ESRF	**Experiment title:** **Microstructural characterization and in-situ mechanical testing of snow**	**Experiment number**: **MA 513**
Beamline: ID 19	**Date of experiment**: from: 07/03/2008 to: 11/03/2008	**Date of report**: 28/07/2008
Shifts: 12	**Local contact(s)**: W. Ludwig	*Received at ESRF:*

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Report:

Objectives

Our objective is to understand *how snow deforms at the grain scale*. Snow consists of ice grains linked by bonds, air, and eventually some liquid water. Since snow conditions arise usually at “high” temperature (from the materials science point of view -20°C corresponds to a ratio T/Tm >0.93) and because of the *extreme viscoplastic anisotropy of the ice crystal* in this temperature range, we want to assess the relative influences of *intra-granular deformation* vs. *grain boundary sliding* (GBS) at snow grain-to-grain contacts. To this aim we plan to perform compression tests on snow under synchrotron X-ray radiation in order to record the evolution of its microstructure, then interpret the results in terms of constitutive behaviour through micro-scale numerical simulations.

Introduction

In order to characterise the microstructure of the snow specimens relevant for subsequent numerical modelling, *Diffraction Contrast Microtomography* (DCT) and *Microtomography in absorption mode* (μCT) were applied. The feasibility of the study was proved during experiment MA412 (August 2007).

- DCT was used to characterise the grain microstructure at a given deformation stage. This technique gives access to the grain micro structure of polycrystalline materials in terms of 3D grain shapes and *crystallographic orientations*.
- μCT was used to monitor subtle changes in the snow microstructure during the uniaxial deformation.

The MA513 experiment was divided into two parts: part-1 was performed in January (3 shifts) and part-2 in March (12 shifts, of which 4,5 were necessary, following a general power failure prior to the experiment, to reset the whole beamline). The report on part-1 has been submitted 15/03/2008 (38654_A). The present report concerns MA513-part-2.

Experimental set-up and protocole

During this experiment, we analysed laboratory processed specimens that exhibit a large average grain size (~2mm), so as to make the reconstructions easier. Figure 1 shows the type of grains used to prepare the samples (density ≈ 0.65 g/cm^3 with about 150 grains). The specimen was placed into a specially designed cold cell in which a low temperature gradient and controlled humidity prevent snow from sublimation. Snow compression was achieved through a mobile piston loaded with dead weights in successive steps.

Both DCT and μCT techniques were applied using the same optical set-up of 20.4 μm pixel size. The MA412 experiment showed that this is a good compromise for both DCT and μCT scan analysis, since changing the optics according to the scan type is too much time consuming.

It was necessary to unload the specimen before each DCT scan, to prevent it from deforming during the scan (each DCT scan, i.e. 7200 radiographs, lasted about 1 hour). The deformation of the specimen during a conventional μCT scan (~ 5 min.) was found negligible enough to allow scanning the snow without removing the load. As a consequence the number of DCT scans performed is much lower than that of µCT scans. During the time effectively used for the experiment (7,5 shifts) 4 specimens were analyzed. In average each specimen was loaded for 12h (the rest being used for cold cell reset, specimen installation and alignement).

Contribution to porous media DCT scan reconstruction

Automatization

DCT reconstruction requires first to gather the information about the recorded diffraction spots (center of mass, area, intensity,...), then for each spot to find, by image correlation, its corresponding "twin" on the radiograph taken at ±180°. Given a pair of twins it is then possible to infer the location of the diffracting grain inside the specimen and, knowing the beam energy and the ice lattice parameters, the nature of the diffracting plane. The number of matching pairs was currently about 4000. The association of all the diffraction spots that belong to a grain allows to reconstruct the 3D grain shape associated with a crystallographic orientation.

The work done to process the data obtained during the experiment has shown that it is possible to *automatise the reconstruction of DCT scans*, at least in the case of a snow sample. Namely the reconstruction parameters used for filtering are the same, regardless of the size of the grains.

Error assessment

The *error made on DCT reconstructed grains* in terms of shape and size has been estimated by comparing the grain shapes derived from the DCT analysis with that obtained by µCT. In most cases, we found an envelop of several pixels (2 to 5) missing around the grains. This probably comes from the adjustment of the DCT reconstruction parameters, which is difficult to achieve. Nevertheless this represents an underestimation of the grain sizes by about 4% only.

