<table>
<tr><td>ESRF</td><td>Experiment title:
In-situ mechanical testing of snow observed by Diffraction Contrast Tomography</td><td>Experiment number:
Ma412</td></tr>
<tr><td>Beamline:
ID19</td><td>Date of experiment:
from: 28/08/07 to: 30/08/07</td><td>Date of report:
08/10/07</td></tr>
<tr><td>Shifts:
6</td><td>Local contact(s): W. Ludwig</td><td>Received at ESRF:</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
<u>J. Meyssonnier</u>, A. Philip, N. Belki, S. Rolland du Roscoat (LGGE, Grenoble, France)
B. Lesafre, E. Pougatch, J.-M. Panel, P. Pugliese (Météo France, Grenoble, France)</td></tr>
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Report:

1. Introduction

The main goal of this study is to investigate the deformation mechanisms of snow, loaded under uniaxial compression. In order to characterise all microstructural aspects of the snow samples which are relevant for subsequent numerical modelling work, two different imaging techniques were applied

- Diffraction Contrast Microtomography (DCT) for characterisation of the initial grain microstructure in the undeformed state. This technique gives access to the grain micro structure of polycrystalline materials in terms of 3D grain shapes and crystallographic orientations. Due to geometrical restrictions (sample environment), the spatial resolution of DCT is limited to about 50 µm in the current study.
- Microtomography (µCT) in absorption mode using a high(er) resolution (5 µm pixel size) optical setup. By acquiring a series of tomographic scans at different stages of the (interrupted), uniaxial deformation experiment, this technique allows monitoring subtle changes in the snow microstructure.

The current experiment was aimed at testing the feasibility of such a combined DCT / µCT in-situ study on an artificial snow sample (0.7 g/cm^3), containing about 2000 individual grains.

2. Sample environment: cryogenic cell including a mobile piston for uniaxial compression

Figure 1 presents the cryogenic cell which was developed by Météo France and LGGE. The sample is maintained at negative temperature (-5C) by contact with the a cold air stream, The cold air stream was preconditioned by a dry air generator (not shown in the picture) and stabilized to within°±1°C by means of the heat exchanger and two stage Peltier element located in the base part of the cell. During 14 hours of continuous operation, the temperature at the bottom part of the cell volume was -13°±1°C and -6°±1°C in the top part, close to the mobile piston. By placing different weights on top of the piston, the sample can be loaded in uniaxial compression (constant load).

3. Diffraction Contrast Tomography

Snow is a polycrystalline two phase material (air and ice). Provided some conditions on grain and sample size being fulfilled, DCT can be applied to reconstruct the 3D grain microstructure of snow samples, by record simultaneously the transmitted and the diffracted beams (Figure 2a). Figure 2b shows a zoom on the direct, transmitted part of the beam, with one of the snow grains (top left corner) fulfilling the diffraction condition and giving rise to an extinction spot. In order to reconstruct the 3D grain shapes and orientations of the individual grains, this technique requires a relatively complex analysis procedure. The preliminary results are encouraging and confirm the feasibility of grain reconstruction for the present case of samples containing more than one thousand grains. Figure 3 illustrates the principal steps of the DCT analysis procedure: from isolation of the extinction spots corresponding to a given grain, backprojection into the sample plane and 3D ART reconstrcution of the grain shape.

4. Microtomography in absorption mode under compression

As mentioned in section 1, a compression system was included in the cold cell. By adding weight on a piston, the snow sample can be compressed. Figure 3 illustrates the results obtained during this first feasibility study. The depicted slices were extracted at the same physical position inside the sample for the five different compression levels. As snow presents a visco-plastic behaviour when compressed, the following experimental procedure was applied: The sample is deformed under constant load for a given time (up to 300 g, 30 min). After this the load is and the sample is allowed to stabilize during another period of 30 min (possibility of viscoplastic relaxation) before a new tomographic scan is acquired.
Due to technical problems (failure of the air saturation device, temperature gradient in the cell) those parts of the sample which were in direct contact with the cold air stream sublimated. Unfortunately, the rate of sublimation was superior to the rate of deformation applied in this first test experiment.

