ESRF	**Experiment title:** Ms-scale time-resolved X-ray diffraction of Water (H2O) at high pressure and high temperature	**Experiment number**: HC4895
Beamline: ID24	**Date of experiment**: from: 31/05/2022 to: 06/06/2022	**Date of report**: 30/08/2022
Shifts: 18	**Local contact(s)**: Matteo Levantino	*Received at ESRF:*

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Report:

The study of non-equilibrium transition dynamics for structural transformations in the s-to-µs regime is an emerging field in high pressure research. It is made possible by the development of a new device, the dynamical-DAC. Phase transformations (liquid – solid, solid – solid) are often driven by diffusion mechanisms, a process occurring typically at intermediate timescales spanning from hundredth of µs to tenth of ms. Such timescale lies between the characteristic time of shock compression (~ nanoseconds) and of static compression (~ seconds). Discrepancies between these two types of compression have been observed and experiments at an intermediate timescale of µs-ms are now helpful to resolve these disparities. Also, there are now ample opportunities to discover new phenomena by positioning the time scale of the compression respectively to the nucleation rate, the motion of crystal defects or the crystal growth kinetics. Water (H2O) has been one of the first system to be studied in the d-DAC because of the implication for fundamental physics of diffusion-mediated properties and for the thermodynamic modeling of collision/ impact events on ice-rich planetary bodies. Based on visual and Raman observation, super-compressed liquid water was first reported to crystallize into ice VII in the stability field of ice VI. However, it was then reported that the metastable ice VII in the stability field of ice VI forms a high-density amorphous ice.

Using our newly developped piezo-driven dynamical-DAC combined with the JUNGFRAU 1M detector implemented on the beamline for this experiment, we were able to obtain high quality results on the nucleation of ice at ms-timescale/compression rates of GPa/ms.
Water samples were loaded with a piece of copper as a pressure marker. Using the equation of state P(V) of copper and ice VII, one has then access to the evolution of pressure as a function of time, as shown in figure 1.

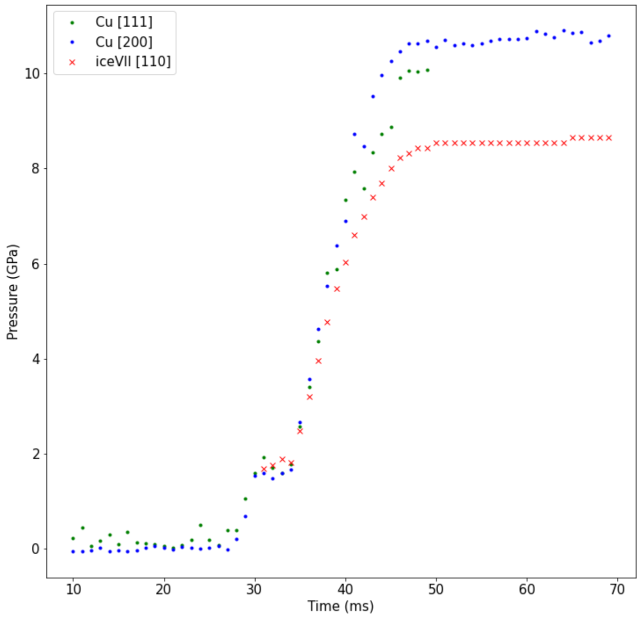

Figure 1: Pressure evolution in the sample extracted from the [111], [200] diffraction peaks of Cu and from the [110] diffraction peak of ice VII.

Our results show no evidence of the formation of a high-density amorphous ice like it had been previously reported using spectroscopic techniques (J-Y Chen et al. High density amorphous ice at room temperature. PNAS 108, 7685 (2011)). Instead we observed the nucleation of ice VII as expected.

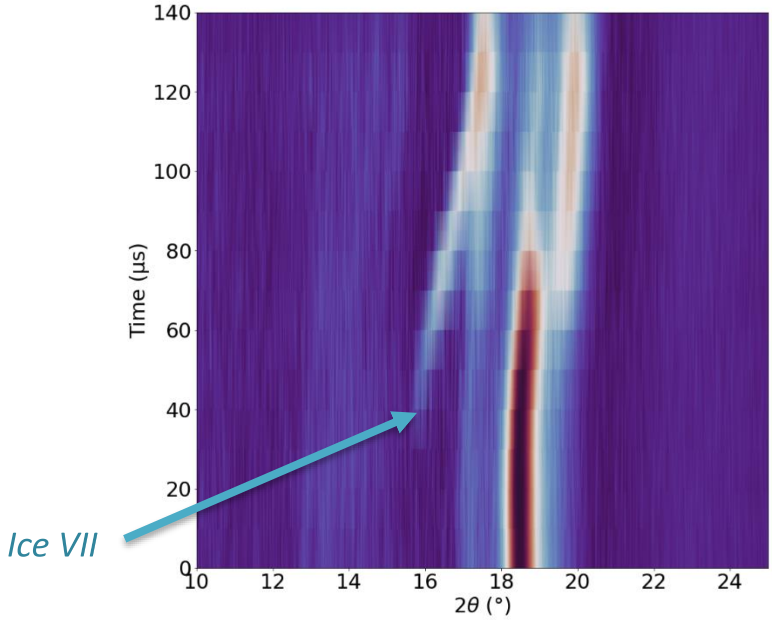

Figure 2: Nucleation of ice VII evidenced using the burst mode of the JUNGFRAU detector.

This was further evidenced when using the unique capability of the JUNGFRAU detector to work in burst mode. In this mode we were able to aquire 16 images with 7 µs of integration time and 1 µs of interframe.

The combination of the piezo-driven dDAC and of the JUNGFRAU 1M detector using the burst mode now offers the unique possibility to perform time-resolved experiments on materials at very high compression rates with an excellent signal quality and a very good time resolution.