ESRF	**Experiment title:** An EXAFS study of the structure of solid and liquid Ag2Se under extreme temperatures and pressures	**Experiment number**: HS1802
Beamline: BM29	**Date of experiment**: from: 16 May 2002 to: 21 May 2002	**Date of report**: *Received at ESRF:*
Shifts: 15 shifts	**Local contact(s)**: S. Ramos	

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Report:

The experiment was carried out using a Paris-Edinburgh press to measure the samples under high pressure and high temperature conditions. The sample was composed of MgO, BN and Ag_2Se fine powder in the appropiate ratios (1:22:2 corresponding to the order in the text). This mixture was pressed into 1.5mm pellets and then loaded into 7mm boron-epoxy gaskets.

The amount of Ag_2Se required to carry out the experiment was obtained from a lump of this compound prepared in Bristol in advance. In order to produce it, a furnace was used to melt and combine the Ag and Se high purity components. Small mesh powder was obtained with fine grain sandpaper. The smallest particles were separated by a suspension and filtration procedure and, once they had been dried, they were mixed in with the MgO and BN powders.

This experiment required the combined use of two techniques: standard XAFS measurements and energy scanning X-ray diffraction for sample characterization and pressure calibration [1]. In order to carry out both types of measurements, 7 CdZnTe semiconductor diodes detectors at fixed angles were added to the standard XAS set-up at the beamline. The MgO and BN in the sample were the pressure markers of the sample and their diffraction peaks were used to calibrate the real pressure.

A single sample was measured during the experiment at different temperatures and pressures. The T-P points studied were chosen according to the previous knowledge of the phase beaviour of Ag_2Se at ambient pressure [2]. It is known that the structure of the system at room temperature is characterised by the orthorombic arrangement of the Se atoms. At ~420K it becomes cubic and the silver ions mobile within it. The melting temperature of the system is just below 1200K. During the experiment, the pressure on the sample was increased in several steps (see Fig.1) from 80bar (oil pressure) up to 700bar (oil pressure) and the temperature at which the transition between the orthorombic and cubic phases occurred was measured by cycling the temperature and measuring the absorption at a fixed energy point. Once the temperature was determined, absorption and diffraction were measured just above and just below this transition temperature (see Fig.1).

All XAS and diffraction measurements at the different temperatures and pressures up to 600bar show that the structure is consistent with the existence of two different phases that go through little change under pressure. A surprising result was obtained when the system was measured at 700bar, where a different structure is clearly observed in both the diffraction and XAS measurements at room temperature (see Fig. 2 for preliminary XAS data). Some preliminary data analysis has shown that the cubic structure is recovered above the expected orthorombic to cubic phase transition temperature (see Fig. 3 for preliminary XAS data). Futher data analysis of the complete set of data is in progress.

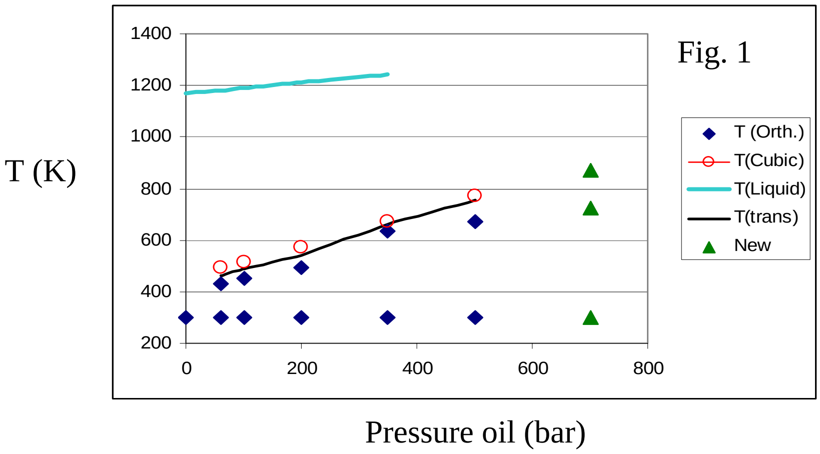
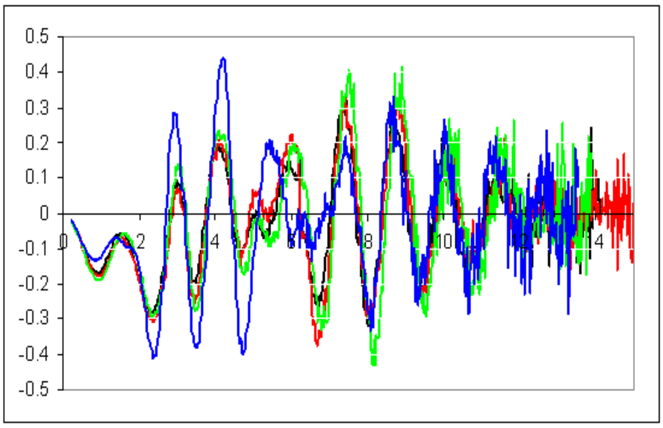


Fig. 1



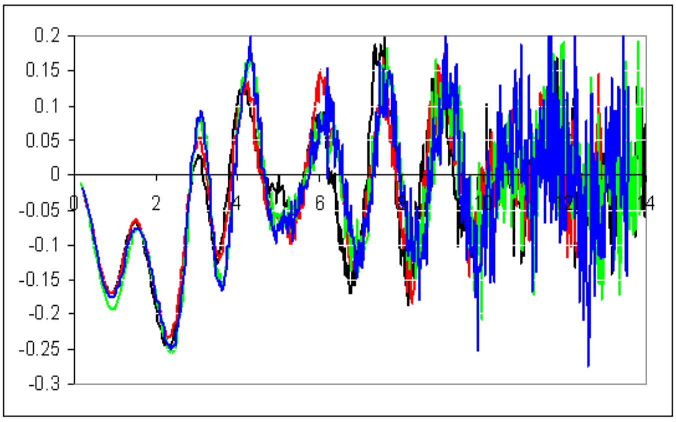


Fig 2: Preliminary $k^2\chi(k)$ data at the Ag K-edge (room T).

Fig 3: Preliminary $k^2\chi(k)$ data at the Ag K-edge (highest temperatre at each pressure point)).

[1] A. filipponi et al. (2000) *Rev. Sci. Instrum* **71**, 2444.
[2] S. Ohno, A. C. Barnes et al. (1994) *J. Phys.: Condens. Matter* **6**, 5335.
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A. C. Barnes et al. (2000) *J. Phys.: Condens. Matter* **12**, 7311.