ESRF	Pressure-induced transformations in LBG glass: a better understanding of polyamorphism	**number:** HD 203
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Report:

Introduction

This experiment on ID24 was devoted to complete previous Raman experiments performed on the LBG glass (made with the Lanthanum, Boron and Germanium oxides) which showed a singular behaviour under pressure. The medium range order of this glass is known but no EXAFS experiments have been performed up to now to probe the influence of pressure on the short range order. The aim of our experiment was therefore to carry out *in situ* X-Ray Absorption Spectroscopy up to 20 GPa and then to derive from the data the structural transformations happening during compression and decompression cycles.

Experimental and Results

For our experiments, the sample was introduced in a Diamond Anvil Cell (DAC) that we brought from our laboratory. We firstly performed experiments at ambient temperature and the pressure was increased until 17 GPa. Then we increased the temperature by using an external heating system supplied by the beamline. We worked at the Ge K-edge.

Experiments are summarized below:

Experiment	Sample	Transmitting medium	Pressure	Temperature
1	LBG grain	methanol	$P_{atm} \rightarrow 17$ GPa	T_{amb}
2	LBG grain	meth/eth/water	$P_{atm} \rightarrow 16$ GPa	T_{amb}
3	LBG grain	meth/eth/water	$P_{atm} \rightarrow 10$ GPa	T_{amb}
4	LBG powder	meth/eth/water	$P_{atm} \rightarrow 15$ GPa	240^oC
5	$LaBGeO_5$ powder	meth/eth/water	$P_{atm} \rightarrow 17$ GPa	240^oC
6	LBG powder	meth/eth/water	$P_{atm} \rightarrow 16$ GPa	T_{amb}

We also performed EXAFS experiments outside the DAC on crystalline quartz and rutile GeO_2 as references for four-fold and six-fold coordinated Ge respectively.
For each experiment, we measured XANES spectra at differents pressures and we observed for each an evolution of the edge region with pressure as shown in figure 1. The position of the white line at lowest and highest pressures can be compared with those of q-GeO2 and r-GeO2. This comparison shown on figure 2 leads us to confirm that the change of coordination numbers from 4 to 6, previously suggested by Raman spectroscopy, is happening.

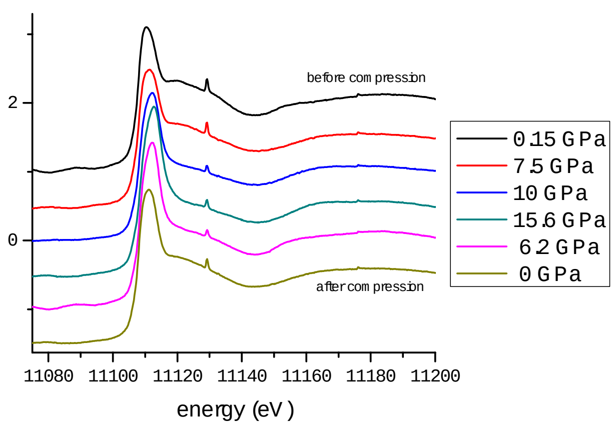

fig.1: Influence of pressure on XANES spectra for experiment 2

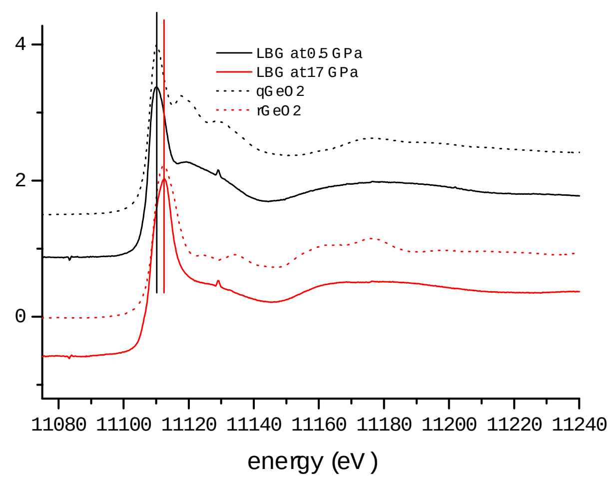

fig.2: Comparison of XANES spectra with q-GeO2 and r-GeO2 at extreme pressures for experiment 1

With the experiment 3 we wanted to check the reversibility of the structural transformations up to 10 GPa. This point seems to be different from what was observed with Raman spectroscopy and it will be discussed. The aim of the experiment 6 was to recover the densified LBG glass in order to measure it outside the cell, where the energy range is not limited by the glitches from diamonds. The influence of temperature was also studied in experiments 4 and 6.

Conclusion We obtained a lot of precious results from these days spent on ID24, about the structural transformations occurring during compression of LBG glass. Contrary to other glasses containing Germanium oxides, the LBG glass shows an irreversible transformation under pressure up to 17 GPa. This ability to show polyamorphism is noticeable and we should now explain why it has such a behaviour. We would like to thank our local contact who showed a very deep interest to our experiments!