	Experiment title:	Experiment number:
ESRF	Structural transitions in superionic systems at high pressure and temperature	HS-1196

Beamline:	Date of experiment:	Date of report:
BM29	from: 30-5-2000 (8-11-2000) to: 6-6-2000 (9-11-2000)	16 Feb 2001
Shifts: 21	**Local contact(s):** S. Ansell and M. Borowski	*Received at ESRF:*

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Report:

to achieve the aims proposed, we have collected a series of x-ray absorption spectroscopy (XAS) measurements of AgI over a wide interval of the (P,T) phase space, both at the K-edges of Ag and I. In Fig. 1 we present two prototypical spectra collected during this experiment.

The conditions of high pressure ($1-3$ GPa) and high temperature ($300-1000$ K) have been obtained by a large volume Paris-Edinburgh press, coupled with a 10 mm boron/epoxy biconical gasket. The absorption spectra have been collected in transmission mode through the gasket, simultaneously recording the incident x-ray intensity and the transmitted intensity by means of two Kr/He filled ionization chambers. An AgI sample at ambient pressure and temperature, positioned after the second detector, was used to check for the reproducibility of the energy scale during the entire experiment, by collecting reference spectra through a third ionization chamber.

The optimal sample for the XAS measurement has been obtained mixing a commercial high purity AgI powder with a fine BN matrix powder in a 1:11 weight ratio. Particular care has been devoted to limit any contact of the AgI powder with light or moisture. A small amount of Ge fine powder has been added to this mixture in order to work as pressure marker. This mixture has been then pelleted to a cylindrical shape of about 1.6 mm^3 and inserted into a graphite furnace. This assembly has been finally hosted in the gasket, longitudinally to the direction of the force. A thermocouple has been inserted through the gasket to monitor the temperature.

Using a set-up developed at the BM29 by our group, we have also collected several energy scanning x-ray diffraction (ESXRD) patterns as a function of P and T. By this measurements and using the known Ge and BN equation of state, we calibrated the pressure acting onto the AgI sample. Also, we managed to identify the different cristallographic phases of the AgI at which we executed the XAS acquisitions. Eventually, the disappearence of any diffraction peak from the AgI while rising the temperature attested the melting of the sample.
The phase diagram of AgI in a large portion of the (P,T) space is shown in Fig. 2, where we also indicate the points where we were able to collect both the XAS and the ESXRD measurements. The dashed lines indicate the path connecting the successive measurements.
In addition, we also performed several x-ray absorption temperature scans at fixed photon energy, in order to pin the temperature at which the transitions among the various phases occur as a function of the pressure.
In conclusion, we have been able to probe the local structure of silver iodide under extreme conditions by means of XAS and ESXRD in the room temperature solid, the superionic solid and the liquid, up to 4 GPa and 1100 K. The XAS data are of excellent quality and we are able to observe the structural evolution of the local order as a function of pressure and temperature.
The qualitative results discussed in this preliminary report are very promising and the data are currently under treatement. To complete this project and to obtain the complementary information, we plan to submit a new beamtime request to perform a more focused experiment by angular dispersive x-ray diffraction on this and related systems.
Due to a heavy beam dump during the allocated beamtime in May/June, this experiment had 3 recuperation shifts allocated in November 2000.

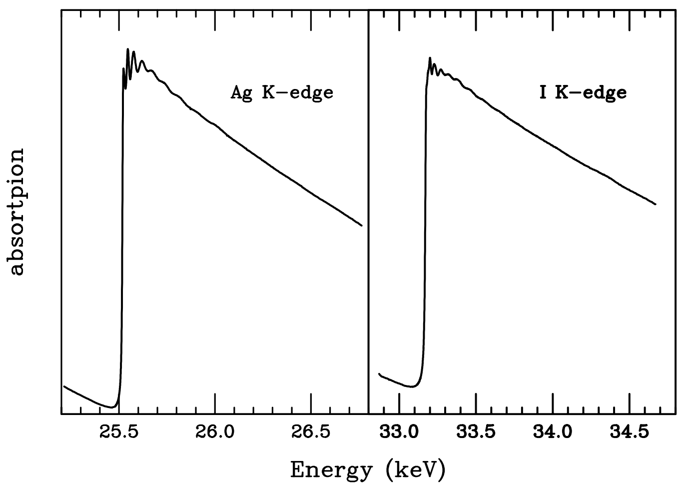

Fig. 1. Prototypical spectra of AgI collected during the experiment HS-1196. Both the Ag and I K-edges are presented for the case of solid AgI at 1.1 GPa and 300 K.

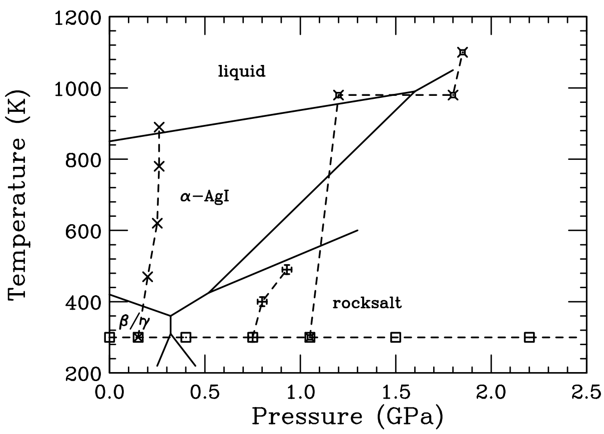

Fig. 2. Phase diagram of AgI. The symbols mark the point of the the (P,T) space where we have collected the XAS and ESXRD measurements. The dashed lines indicate the path between the successive measurements.