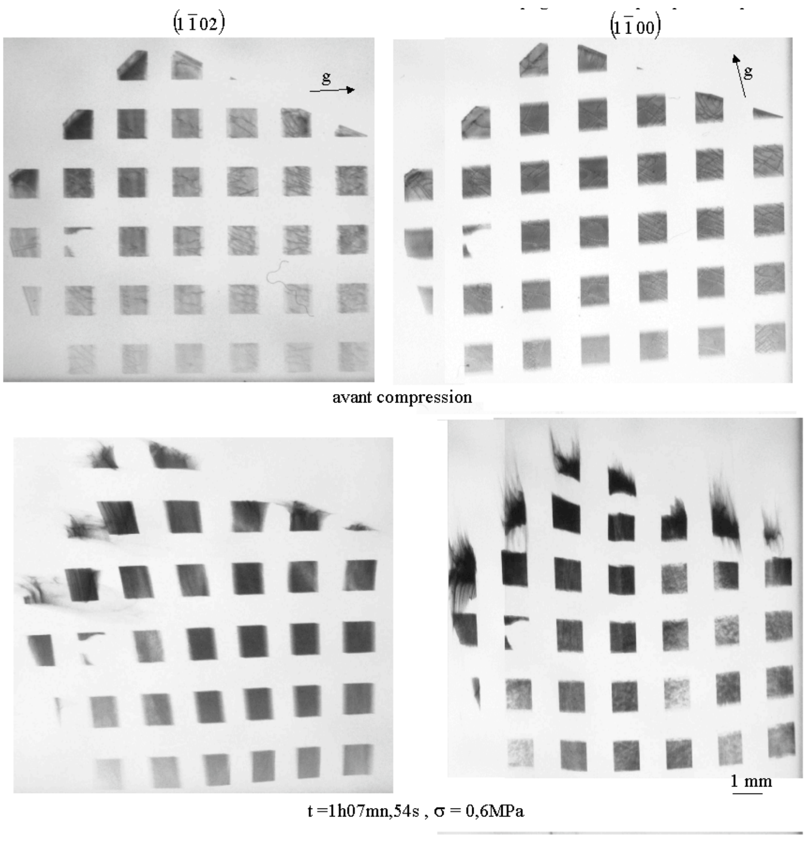

Figure 1: Topographs of a pyramidal plane and a prismatic plane before compression and after 1 hour of compression at 0.6MPa.

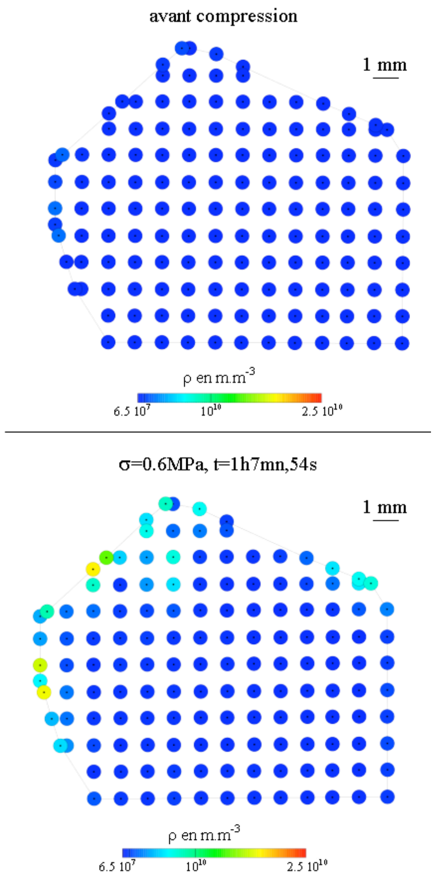

Figure 2: Geometrically necessary dislocation density repartition determined from the pyramidal and prismatic topographs

Capolo L.. 2007.*Contribution à l'étude des hétérogénéités de déformation viscoplastique de la glace Ih mono et multi cristalline : essais de compression in-situ sous rayonnement X*, Thèse Université Joseph Fourier.