| ESRF | **Experiment title:** Static structure of liquid Rb at high pressure and temperature investigated by means of x-ray absorption (XAS) and diffraction (XRD). | **Experiment number:** HD-635 |
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| **Beamline:** BM23 | **Date of experiment:** from: 13 Feb. 2013 to: 19 Feb. 2013 | **Date of report:** 28 Feb. 2013 |
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Report:

We performed a XAS experiment aimed at studying the short range order in solid and liquid Rb as a function of pressure. The experiment was extremely challenging, and we believe we obtained extremely good results out of it.

Rb is highly reactive and we had to confine the sample into containing rings of inert material (KBr or polyethylene) to avoid chemical contact with the gasket. The experimental requirements from the XAS point of view were also very stringent: we had to use nanocrystalline diamonds to avoid single crystal scattering (glitches) in the absorption spectrum which represent a major impediment to XAS measurements at the relatively high energies of the Rb K-edge (above 15 keV). Also, the x-ray beam had to be focused within the sample transversal dimensions ($\sim 100\mu$m). To this we exploit the excellent performances of the K-B focusing device available on the beamline.

A strong collaborative effort with the beamline staff allowed us to collect excellent quality XAS data (signal-to-noise ratio within the $10^{-4}$ limit, result which was unexpected due to the over whole challenges imposed by the experimental requirements) for solid Rb in different crystalline phases up to 31 GPa and room temperature.

We could study two samples, but in both cases it was impossible to probe liquid Rb, even though we managed to heat up the sample above 600 K. Probably the nanocrystalline diamonds which were available at the time of the experiment, and that we could mount on the high pressure membrane cell, were defective, and liquid Rb penetrated the diamonds destroying them.

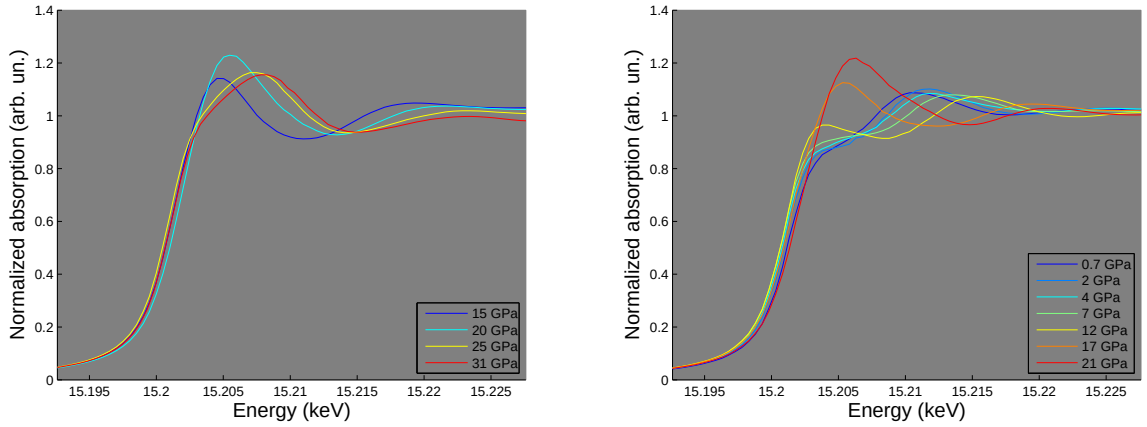


Figure 1: XANES spectra of the Rb samples. Left panel: sample A. Right panel: sample B.

Still, we believe the results we obtained in studying solid Rb as a function of pressure are of unprecedented quality and we believe the experiment was extremely successful: no other XAS measurements are reported for solid Rb at such high pressures. We believe these excellent results put BM23 on the podium of the best XAS beamlines at world level and they allows one to plan further experiments on highly compressed matter. We ourselves are in the process of submitting new proposals along these lines.
Data were collected on BM23 in transmission mode, using a K-B geometry mirror focusing device. Two different samples were loaded in a standard membrane DAC, equipped with nanocrystalline diamond anvils. A polyethylene (sample A) and a KBr (sample B) isolating rings were used to avoid chemical contamination of the Rb sample by the metallic gasket (respectively Re and steel for the two loadings).
Data were acquired at room temperature ($\sim$ 298 K), for pressures up to 31 GPa.
The only experimental issue came from the energy stability of the incoming beam. Apparently, for unknown reasons, the monochromator suffered of some thermal drifting, which resulted in energy shifts $\Delta E$ of few eV. The acquisition of Kr spectra immediately before each Rb spectrum allowed us to realign the spectra *a posteriori*.
In Fig. 1 we show the edge region of the normalized XAS data of both the two samples at several pressures. As said, the data are of excellent quality. The effect of pressure is very well visible, and it nicely agrees with the know phase transformation observed in solid Rb by means of XRD measuements. On top of that, the transformations we observe in the position and shape of the edge provide us with novel information on the electronic evolution of solid Rb with pressure.