ESRF

ESRF	**Experiment title:** Recrystallization of ice Ih close to the melting point	**Experiment number**: ME-265
Beamline: ID 11	**Date of experiment**: from: 19/06/2002 to: 24/06/2002	**Date of report**: 16.09.2003
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Report:

Experiment description

The aim of the proposal was to study the change of the morphology and orientation of the ice Ih grains at temperatures closed to the melting using *in situ* synchrotron diffraction and analysing the data with the appropriate software available at ESRF. Artificial and natural ice samples were investigated in the T-range between -5°C and -0.1°C.

Fine-grained ice with an averaged grain size of 50 μm was prepared in Göttingen. The ice powder was placed in thin-walled aluminum cans which were transported to Grenoble at liquid N_2 temperature. A Peltier cooling cryostat specifically built in Göttingen for this experiment was used to keep the temperature of the sample at a constant value during the *in situ* X-ray measurements with a precision of 0.02° degrees.

Time resolved image 2D data were taken with 0.1 degree ω steps (horizontal rotation of the cryostat). The acquisition time was 0.25s per image. Data were collected in a following way: long sets measurements in a range of 90 degree (900 pictures) followed by ten short intervals in a range of 0.5 degrees (10x5 pictures). Series of measurements were performed at temperatures of -5°C, -2°C, -0.5°C, -0.2°C, -0.1°C for a period of around 12h/ per measurement. Fig.1 shows an image taken during one of the measurement at -2°C.

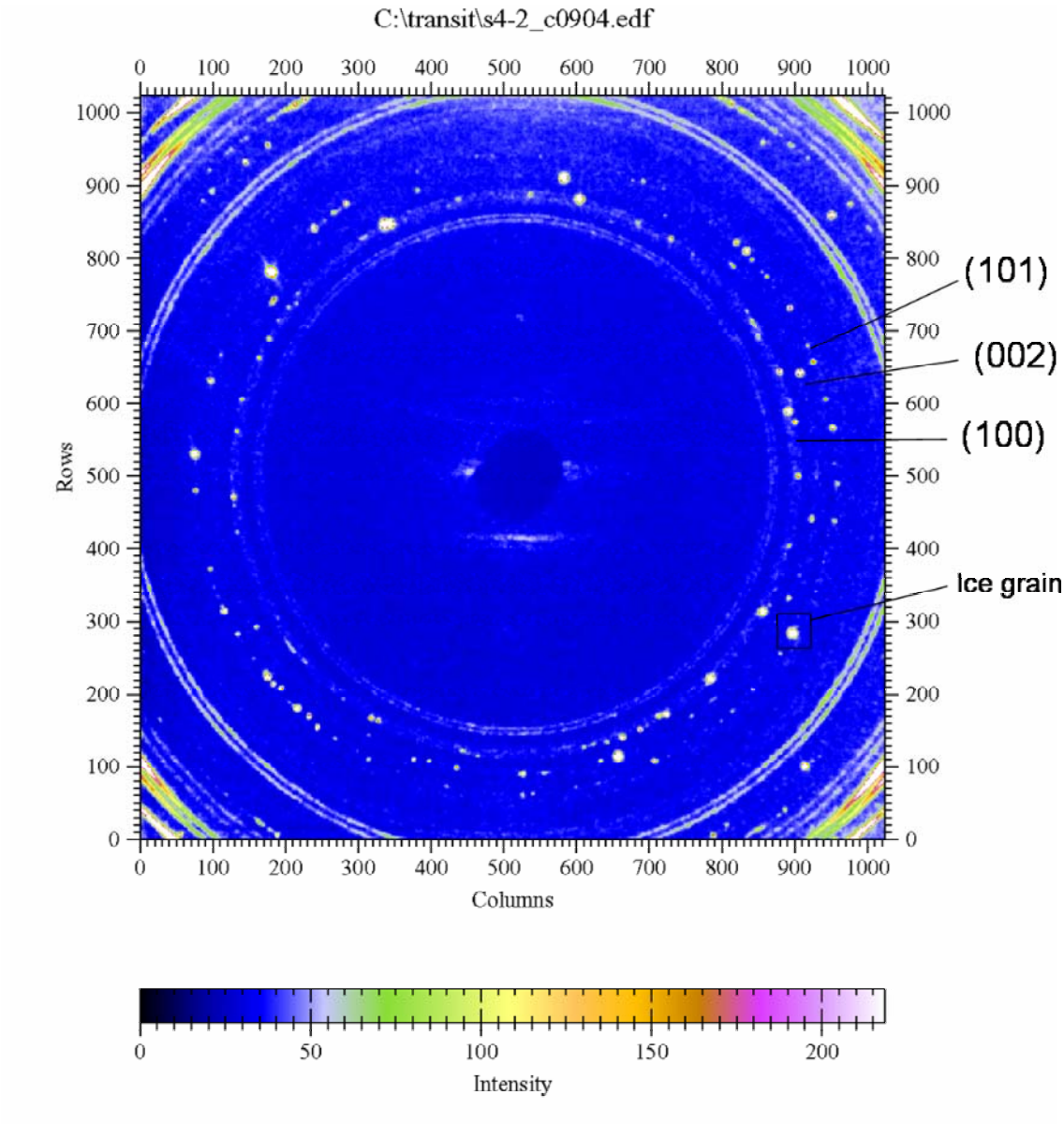

Fig.1 One of the collected images showing the ice Ih sample at -2°C. The squire marks one of the observed ice grains. The numbers in the brackets show the (hkl) Miller indices of the strongest ice lines. The picture belongs to the first range of 0.5 degree measurement (short data set) at this temperature.

Results and Discussion

The FIT2D program was used for a preliminary analysis of the collected images. The initial data analysis was done only in the short ranges. We focused on a few grains in the position ω = 0.3°. The intensity of these grains was recorded as a function of time.

Up to now we can conclude the following:

- At a temperature of -5°C at least one grain can be identified for a period of 21h.
  Some other grains moved out of the reflection position after 2, 3 or 11h.
- At -2°C two grains were followed for 12h (the total measurement duration at this temperature).
- At - 0.5°C and -0.2°C the grains turned out of the reflection position very fast and there was no grain which can be seen in the region of interest for more than 5-6 min.
- During the observed period (-5°C and –2°C) some small change (+ or - 0.1°) in the grain orientation can be detected for some of the grains.
- For the quantitative analysis of the data we observed a serious problem with the integrated intensities. The intensity change with time for all observed grains (-5°C and –2°C- samples) were erratic. There were jumps up and down with amplitudes of a factor 2 to 7 of the initial intensity (even at -5°C), which we believe must be due to some shortcoming of the data collection procedure. We certainly can expect continuous intensity changes and not the erratic behavior but born out of the analysis. We believe that these changes are unphysical and do not come from the sample nor form the FIT2D procedure. There was a problem in the way we took the intensity data. If this problem can be solved by changing the mode of data aquisition we think that experiments following numerous grains should be possible up to -2°C (or possibly -1°C). Closer to the melting point the full scan takes too long for the fast changes observed to successfully apply this procedure.