ESRF	**Experiment title:** **High pressure XAFS measurements using boron carbide anvils**	**Experiment number**: MI800
Beamline: BM29	**Date of experiment**: from: 26/08/2005 to: 30/08/2005	**Date of report**: 31/08/2005
Shifts: 12	**Local contact(s)**: Sakura Pascarelli	*Received at ESRF:*

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Report:

In the vast majority of high pressure experiments diamond anvils are used to generate pressure. Diamond anvils have several advantages based on their transparency and hardness. The only short-fall of the diamond anvil in x-ray absorption fine structure (XAFS) experiments is its crystal structure. In a XAFS experiment the absorption spectrum is measured as a function of energy. The energy bandwidth is typically > 1000 eV. These photons will inevitably cover energies that satisfy Bragg conditions for the crystal structure of the diamond. This causes the x-rays to be reflected out of the line of the x-ray beam leading to large dips in the transmitted x-ray intensity, that appear as peaks in the spectrum. The occurrence of Bragg peaks remains the main limit to the energy range of the high pressure XAS data and strongly penalizes their exploitation. The aim of this proposal was to implement, for the XAS beamlines at the ESRF, the use of B_4C anvils, mounted on a Chervin typediamond anvil cell (DAC) already available on ID24.

During the months preceding the experiment, Sebastien Pasternak (technician of ID24) established an alignment procedure for non-transparent anvils. The alignment exploits the geometry of the Chervin DAC. We have carried out our first measurements at BM29 using B_4C anvils of 900 μm culet and we used a Cu gasket indented to 55 μm of initial thickness and with 300 μm of diameter hole. Under controlled atmosphere we loaded the sample chamber with a fine powder of 3:1 in volume of arsenic/lithium fluoride. The lithium fluoride served both as pressure transmitting medium and as pressure calibrant. Given the size of the sample the x-ray beam was slitted to 200 μm horizontally and focussed to 100 μm vertically using the mirrors recently implemented of BM29 aligned on the Rh stripe at 3.2 mrad of grazing incidence (3 mrad for diffraction at 20 keV).

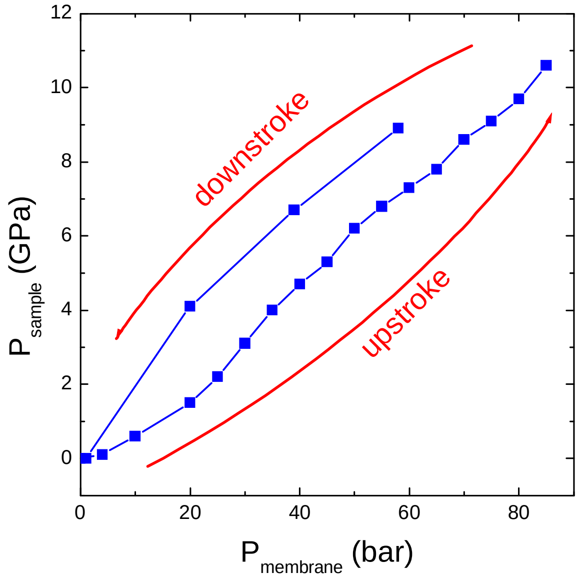

Figure 1*: Pressure in the sample chamber as a function of the membrane pressure*

Diffraction patterns of the sample and of the gasket were recorded at 15 keV and 20 keV respectively using a MAR345 image plate placed in an offset position. The pressure was calibrated using the EoS of LiF and cross checked with that of Cu. Figure 1 shows the pressure of the sample and as a function of the membrane pressure. We reached a maximum pressure of 85 bar on the membrane corresponding to a pressure of 10.7 GPa. We underline that this value is comparable with that achievable using diamond anvils of the same size.

EXAFS spectra (shown in figure 2) of As were recorded at all the pressures at ambient temperature. The k-range of the present data set recorded using B_4C anvils is double of that obtained for the measurements performed using diamond anvils. This data set will allow to verify experimentally the validity the theoretical model according to which Peierls distorted systems such as As have the counterintuitive beheviour of an increasing Debye waller factor as a function of pressure.

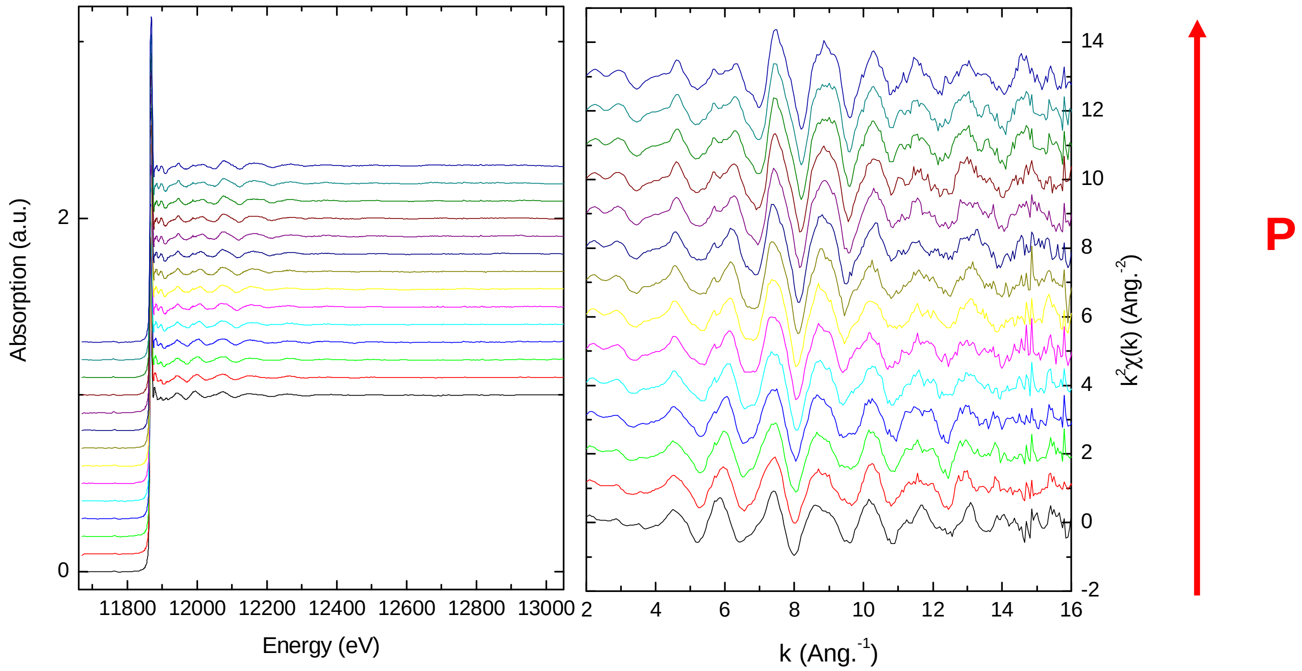

Figure 2: *Raw XAS data are relative extracted $k^2\chi(k)$ of arsenic as a function of pressure*

As a final comment, it is worth to pinpoint that although pressures of ~10 GPa can be reached with the Paris-Edinburgh press routinely installed on BM29, this new set-up allows to handle toxic or air sensitive samples in a safe way thanks to the possibility for these kind of high pressure cells to isolate the sample inside the gasket.