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|---|---|---|
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## Report:

A better knowledge of the mechanical behaviour of polycrystalline ice is essential for improving ice cap simulations as well as ice-structure interaction previsions. In order to study the physical mechanisms involved in polycrystalline ice deformation, we need to test laboratory grown ice crystals of a good quality. The aim of this project was to determine a growth method for ice crystals and a specimen-preparation protocol resulting in ice with a low dislocation density.
Crystal quality was analysed by X-ray diffraction, using two different methods to optimise the analyses. The first is diffraction topography in transmission at the ESRF-ID19 beamline. This method reveals defects within the crystal volume and thus can allow to image individual dislocations and to determine their Burgers vectors by analysing topographs of different crystallographic planes. The second method, performed at Institut Laue Langevin, is based on the principle of focused hard X-rays, and gives global information on defects by analysing the shape of the diffracted X-ray beam [Bastie and Hamelin, 1996].

The main results from these studies are :
- From previous works (ME-306, ME-579) we know that it is possible to obtain very good crystals by freezing ultra-pure de-ionized water in a tank placed on a refrigerated table with adjustable temperature. In the vertical temperature gradient, columnar ice (lake ice type with column shaped grains) is obtained. 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, up to 80mm in diameter. Different conditions, as growth rate and using a seed or no seed, were tested. Because we are interested in studying the deformation of polycrystalline ice, we need to obtain a group of grains with good crystalline quality and not only one single crystal. The best crystal quality is obtained when at the beginning of growth, no seed is used and the growth rate is less than 10 mm per day.
- Tri-crystalline specimens were cut in the shape of thin sections (21x17x1 $mm^3$) by using a milling machine. If the milling tool is well sharpened and the cutting speed well chosen, few crystallographic defaults are observed. Cylindrical specimens 13mm in diameter, machined with a lathe, were also tested but their central zone is too thick to allow observing dislocations in good conditions (too much superimposed dislocations).

- In order to limit sublimation during X-ray diffraction experiments, the thin sections of ice were inserted in an adjusted box transparent to X-rays (Makrolon).
- Ageing at -10°C was also studied by analysing ice specimens grown at different dates (the longest time between growth and X-ray analysis was 6 months) and machined at different dates (the longest time between specimen preparation and X-ray experiment was 3 months). In the tested conditions, neither one nor the other seems to make a noticeable difference as concerns the dislocation density. Moreover, although a better surface seems to be observed if the machining is done few months before (surely owing to a slight sublimation), thus allowing a better observation of dislocations on topographs, too few experiments have been done to reach a definitive conclusion.

In conclusion, different growing and specimen preparation conditions have been tested. Although it was possible to test only a limited number of specimens during the allotted beam time (breakdown of the X-ray beam for 5 hours), it is possible to reach optimum conditions for growth, specimen shape, machining, preservation and storage time prior to testing, that lead to crystals with a low dislocation density. An example (grain A in Fig.1) is presented on Figs 2 and 3. The ice specimen exhibits a triple junction close to equilibrium (grain boundaries at angles close to 120°). Because our interest is in the movement of dislocations, dislocations multiplication and interaction during the deformation of ice, the quality of such a specimen, with its low dislocation density, should allow us to follow the movement of individual dislocations under an applied stress.


**Figure 1** :Photograph of a tri-crystal, with cross-polarized light. (A : studied grain shown on figures 2 and 3)

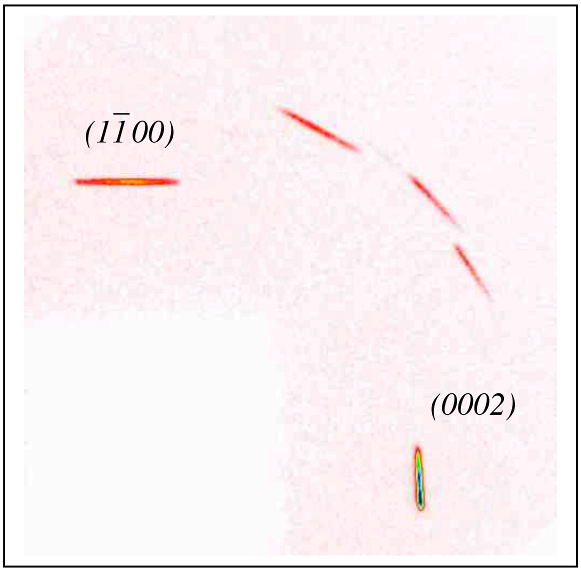


**Figure 2** : Diffraction spots obtained by re-focalized hard X-ray diffraction. The broadening of the spot lines allows to quantify the crystal mosaicity, while the curvature of the spot (or eventually its splitting) gives the crystallographic lattice curvature. For this studied grain, the spots are quite straight with a small broadening (of the order of the focussing error of the X-ray generator accounting for the crystal thickness of 1mm) This indicates that the crystal is quasi-perfect [Bastie & Hamelin, 1996].

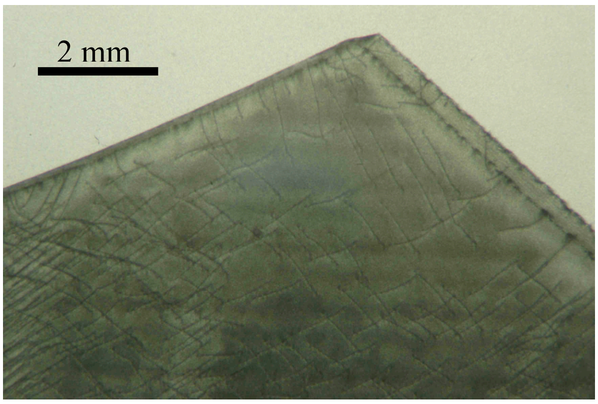


**Figure 3** : Topograph of the prismatic plane $(1\bar{1}00)$ of grain A in Figure 1.

**Ref :** Bastie P., Hamelin B., 1996. La méthode de Laue refocalisée à haute énergie : une technique d'étude en volume des monocristaux, *J.Phys.IV, Colloque C4*, vol. 6, pp. C4-13,C4-21.