ESRF	**Experiment title: Effect of pressure on depolymerized lithium borates**	**Experiment number**: HC1982
Beamline: ID20	**Date of experiment**: from: 15/07/2015 to: 21/07/2015	**Date of report**: 08/07/2016
Shifts: 18	**Local contact(s)**: Christoph SAHLE	*Received at ESRF:*

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Report:

Recent non-resonant inelastic X-ray scattering (NRIXS) studies showed that borate glasses exhibit pressure-induced coordination transformation, with the conversion of three-fold coordinated boron ($^{[3]}$B) into four-fold coordinated boron ($^{[4]}$B).[1–3] It has been observed that Li-borate (Li_2O-$2B_2O_3$) and pure borate (B_2O_3) glasses show a non-linear boron coordination change while Na-borate glasses show a linear transformation.[2,3] This effect of the network-modifying cations, and also observed for the network-forming cations by X-ray absorption spectroscopy,[4,5] shows that the composition and the nature of additives control the pressure-induced structural modification.

Recently, we evidenced a clear signature of the NBOs in the NRIXS spectra of lithium borate crystals around the O K-edge.[6] This feature around 535eV is only observed in partially and fully depolymerized lithium borate glasses and crystals containing at least 30-40% lithium oxide content. While the previous studies[1–3] were focused on fully polymerized borate glasses (B_2O_3 and $Li_2B_4O_7$ and Li content < 33%), the present experiment deal with lithium borate glasses characterized by a depolymerized borate framework.

We have performed an NRIXS experiment on ID20 in order to access the boron and oxygen K-edges in a high-pressure environment. We have used a diamond anvil cell provided by ESRF and offering a large aperture. Measurements were made through Be gasket. An energy resolution as high as 0.6 eV was required in order to be more sensitive to slight changes in the pre-edge profile. Two compositions were studied: Li_2O-B_2O_3 and $3Li_2O$-$2B_2O_3$. For a better understanding of the high-pressure data, we have first measured under pressure two reference samples in the crystalline state, in order to follow the evolution of the local environment around B and O atoms. Then, in a second set of measurements, the high pressure spectra of the two vitreous samples v-$LiBO_2$ and v-$Li_6B_4O_9$ were collected at 0, 10 and 20 GPa. As an example, the B and O K-edges spectra of vitreous $LiBO_2$ are shown on the Figure 1. We can see important modifications of the electronic structures of both atoms induced by pressure, which are caused by severe structural rearrangements. The most spectucular is the threefold to fourfold-coordinated boron conversion. The O K-edge spectrum experiences also important modifications with pressure, highlighting an important modification of the angular distribution of the B-O-B

inter-polyhedra angle. A decrease of the NBO feature around 535eV is also pointed out, which indicates a recombination of the NBOs during pressurization. Ab-initio calculations are under progress in order to determine the mechanisms at the origin of this pressure induced polyamorphism.

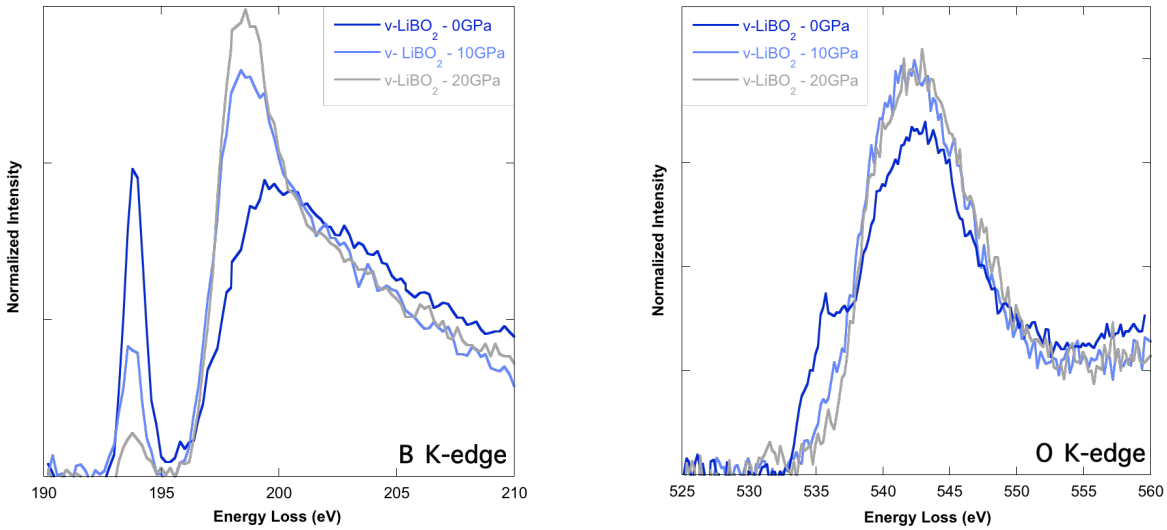

Figure 1 : B K-edge (Left) and O K-edge (Right) of vitreous $LiBO_2$ measured in a diamond anvil cell at 0, 10 and 20GPa.

In conclusion, the reference samples clearly indicate the possibility to follow the evolution of both the boron and oxygen local environments as a function of pressure thanks to the great capabilities of the ID20 beamline. Successful measurements have been obtained at high-pressure using a panoramic diamond anvil cell in a reasonable accumulation time, which will be improved in further experiments.

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