5. Conclusion and perspectives

The current study showed the feasibility of DCT on snow samples containing more than 1000 grains. The data acquired have allowed us adapting and optimizing the DCT analysis procedures for the case of a two phase material with hexagonal crystal symmetry.
The cryogenic cell allows maintaining the sample for extended periods at constant, negative temperature. However, the presence of a temperature gradient and the problems caused by sublimation require a revision of the device (additional sample container avoiding direct contact with the cold air stream, reduction of gradients by increasing the flow rate).

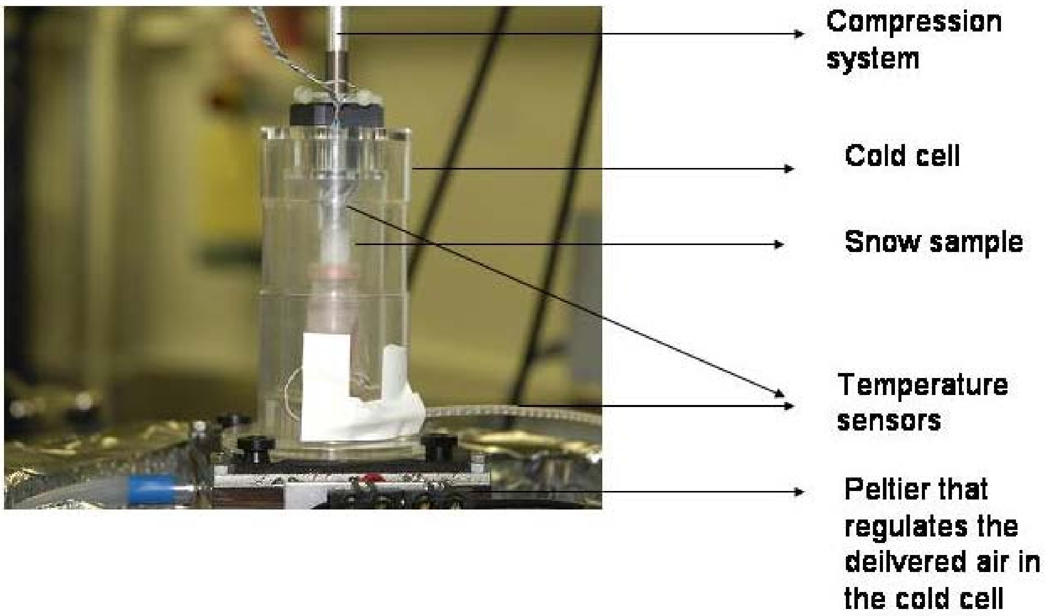


Figure 1: Cryogenic cell mounted on the ID19 microtomograph.