When superposing the DCT and µCT reconstructions, there are zones around a few grains where µCT indicates that there is ice, but where DCT fails to provide any orientation information (Fig. 2). In these few occurrences, we have simply filled the unknown orientation zones with the closest orientations using a propagation algorithm. For the time being, this technique can only be assessed qualitatively. As shown on Fig. 2, it gives physically acceptable results when the incomplete grain is isolated (tag A), however it can sometimes lead to grain boundary shapes that are very unlikely (tag B). A further improvement will be to automatically detect the unlikely junctions between two grains, then to use the curvature maps of the grains (derived from µCT reconstruction) to localize the grooves that are thought to mark the intersection of the grain boundaries with the grains surface (see Fig 1, tag A). In a first stage the micro-scale numerical simulations will assume plane grain boundaries, although the actual shape may be influential when modeling GBS. Since it seems difficult to gain more accurate information about the actual shape of the grain boundaries from the present experiment, a specific topography study seems necessary.

Deformation analysis

During the few months following the experiment we have mainly focused our attention on the snow specimen that underwent the highest compressive stress. The strain vs. time curve is shown on Fig. 3. So far the first two scans (DCT1, DCT2) out of 5 have been completely reconstructed and analysed. The others are being processed and the reconstruction happens to be difficult due to overlapping diffraction spots. A consequence of the intragranular deformation increasing with the total deformation is the development of grains mosaicity (cf. Table1). This results in diffraction spots which spread on several radiographs (like a rocking curve), so that the probability of overlapping spots from different grains increases. We are currently developing tools to separate these spots and complete the analysis.

Scan	DCT1	DCT2	DCT3	DCT4	DCT5
Mean mosaicity	0.15	0.29	0.36		0.88
Standard deviation	0.19	0.39	0.39		0.96
Minimum mosaicity	0.01	0.03	0.05		0.02
Maximum mosaicity	0.68	1.01	1.3		3.13

Table1. Mosaicity expressed as the angle (deg.) over which a same diffraction spot spreads.

We have developed a correlation function that allows matching a grain from one DCT scan to another. This was used to quantify the changes in the orientation of the *c* and *a* axes of the grains induced by the two first steps of deformation. For each axis an average change of 1 degree (with a maximum of 3 degrees) has been found. As expected, the grain centres of mass moved mainly in the vertical direction according to the vertical displacement of the piston.

Conclusion

The data collected during this experiment are the core of the French ANR "Snow-White" project dedicated to studying the deformation mechanisms of snow. A post-doc, detached from LGGE to work at the ID-19 beam line under the supervision of Dr. W. Lüdwig, has adapted for ice and developed the automatization of the treatment of raw data. The obtained reconstructions will now be used for simulating the micro-mechanical deformation of snow (on the basis of deforming discrete elements). Since the viscoplastic anisotropy of ice is extremely marked in the range of usual temperatures (i.e. above -25°C), the DCT reconstructions, which provide the crystallographic grains orientations, and their evolution, will allow to obtain original results never published before.

Acknowledgement

The participants wish to thank the ID-19 team, and especially Wolfgang Lüdwig, for their involvement and concern about the success of the experiment.

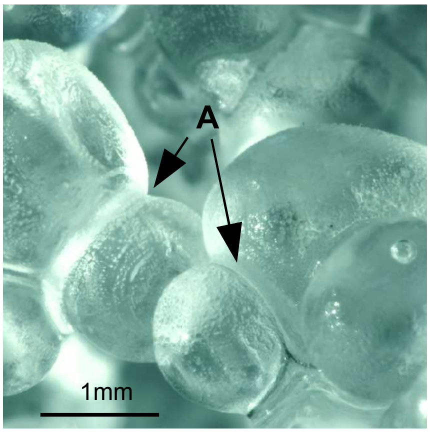

Fig. 1: Laboratory processed snow with large grain size.

