ESRF	**Experiment title:** 3D imaging of snow microstructure. Intercomparison between X-ray microtomography and microtome serial cut on a same sample.	**Experiment number:** HS-688
Beamline:	**Date of experiment:** 8-9/11/98 and from: 27/11/98 to: 01/12/98	**Date of report:**
Shifts: 3 + 12	**Local contact(s):** W. Ludwig, E. Boller, J. Baruchel	*Received at ESRF:*

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Report:

Several snow samples have been investigated by absorption microtomography. The experiment was performed on ID19 using a specially designed refrigerated cell, and the Frelon CCD camera for imaging. The main result is the feasibility of such low temperature microtomographs. Experimental problems related to cold sample handling have been addressed in real size and mostly solved.

Experimental key points

Experimental cell

To perform tomography on cold materials, one has to preserve both low sample temperature (here, T< -40°C) and optical transparency with regard to X-rays, with 360° visibility of sample. Moreover, these conditions have to be maintained during the acquisition time (~2 hour). For this purpose, a refrigerated cell with cylindrical double walls was designed (Fig 1). Liquid nitrogen was evaporated in the bottom of the sample holder, generating a cold and dry gas flow (T~ Td << -40°C), brought around the plexiglass sample holder, which ensures a sample temperature lower than -40°C. Gas nitrogen was then passed inside the (gas tight) bottom of the cell for gas warming up to (T ~ +10°C, Td << -40°C) and finally inside the double shell ; this prevented both dew (outside) and frost (inside). To ensure mechanical stability of the sample and reduced thermal loss, the sample holder was laterally fastened to the cell using a 1 cm thick bakelite plate.

Sample preparation

Divided snow structures, like fresh snow, are fragile and highly sensitive to sublimation. Vapour transfer was avoided by embedding the sample in diethyl-orthophtalate. Unfortunately, the absorption of this compound is close to ice absorption, leading to poor contrast. By adding 4% dibromoethane, a highly absorbing compound, contrast could be restored but reconstructed images were distorted for unknown reasons. Moreover, at this high concentration, the solid matrix was difficult to machine, even below -25°C. Further attempts are planned to find the adaquate impregnation conditions.

The transfer of samples between the storage cool box and the cell was also a source of trouble, because of the small size of samples. The most convenient way was to « follow » the sample with cold gas nitrogen (~-150°C) until the cell is filled and closed. We are planning to improve this procedure by having a sufficient number of sample holders to avoid « naked » samples transfer outside the cold laboratory.

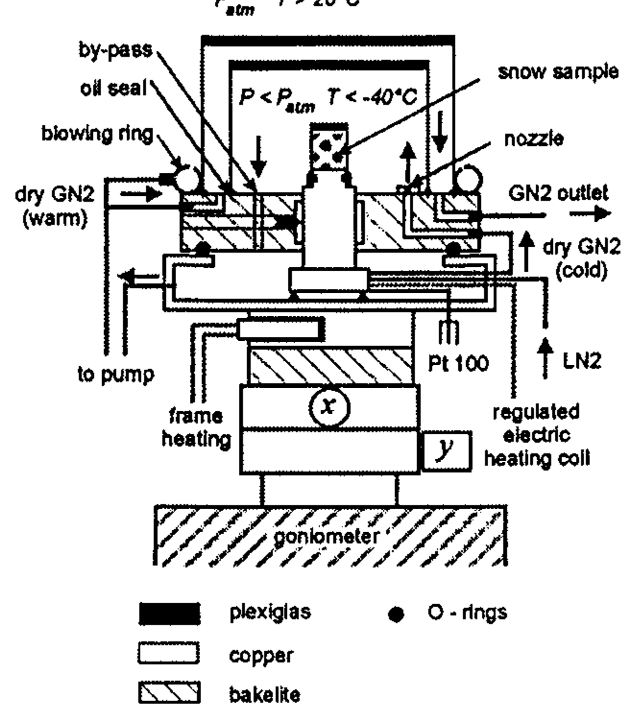

Fig 1 : LGGE/CEN Refrigerated cell

Ice / water detection

We also visualized the melting of an ice sample. Owing to the significant difference of density between ice (0.917) and water (1), the ice/water interface could be seen on raw absorption images. If the difficult task of controlling sample temperature and dewpoint at 0°C happened to be solved, the first 3D imaging of water menisci and percolation paths in wet snow might be imagined in the future.

Scientific outlook

Until recently, only the 2D local curvature of grains could be used. This gave valuable information on grain types for modeling snow metamorphism but could not, for instance, account correctly for water percolation. The availability of direct 3D information makes it possible to use the full curvature. We have developed a code for curvature computation from the 3D images, and we checked it on a set of microtome serial cuts of refrozen wet snow. Applying this code to the data files obtained three snow samples produced the curvature histograms shown in Fig 2. Samples a, b, c, obtained through three different physical processes, lead to three quite different curvature « signatures ». For instance, the histogram of refrozen snow (sample a) shows two modes. One of them, being positive, is characteristic of grain size. The other mode points out negative curvatures and should correspond to interconnected liquid water menisci (concave).

Tomographic high resolution images will clearly allow further investigation of other features of snow microstructure such as specific area, relevant for metamorphism dynamics, or ice bond geometry, relevant for mechanical and thermal properties.

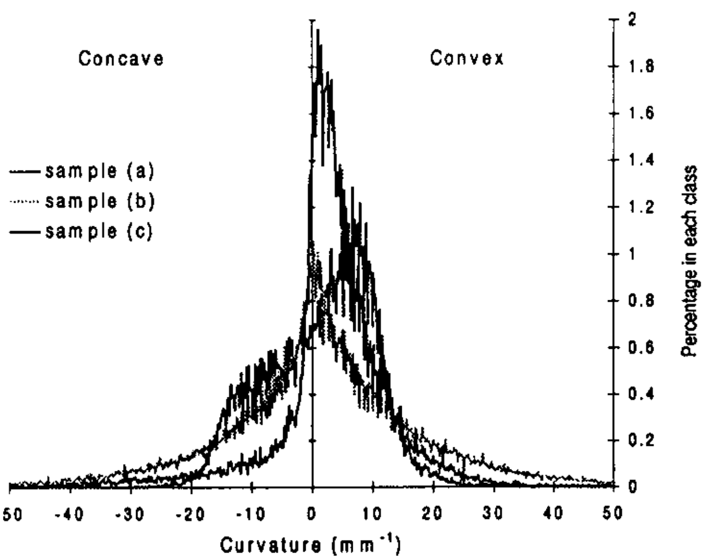

Fig 2 : 3D curvature histograms (measured over 128^3 voxels) of snow samples