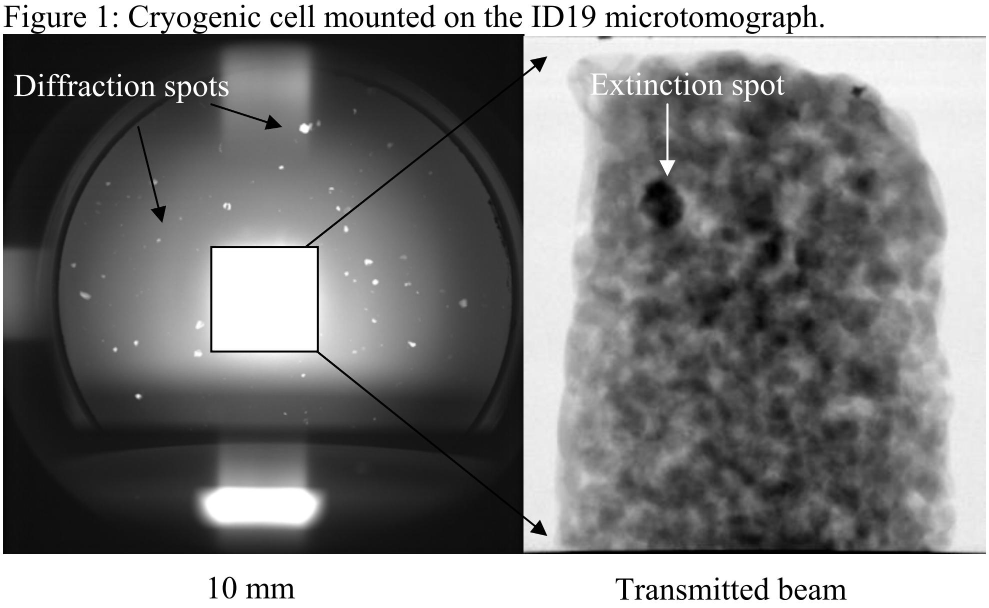


Figure 2: a) Raw projection image showing transmitted beam and diffracted beams. b) zoom on transmitted beam extracted from the DCT radiograph (9.9 mm * 11.070 mm)

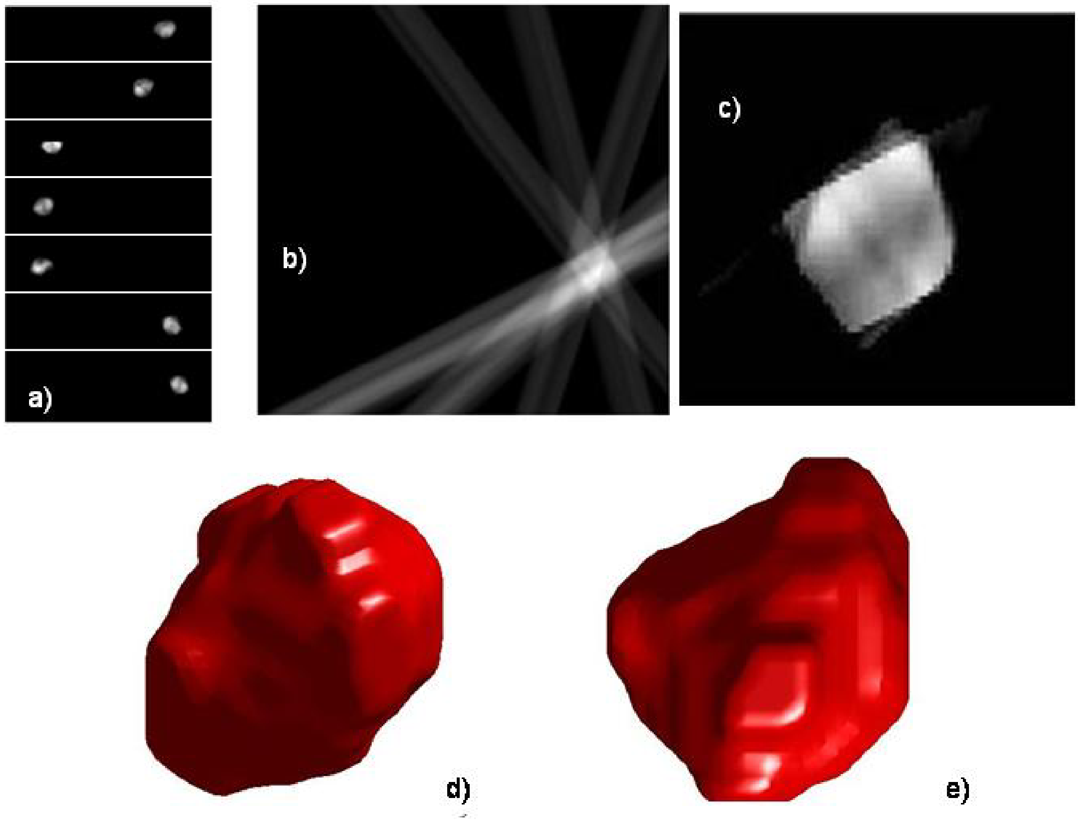
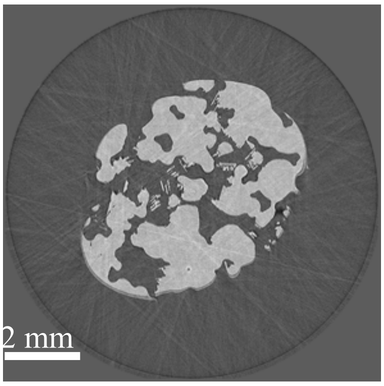
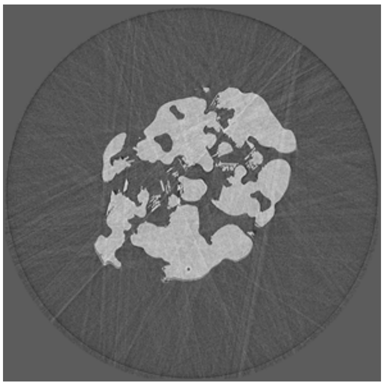


