	Experiment title: Effect of network modifiers on silicate melt densification at Earth's lower mantle conditions	Experiment number: ES1177
Beamline: ID24	**Date of experiment:** from: 13/06/2023 to: 19/06/2023	**Date of report:** 29/08/2023
Shifts: 18	**Local contact(s):** Angelika Rosa, Olivier Mathon	*Received at ESRF:*

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Report:

The project aims at investigating the local structure of minor network-modifying cations in silicate glasses and melts at conditions of the lower mantle. This experiment represented the first one that used the newly installed laser-heating setup that is dedicated to EXAFS and XANES at extreme conditions at ID24. Just before and during this experiment the laser heating was successfully commissioned and calibrated. All optical components were aligned, which included the laser path as well as the optical path for sample observation and temperature measurement using Schwarzschild optics. In order to collect spectra of minor components a Vortex SDD fluorescence detector was set up in back scattering arrangement. The fluorescence signal from the sample in the diamond anvil cell (DAC) was spatially filtered using a focussing poly-capillary in front of the detector in order to suppress elastic and inelastic scatter background and improve the fluorescence-signal over background (Figure 1). Spectra were recorded in transmission mode for Sr (16 wt% SrO) and in fluorescence mode for Y (5000 ppm Y). The beam was focused to 0.5 × 0.5 μm² using the KB-mirror.

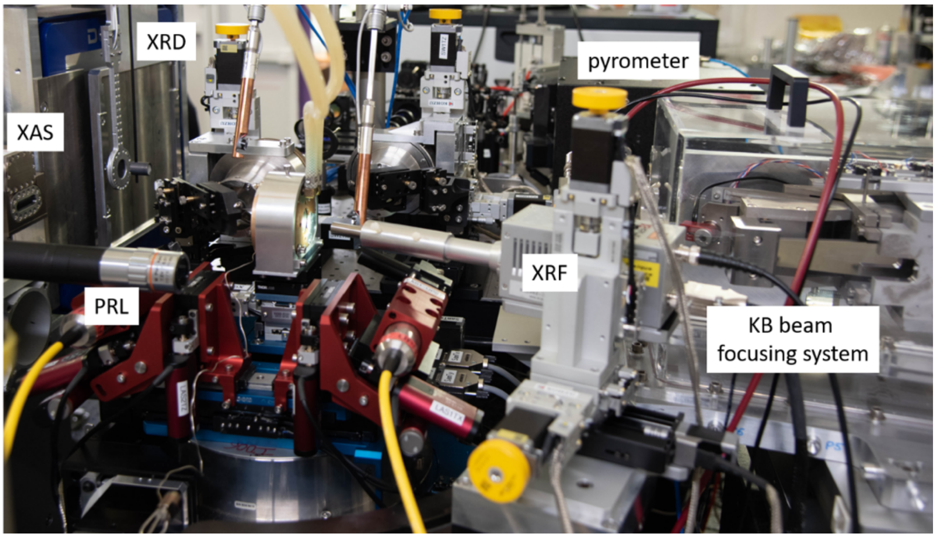

Fig. 1. Experimental setup at ID24-DCM.

Along with the laser-heating commissioning, we were able to collect compression series of spectra on both Sr and Y in a simplified AbDi glass (Krstulovic et al., 2021) up to 117 GPa as shown in Figure 2. For Sr, XANES spectra indicate a continuous decrease in the Sr-O distance by the shift of the first maximum of the EXAFS to higher energies as well as an increase in site distortion as indicated by enhanced intensity of the pre-edge at ca. 16090 eV. For Y, the strongest changes are observed between 2 and 16 GPa, which indicate a change in site symmetry. At higher pressures spectra indicate a continuous decrease of the Y-O distance.

Laser-heating was tested on the same glass composition at various pressures. Successful heating up to complete melting with in-situ spectra recording is shown in Figure 3 for 2 and 9 GPa. At higher pressures and up 1o 115 GPa, it was difficult to reach stable heating conditions above liquidus temperatures. Spectra recorded below 3000 K indicate recrystallisation of the glass as shown in Figure 3 at 52 GPa. We suppose that the difficulties during laser-heating are mainly related to the low Fe content of the used glass (2 wt% Fe_2O_3). The heating performance could be improved in a much better way at higher Fe-contents as would be the case for natural basaltic compositions. Analysis of the EXAFS region of all spectra is ongoing.

