ESRF	**Experiment title:** Stuctural study of dense water at high pressure and high temperature in a diamond anvil cell.	**Experiment number:** HS1213
Beamline: ID30	**Date of experiment:** from: 16/02/2000 to:19/02/2000	**Date of report:** 29/02/2000
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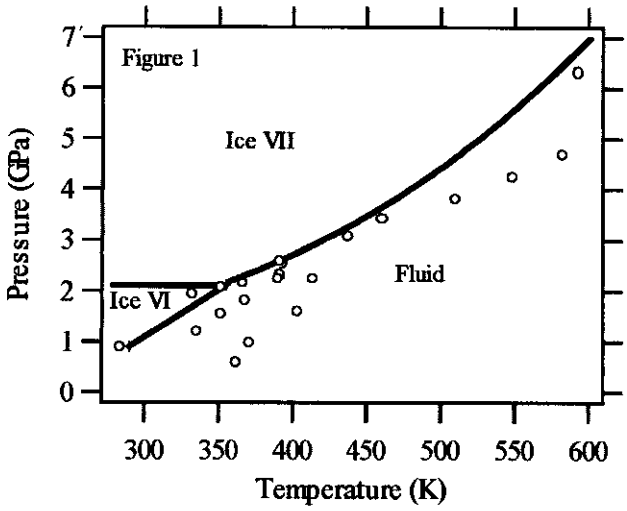
Report:

We have performed angle dispersive diffraction on fluid water at a variety of pressures and temperatures in three samples on the ID30 beamline. We used a diamond anvil cell (DAC) equipped with a large x-ray aperture of 35° and a boron backing plate offering low absorption to an angle of 63°. The sample was heated with a rhenium heater epoxied onto the rhenium gasket and the DAC body was water-cooled. This configuration allowed us to rapidly heat, temperature control, and stabilize the gasket, sample, and diamond while keeping the DAC body relatively cool. To preclude graphitization of the diamond anvils we enclosed the DAC in a vacuum chamber evacuated to less than 10^{-4} torr.



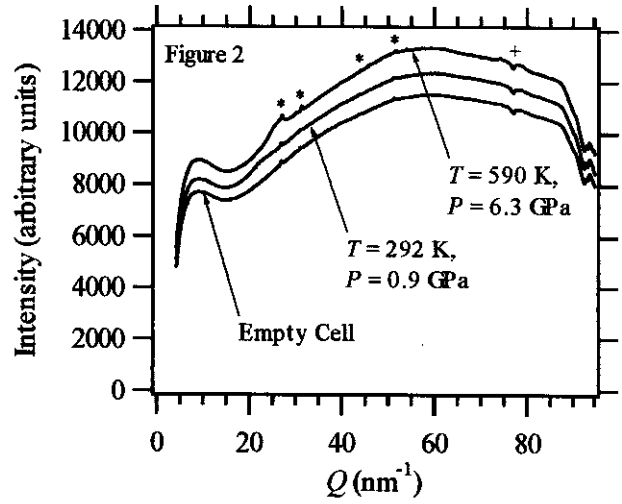
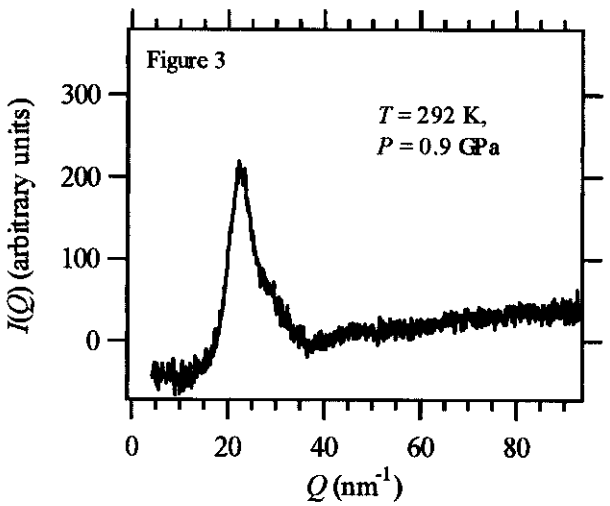
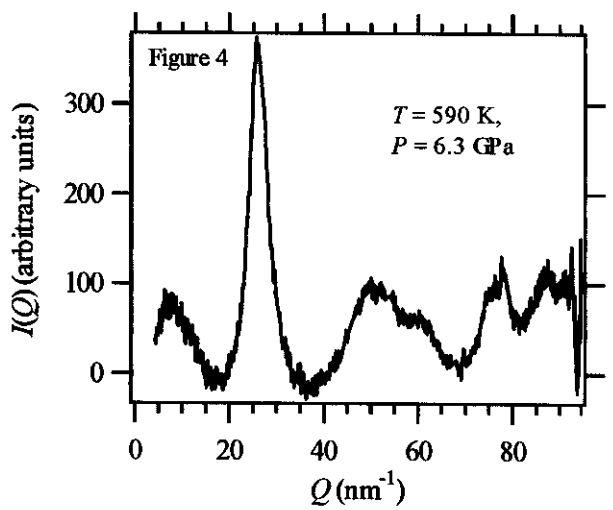


