ESRF	**Experiment title:** Quantification of lattice misorientations in an ice-tricrystal	**Experiment number**: MA 621
Beamline:	**Date of experiment**: from: 30/09/2008 to: 03/10/2008	**Date of report**: March, 18th 2009
Shifts:	**Local contact(s)**: Baruchel, José	*Received at ESRF:*

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The objective of the experiment was establishing the techniques to measure the lattice misorientation of ice tricrystals during a viscoplastic deformation. Ice single crystals have prefered glide lines for plastic deformation. A polycrystal with differently oriented grains will therefore develop a complicated strain field. Our starting technique to locally measure the effective misorientation inside the sample was from Rocking Curve Imaging (RCI) .

Due to the monochromatic illumination in RCI, any deviation from the theoretical Bragg angle is directly correlated to the effective misorientation $\Delta\theta = (\Delta d/d)$ tg $\theta + \delta\theta$, which contains the deformation and rotation components. To determine independently these values, a rocking curve must be recorded twice, where the second one refers to the same lattice planes but rotated by 180 degrees around the lattice plane vector. However we can restrict to a single scan because the deformation component is small with respect to the rotation one in the ice case, and can be neglected to a very good aproximation.

The first part of the experiment was devoted to establishingh the adequate techniques by using LiF single crystals instead of ice samples. This allowed to verify, without the complications associated to the cryostat, that the beam parameters at the beamline BM05 were sufficient to observe a misorientation due to compression as it would occur in ice. The distortion was observable by subtraction of integrated reflection images at different compressions.

The main experiment was executed in the second part in december. Unfortunatelly we had to struggle with several problems at the beamline and managed only to record some rocking curves on ice single crystals in Laue geometry. Two rocking curves, one on the basal planes (0001) and one on the prismatic planes (11-20) were recorded on two crystals. Due to the thickness of the crystals (4 mm) and the insuffficient surface quality, we used micro-

multisections to substract the bulk information from the surface information and to avoid the superposition of images coming from a too large volume (section topography).

ANALYSIS:

To get the local misorientation out of the data, the local rocking curve in every pixel on the detector was fitted and the local Bragg peak maximum was calculated. However several geometrical corrections have to be taken into account. First of all the wavelength dispersion of the Si 111 monochromator and the angular dispersion due to the beam divergence. Then the inclination of the sample towards the incoming beam. Once these corrections are taken into account, we can easily calculate the angular misorientation.

Figure (1) shows a preliminary (the data analysis is still in progress and we will be able to present further misorientation maps of our crystals) corrected first component of the angular misorientation in arcseconds of our first ice crystal. The crystal appears to be bent. This result must be confirmed to be sure that all the adequate corrections had been applied. Therefore we will use some inhouse beamtime to record further rocking curves on a perfect silicon crystal, this additional data allowing calibrating our corrections.

A second problem we had to struggle with, is multiple peaks per pixel that occur, when the sample has subgrains or when beam parts coming from different regions of the crystal are diffracted onto the same pixel of the detector (figure 2). A simple back projection from the detector towards the crystal surface can then no longer be applied and it is not possible to correlate single Bragg peaks to their origin region. The back projection is the biggest task and can only be solved with additional data (turning the diffracting planes by 90 degree or information from other, non perpendicular, lattice planes). However we hope to eliminate to a large extent this multiple Bragg peaks problem by reducing the distance between the detector and the crystal, and by lowering the energy of the X-ray beam. In addition we are in the process of fixing some problems at the level of the experimental device (application and control of the strain on the sample).

The first results are very encouraging, but more beamtime is clearly needed to achieve the objectives described in the proposal.

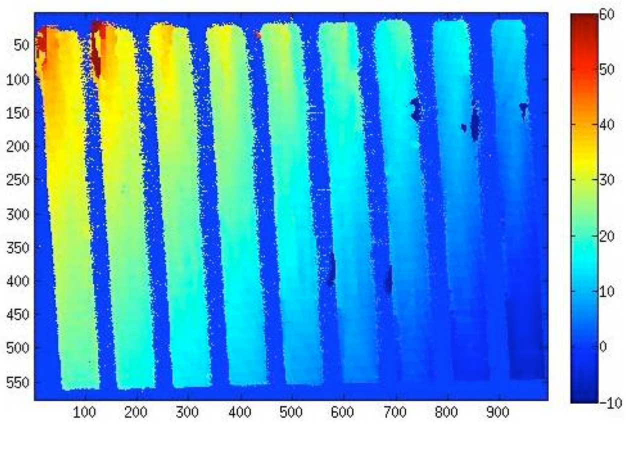

Figure 1: Corrected misorientation (arcsec) measured on "multisections" of an ice crystal

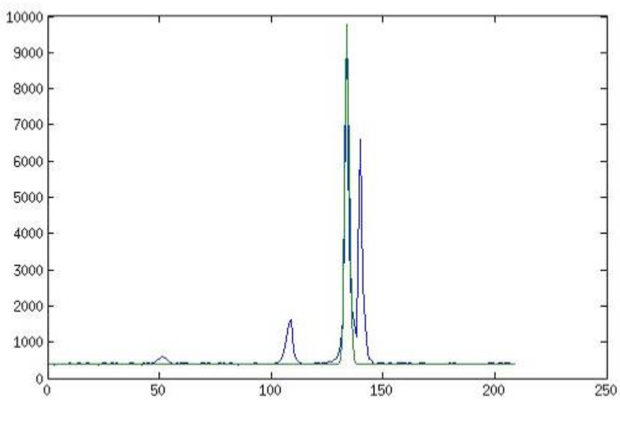

Figure 2: multiple Bragg peaks recorded on a single pixel of the detector.