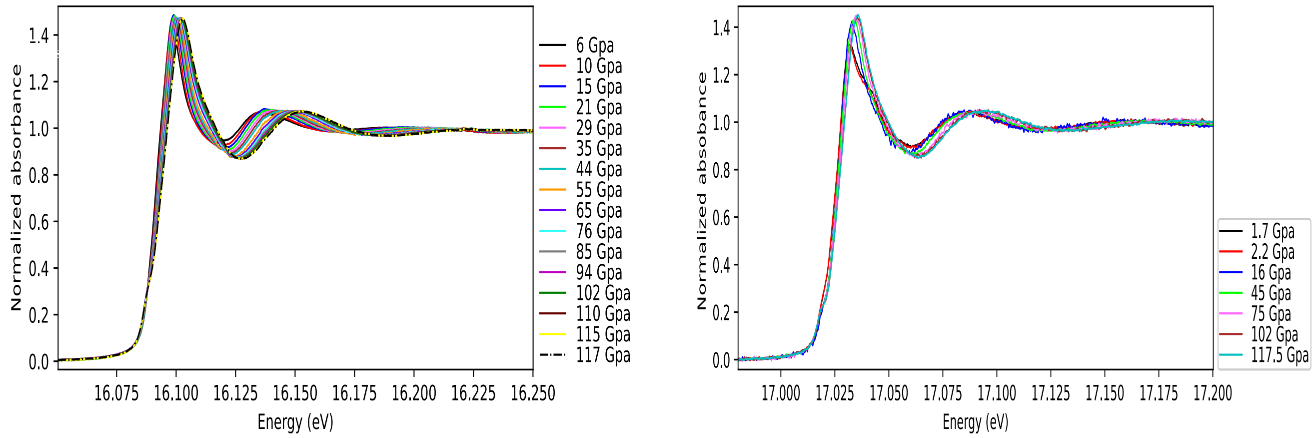

Figure 1: Left: Evolution of XANES at the Sr K-edge with pressure. Please, note the shift of the 1^{st} EXAFS maximum (ca. 16130 eV) to higher energy representing the shortening of the Sr-O distance. Further, enhancement of a pre-edge feature (ca. 16 090 eV) indicates increasing site distortion. right: Evolution of XANES at the Y K-edge. Largest changes in the spectra occur between 2 and 16 GPa, especially in the white line region, which may indicate a change in site symmetry. 1^{st} EXAFS maximum (ca. 17080 eV) shifts to higher energy representing the shortening of the Y-O distance.

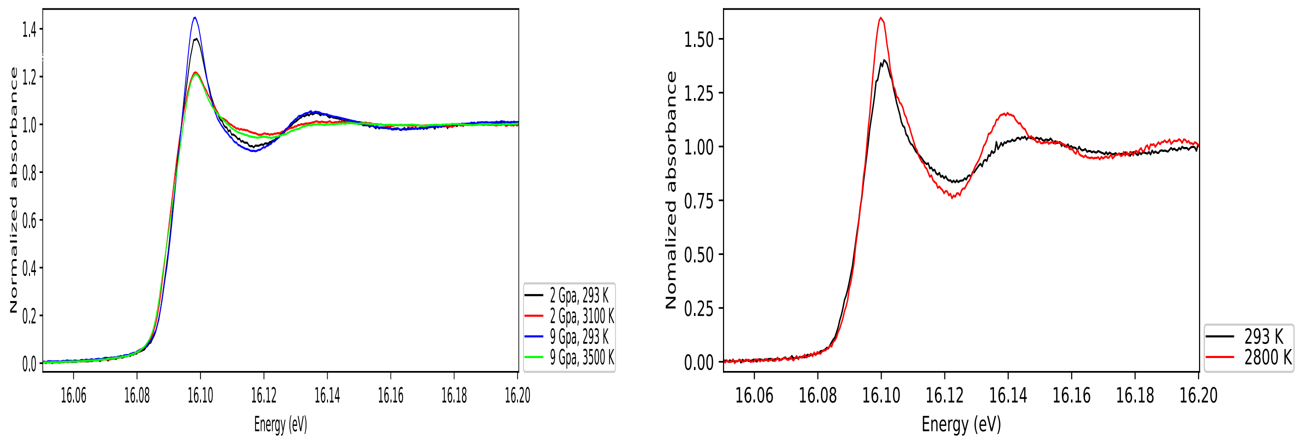

Figure 2: Left: Comparison of Sr spectra between glass and melt at pressures and temperatures indicated. The loss in amplitude is related to the increased disorder in the melt. right: Comparison of Sr spectra of glass and heated sample below melting at 52 GPa. The enhanced amplitude of the heated sample is related to the enhance order of the crystal. At higher temperatures no stable heating could be established.

Reference:

Krstulović M et al. (2021) Chemical Geology 560, 119980, https://doi.org/10.1016/j.chemgeo.2020.119980