ESRF	**Experiment title:** **Quantification of the evolution of the microstructure of ice multicrystals under compression, by in-situ Bragg diffraction imaging**	**Experiment number**: MA - 621
Beamline: BM05	**Date of experiment**: from: 01/10/2008 to: 03/10/2008 from: 05/12/2008 to: 08/12/2008	**Date of report**: 03/09/2009
Shifts: 15	**Local contact(s)**: José Baruchel	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists):		
Kluender, R. * ; Meyssonnier, J.* ; Brutel-Vuilmet, C.*; Philip, A. * ; Baruchel, J. * ;		

Report:

Objectives and methodology:

The aim of the experiment was the in situ measurement of the strain anisotropy in crystalline ice during their mechanical deformation (see proposal). We started with the simplest case, i.e. single crystal samples. The crystals were firstly illuminated with a white beam, and several reflections were recorded on a film (White beam topography). The reflections were indexed and one or two of them were chosen to be analysed with the rocking curve imaging (RCI) method. This method [1] has a higher strain sensitivity and reveals the lattice plane distortion. The experiment was performed in Laue (transmission) geometry, compatible with the sample environment (cryostat, compression device). However the Laue geometry implies further technical and analytical problems: a RCI scan measures one of the two vectorial components of the lattice misorientation. In the case of Bragg (reflection) geometry the second component is usually measured in a second scan where the sample is reoriented in a way such that the diffracting lattice planes turn by 90° [2]. In Laue geometry this would turns to be usually unfeasible because the X-ray beam would have to go through very large (centimeter) material thickness. An alternative way to measure the second component is based on the measurement of the spatial deflection of the diffracted beam associated to this second component. Fig.(1) illustrates the way a lattice distortion perpendicular to the incoming beam deflects the diffracted beam.

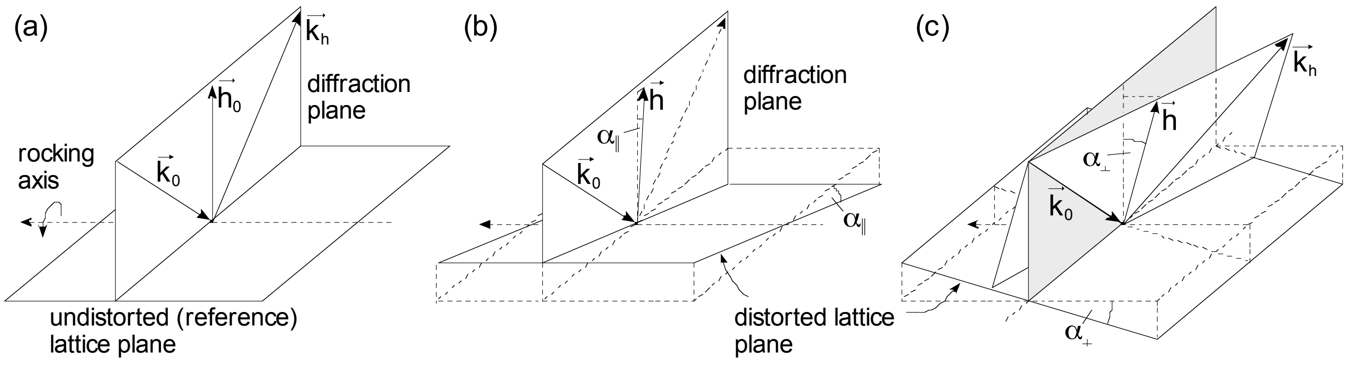

Figure (1) left: the X-ray beam is diffracted by an undistorted lattice plane if the Bragg equation is fullfiled; centre: the component of the diffraction vector parallel to beam does not deflect the diffracted beam, however the beam is diffracted under a different incoming angle; right: the component of the diffraction vector perpendicular to the beam nearly does not modify the incoming angle under which the beam is diffracted, however the diffracted beam is deflected. The deflection can be measured on a 2d detector (camera)

In addition dispersive effects of the beam have to be taken into account because they locally shift the Bragg peak. The angular and wavelength dispersion was calibrated with the aid of additional RCI scans of a perfect Si crystal. These experiments about dispersive effects on a synchrotron radiation diffraction topography transmission experiment were performed as inhouse research, and are being processed to be published .

To measure both the rocking curve images and their deflection we used a grid providing multiple 50µm x 50µm dot beams, separated by one millimetre, over more than 1 cm^2. This means that for a scan of entire sample 20 x 20 = 400 RCI scans should be performed, a task that would require a huge amount of beamtime. In addition, over such a long time, the temperature stability of the sample cannot be retained. We therefore restricted the scans to smaller zones, which were repeated after each compression of the sample.