Figure 1 plots the 21 P, T points measured so far along with the relevant phase diagram. When present, solid ice is readily apparent in the spectra due to the appearance of sharp Bragg peaks. As suggested by figure 1 two of our measurements are metastable fluid within the ice VI stability field and one spectra shows ice VII -- fluid coexistence.

By using $\lambda = 0.3738$ Å and the FastScan image plate online detector with a distance $L = 235$ mm we are able to measure diffraction in our system up to $Q \cong 95$ nm^{-1}. Figure 2 shows the raw data integrated by FIT2D for the empty DAC and the DAC plus sample for two measurements. Compton scattering from the diamond anvils dominates the spectra and the intensity drop beginning at 50 nm^{-1} is from absorption in the boron backing plate. The sharp peaks marked "*" are Bragg diffraction due to the Kapton windows of the vacuum chamber, while the dip marked "+" is due to imperfect masking of single crystal Bragg diffraction from the diamond anvils. Due to improved photon flux and electronic readout following a recent rewiring and realignment of ID30 our accumulation time for the spectra fell from 900 to 30 seconds. Thus, in the current configuration the majority of the 30 minutes needed between each accumulation is devoted to obtaining pressure and temperature stabilization.





After subtracting the empty cell spectra we obtain the scattering from water, $I(Q)$, shown in figures 3 and 4. Due to the extremely weak sample signal and complications in the correction

for boron absorption, we have not yet attempted to separate the coherent and incoherent scattering from water included in our $I(Q)$ necessary to extract the structure factor $S(Q)$ and the radial distribution function $g(r)$. We have removed by hand several residual sharp features in the spectra due to imperfect subtraction of the Kapton and diamond peaks. In figure 3 we see the remnants of the characteristic double peaked (at 22 and 30 nm^{-1}) structure factor of ambient P, T water [1,2] as a weak shoulder at about 30 nm^{-1}. As in these previous experiments the double peak gives way to a singlet at higher P, T as shown in figure 4. In addition, there is a dramatic intensity increase of $I(Q)$ in the 40 to 90 nm^{-1} range at high P, T not seen in the previous measurements.

Although the FastScan accumulations take only 30 seconds, the experimental setup includes a high pressure DAC, a 24V/30A temperature controller/power supply, a vacuum chamber and turbomolecular vacuum pump, and cooling water. The installation of this equipment plus the standard PRL (pressure by ruby fluorescence) pressure measurement system initially requires about 8-10 hours of installation and alignment time, and about 4-6 hours to change a sample. Once the alignment is complete, pressure/temperature adjustment, equilibration, and data collection takes a minimum of 30 minutes per data point.

References:

[1] K. Yamanaka, T. Yamaguchi, and H. Wakita, "Structure of water in the liquid and supercritical states by rapid x-ray diffractometry using an imaging plate detector," J. Chem. Phys. 101, 9830 (1994).

[2] T. Radnai, and H. Ohtaki, "X-ray diffraction studies on the structure of water at high temperatures and pressures," Mol. Phys. 87, 103 (1996).