ESRF	**Experiment title: Structural study of the solidification of water from ice VI to amorphous ice under variable compression rates.**	**Experiment number**: Hc-2183
Beamline: ID27	**Date of experiment**: January, 27 – February, 2 2016	**Date of report**: 01/03/2016
Shifts: 12	**Local contact(s)**: Mohamed Mezouar.	

Names and affiliations of applicants (* indicates experimentalists):

Paul Loubeyre, CEA (France)*.
Florent Occelli, CEA (France)*.
Ramesh Andre*.
Agnes Dewaele*.

Report:

The aim of this proposal was to characterize by x-ray diffraction a suspected transition from solidification into ice VI to solidification into high density amorphous ice under increasing compression rate, in the GPa/ms range. A published study had suspected the formation of high density amorphous ice (HDA), formed from metstable ice VII in the stability field of ice VI [1]. But the characterization was made by Raman spectroscopy and the signature of HDA ice was not unambiguous. It thus seems important to reproduce such an experiment with an x-ray microscopic characterization.

The experiment was the first dynamical-DAC experiment implemented on ID27. We have developped a portable pressure controller module to tune compression rate in the membrane-DAC. Time resolved ruby measurements and micro-photograhic images enable to characterize the compression of the sample with 1ms resolution. The first part of the beamtime was devoted to synchronize the compression ramp with the x-ray detector and the ruby pressure measurement. A large effort was also devoted to adapt a detector to perform these ms-resolution XRD measurements. A dimax camera was coupled to an intensifier or to an optic system. The former gave a factor 100 in the number of counts. That was necessary to observe the diffraction pattern from ice in 3 ms, however a distortion and instabilities were induced by the amplifier that degraded the accuracy of the structural data. Yet, the microscopic arrangement of the phase could be qualitatively determined.
Various compressions of water with different rates, from 0.1 GPa/ms to 1 GPa/ms, have been performed on different water samples.

The major results of this campaign are:
- The structural changes of a material under a compression rate of ~1GPa/ms can be followed with a ms resolution. With a better detector, it will thus become possible to quantitatively investigate the kinetics of

phase transitions, the chemical reactions, the mixing of fluids, the formation of metastable phases…. That opens a new area of investigations on ID27.

- Water was always observed to crystallize in phase VI and then above 2.4 GPa to transform into ice VII. The formation of HDA ice was never obtained even for compression rates faster than the one of the published previous study [1]. That underlines the fact that the microscopic characterization is mandatory to prove the formation of a metastable microscopic arrangement.

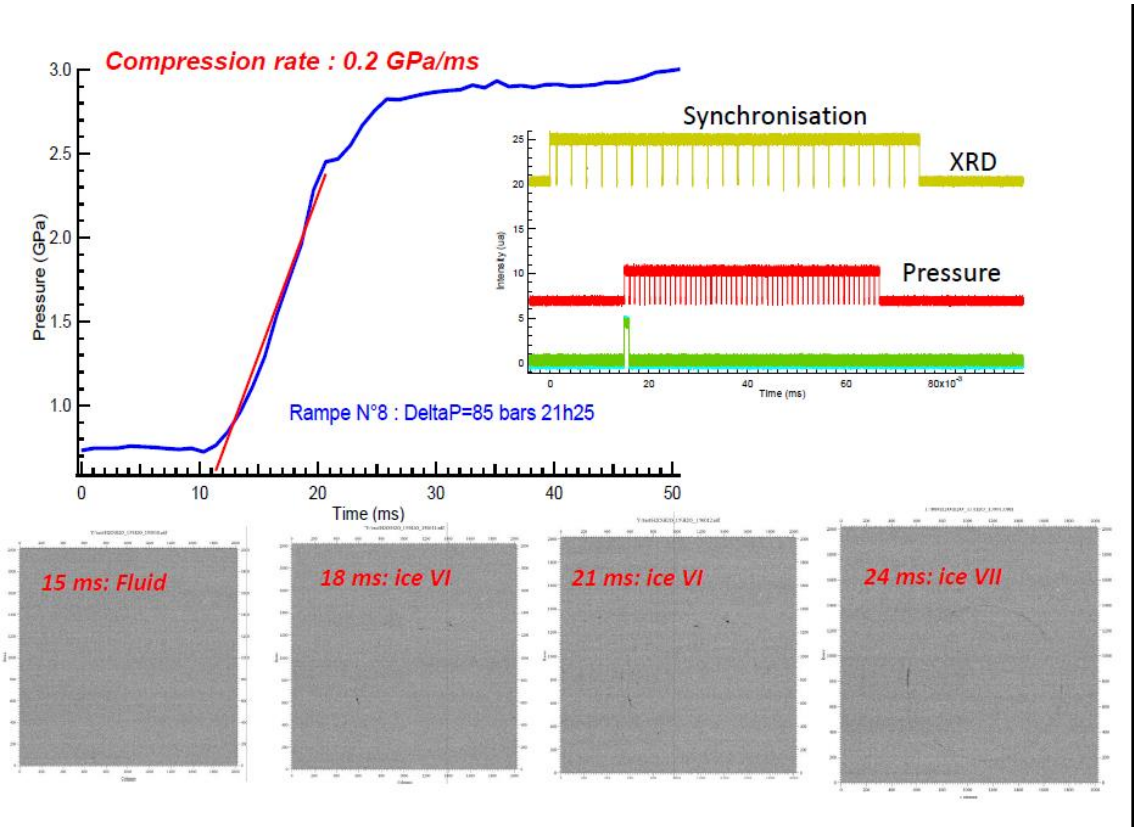

Figure 1: Typical measurements made during a compression rampe. Structural evolution of water in ramp n°8. The compression rate achieved was 0.2 GPa/ms.
The trigerring of the experiment and the synchronization of the XRD detector and of the spectrometer to monitor the pressure are followed on an oscilloscope. The XRD images of the sample accumulated over 3ms are shown at the bottom of the figure at different times. The microscopic state of the sample can be clearly identified.

References:
[1] J. Chen and C.S. Yoo, PNAS 108, 7685 (2011).