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Report:

A new experimental setup for high-pressure high-temperature XAS studies has been developed and used for the first time for this experiment at BM29. We have used the Paris-Edinburgh large volume pressure cell equipped with an a-B gasket in a vertical configuration. The sample was a small cylinder of I_2 NaCl mixture confined in a boron nitride micro-tube. Its diameter was of 0.8 mm and the useful sample dimension after application of the pressure was below 0.4 mm^2, The absorption of the x-rays was monitored by two photodiode detectors placed before and after the cell. In addition to the XAS detectors it was possible to detect the diffraction pattern on the horizontal plane with a NaI scintillator placed behind a narrow vertical slit. The angle was adjusted in the region of the I_2 (112) powder diffraction peak and the peak scan was made at a fixed angle by scanning the monochromator energy. The diffraction measurement has been used to calibrate the pressure scale and to verify the occurrence of melting. A reversible diffraction pattern at melting was observed up to 2.8 GPa. This in-situ sample characterization with XRD was essential to confirm the nature of the sample and to support the reliability of the XAS measurements.

Several samples have been measured at different pressure and temperature points. In particular two samples were pressurized up to 2 and 6 GPa and then heathed, another was pressurized and heathed to follow roughly the melting curve. Melting .was confirmed by the disappearance of the I_2 (112) line.

X-ray absorption spectra have been collected in a wide energy interval and a clear oscillating EXAFS signal was detected both for solid and liquid iodine. From these data it is possible to determine the I_2 molecular bond length with an accuracy of about 0.005 Å. The figures below report the typical profiles of the I_2 (112) peak which shifts to higher energies upon increasing pressure, and typical EXAFS spectra for solid and liquid iodine around 2 GPa.

The main result of the present experiment has been the accurate measurement (± 0.005 A) of the I-I bond length as a function of P and T. The large sensitivity of this parameter to the condensed environment in which the I_2 molecule is embedded has been emphasized in a previous work [l], present determination extend the region of investigation to higher fluid densities up to 2.8 GPa and about 800 K.

[l] U. Buontempo, A. Di Cicco, A. Filipponi, M. Nardone, and P. Postorino, J. Chem. Phys., (to be published 1997).

[2] U. Buontempo, A. Filipponi, D. Martinez-Garcia, J. P. Itiè, M. Mezouar, and P. Postorino, (unpublished).