Experimental Report

Proposal: **5-24-514** **Council:** 4/2012

Title: Structure of hot ice VII

This proposal is a new proposal

Researh Area: Physics

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Samples: D2O

Instrument	Req. Days	All. Days	From	To
D20	2	2	21/11/2012	23/11/2012

Abstract:

We wish to investigate the structure of ice VII at high temperatures and pressures, i.e. 150 °C and 3-4 GPa. Preliminary neutron diffraction studies on D4 in 2010 by our team indicate that ice VII might not crystallize in the expected cubic Pn3m phase, but possibly to a “plastic phase” as predicted by various computer simulations. We think that the proposed experiment might make a significant contribution to the physics of ice.

Experimental report "Structure of hot ice VII" (exp. nr. 5-24-514)

Main Proposer: S. Klotz
Experimental team: S. Klotz, Ph.S. Salmon, H. Fischer, L. Bove, A. Ludl, Th. Hansen

The purpose of this experiment was to investigate the structure of ice VII along the melting line, in the 0-3 GPa and 300-500 K range. The experiments were motivated by theoretical predictions that "plastic" ice phases should exist in this P/T region [1,2]. There was also some experimental evidence from our previous measurements on D4 (Sept. 2010 [3]) where liquid water at 150°C was compressed into ice VII and diffraction patterns showed reflections which could not be indexed in the expected cubic Pn3m structure. The experiment was aimed to clarify this issue, using a recently developed setup [4] which allows probing this region of water's phase diagram using high P/T neutron diffraction.

Experiments were carried out using two types of high pressure cells. The first measurement (10 h) used an ILL Al-cell for liquids, up to 2.5 kbar. This experiment helped to calibrate pressure values relevant to the previous D4 experiment on liquid water [3], a parameter which was crucially needed to advance the data analysis. The main part of the beamtime was then carried out using the ILL Paris-Edinburgh cell, and a setup and a P/T path identical to the previous D4 measurements.

A sample of D_2O was loaded into cBN anvils, using TiZr encapsulating gaskets. The liquid (Fig. 1, top; crystalline peaks are from cBN anvils) was heated at a pressure of a few kbar to 150°C and then compressed in steps until crystallisation occurred at ~3.1 GPa. Upon further compression the liquid transformed then fully into highly textured ice VII where most of the intensity was found in one single (111) reflection (Fig. 1, 2nd panel). To improve the powder quality, the heaters were then switched off, and cooling was achieved by spraying liquid nitrogen onto the anvils which resulted in a measured temperature of -10 °C on the anvils after 20 minutes. Heating back to 150°C gave an improved powder pattern, but with still considerable preferred orientation (Fig. 1, 3rd panel).

Comparison with simulated patterns (Fig. 1, bottom panel) shows that all reflections can be assigned to the expected Pn3m structure. The experiments rule out the existence of plastic phases with non-cubic symmetry in the P/T range investigated. The origin of the non-cubic reflections observed at D4 is hence still not clear.

References

[1] Y. Takii, K. Koga, H. Tanaka, J. Chem. Phys. **128**, 204501 (2008).
[2] J.L. Aragones & C.Vega, J. Chem. Phys. **130**, 244504 (2009)
[3] Experiment no. 6-02-464 "Structure of water at high pressure » (Salmon et al.)
[4] S. Klotz et al., J. Phys. D: Appl. Phys. 44, 055406 (2011).

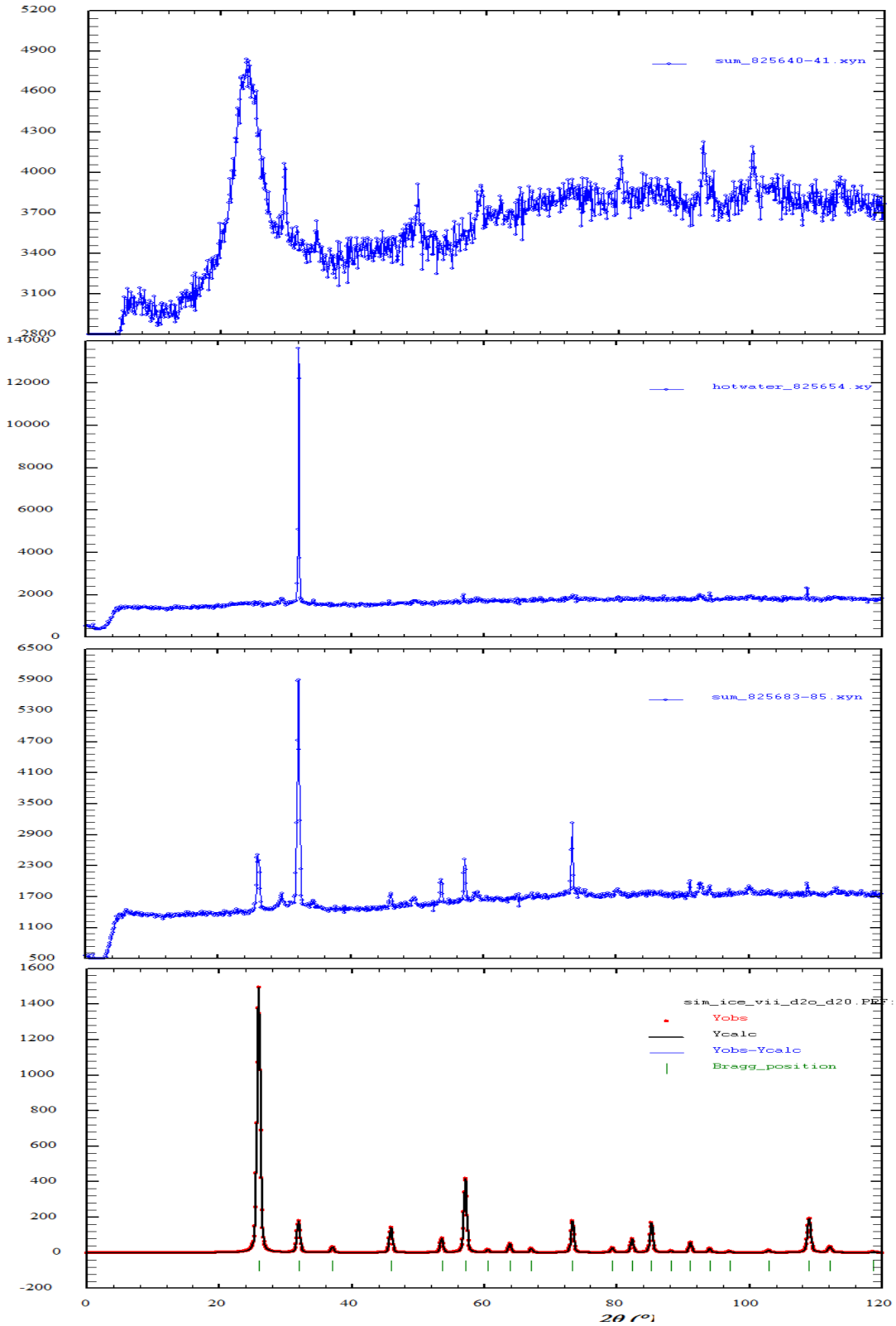

Figure 1: Diffraction patterns of water compressed at 150 °C from the liquid (top, crystalline peaks are from cBN anvils) into ice VII (2nd panel). The pattern in the 3rd panel was obtained after cooling to -10 °C and heating back to 150 °C. The lower pattern is a simulation of ice VII with a=3.315 Å. Note that all sample reflections can be explained by a cubic structure. λ=1.06 Å.

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