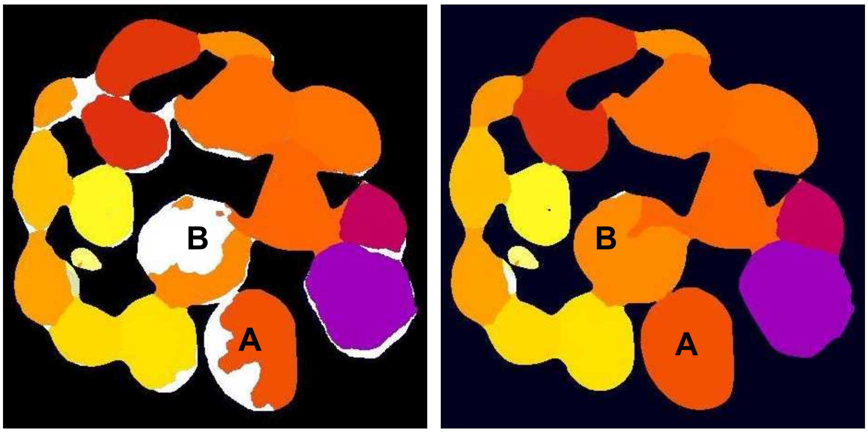

Fig. 2: Grain reconstruction using both DCT and μCT (black is for the pore phase). Left: DCT reconstruction giving grains crystallographic orientation (color) superposed to the μCT reconstruction in white (i.e. missing orientation). Right: improved reconstruction. (the specimen diameter is 10mm)

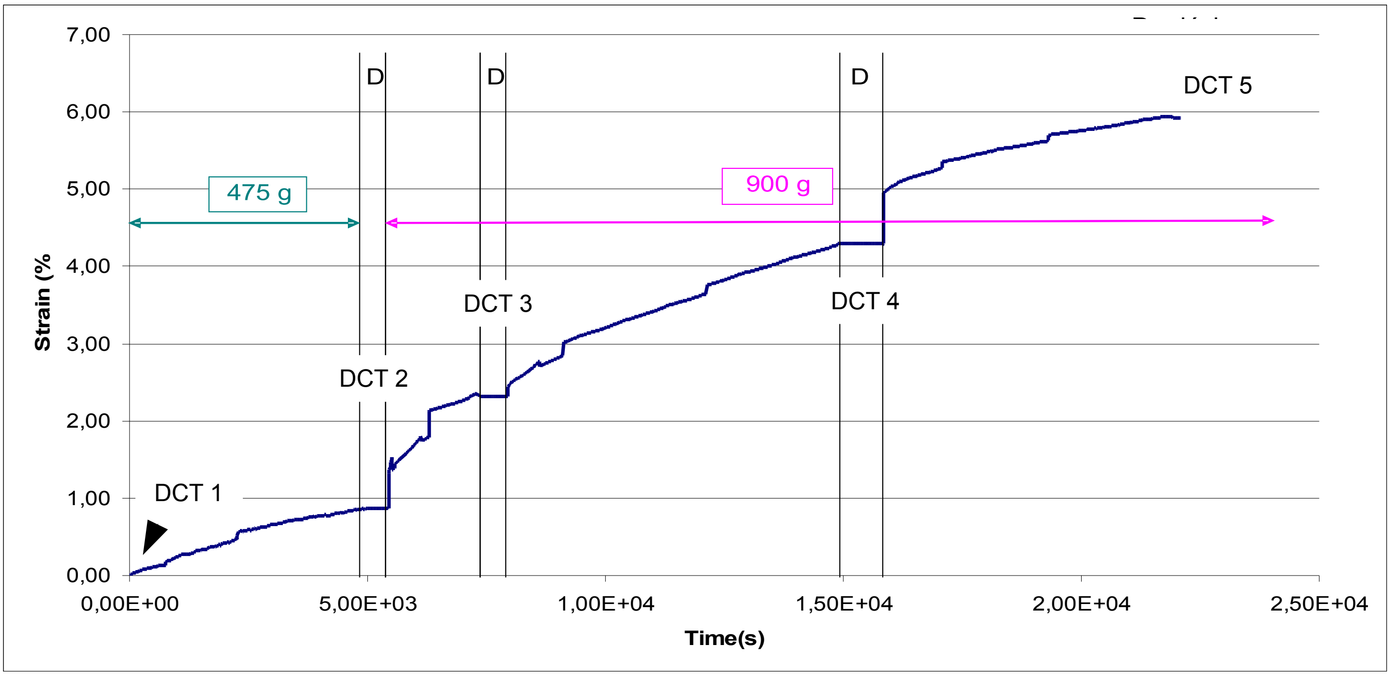

Fig. 3. Deformation undergone by the snow specimen during the compression test. The first step was under a compressive stress of 0.06 MPa *, the second* 0.12 MPa*. DCT scans were recorded after unloading.*