Preliminary Results:

The analysis of the lattice distortion in depth of the single crystal is in progress, but is not advanced enough to be presented here. This analysis is a necessary step before going to the more interesting, but difficult, tricrystal case. Some preliminary results showing the entire image (spatially extended beam), whereas not providing the wished quantitative information about the sample lattice misorientation in depth, are nevertheless presented her because they constitute a first approach, allowing a qualitative analysis, of what is happening in the crystal during its deformation. Examples are presented in fig.(2) and fig.(3).

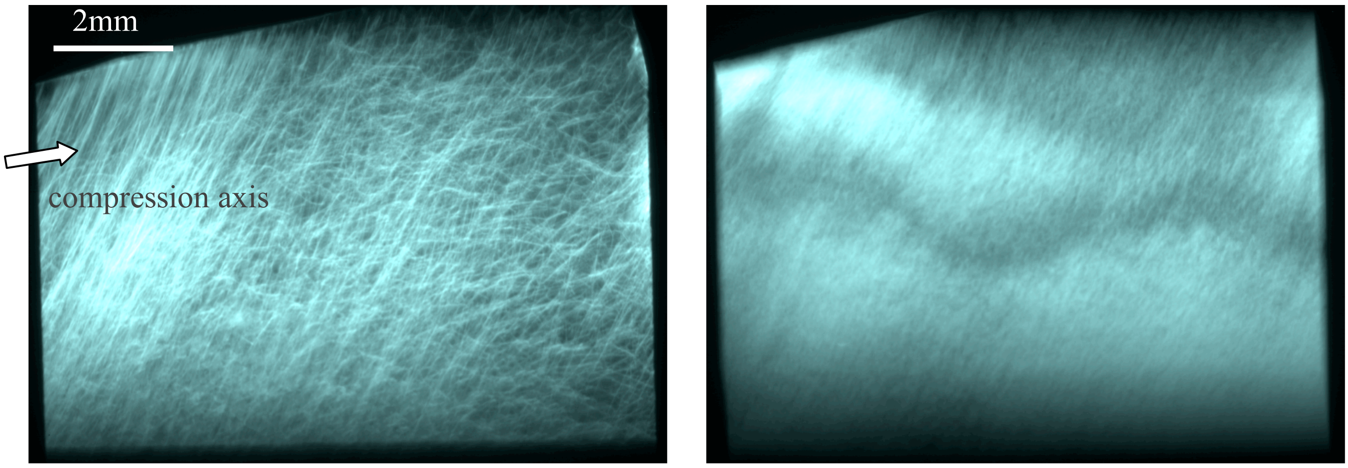

Figure (2) Integrated images of the 2-201 pyramidal lattice planes of an ice single crystal, in its initial state (left) and after 15 minutes of compression with 0.5MPa (right). The beam energy was 18 keV. One can see the rise of dislocation density, slip bands can be observed.

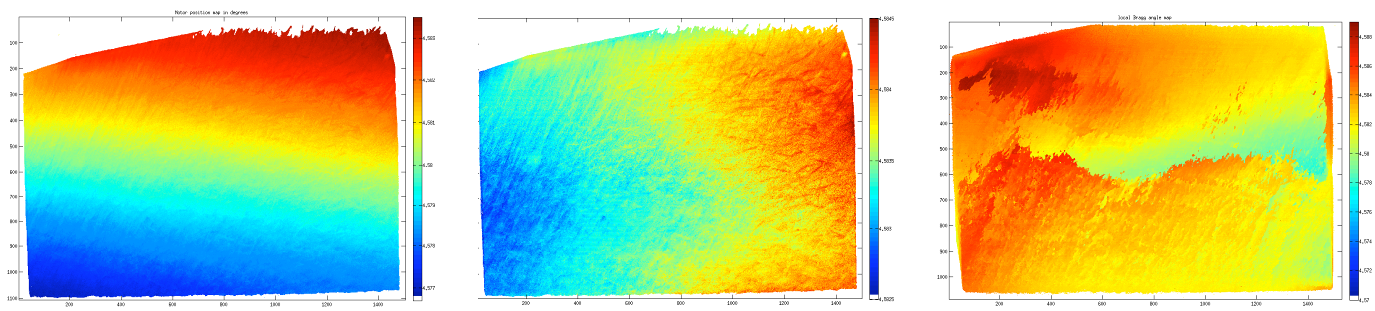

Figure (3) Lattice plane misorientation of the 2-201 pyramidal lattice planes of the ice single crystal from fig.(2). Left: This image corresponds the lattice misorientation at the initial state of the ice crystal, before taking into account the dispersive effects of the beam. The planes appear to be horizontally distorted. Middle: The dispersive effects have been taken into account and one can see that in fact, the sample is vertically distorted. Right: Lattice plane distortion of the same planes, after 15minutes of compression with with 0.5MPa : the deformation introduced two subgrains.
(The scale of colors are not the same in the three images: it looks like the distorsion being of the same order in the three images, which is not true.)

References
[1] D. Lübbert et al., N.I.M. B, **160**, 521 (2000)
[2] P. Mikulik et al., J. Phys. D : Appl. Phys. **36**, A74 (2003)