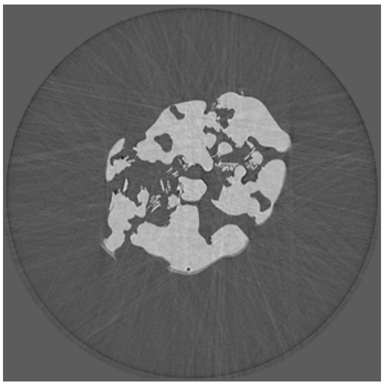
Figure 3: Results of the DCT analysis in the case of snow. These pictures illustrate the different steps of grain reconstruction: a) set of extinctions spots corresponding to projections of the same grain at different rotation angles, b) crude estimate of 2D grain outline and grain position obtained by simple backprojection c) reconstruction of central slice by algebraic reconstruction techniques d) and e) 3D views of the complete grain.



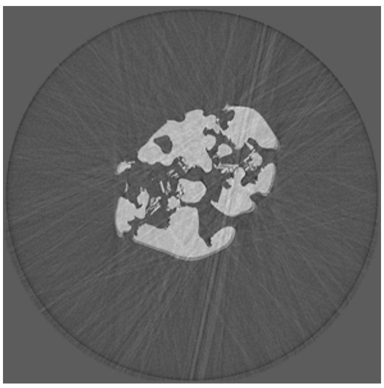
a) sam2_0_
Reference scan



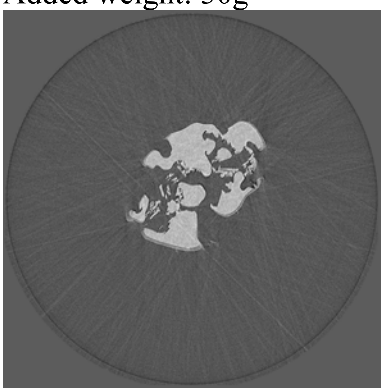
b)sam2_1_
Added weight: 30g



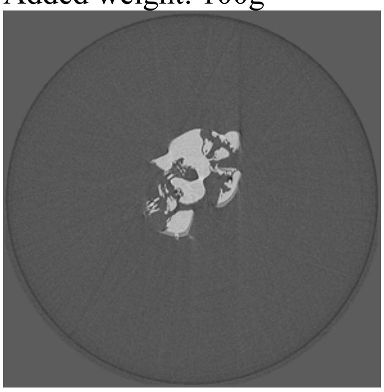
c)sam2_3_
Added weight: 100g



d) sam2_4_
Added weight: 175g



e) sam2_5_
Added weight: 175g



f) sam2_5_
Added weight: 225g

Figure 4: Slices extracted at the same physical height, illustrating the problem of sublimation encountered in this first feasibility study.