



Experiment title: INVESTIGATION OF THE HIGH-TEMPERATURE BEHAVIOR OF TRANSITION ELEMENTS IN NATURAL MELTS

Experiment number:
ME-154

Beamline: ID 26	Date of experiment: from: 20/06/01 to: 25/06/01	Date of report: 25/02/02 <i>Received at ESRF:</i>
Shifts: 18	Local contact(s): Pierre-Emmanuel Petit	

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Report: In order to understand the structural silicate-melts properties, particularly localized around transition elements, we performed some *in situ* measurements under extreme conditions, at high-temperature and high-pressure (HT-HP). We used a new experimental procedure by associating the Paris-Edinburg press (ID 30) with ID 26 beamline. HT-HP measurements were done up to 1200 °C and 5 GPa respectively. We collected series of quick-XANES spectra at different edges, in anhydrous samples. The first sample studied was an anhydrous albite-like glass ($\text{NaGaGe}_3\text{O}_8$), in which aluminum was substituted to gallium, and silicon was substituted to germanium. Both, Ga and Ge K-edges (10367 and 11103 eV respectively) XANES were collected at 2.16 GPa and up to 900 °C. Figure 1 presents the XANES spectra collected at the Ge K-edge. In this fully polymerized glass sample, Ge is 4-coordinated at the ambient conditions. The results show that there is no structural modification around Ge in the albite-like glass due to the pressure effect between 1 bar and 2.16 GPa at ambient temperature. Moreover, no changes in the XANES spectra can be observed when the

temperature increases up to 900 °C. Consequently, the structural environment of Ge is not modified under these HT-HP conditions. However, the melt-state was not obtained because of the too low signal-to-noise ratio on XANES spectra at these extreme conditions.

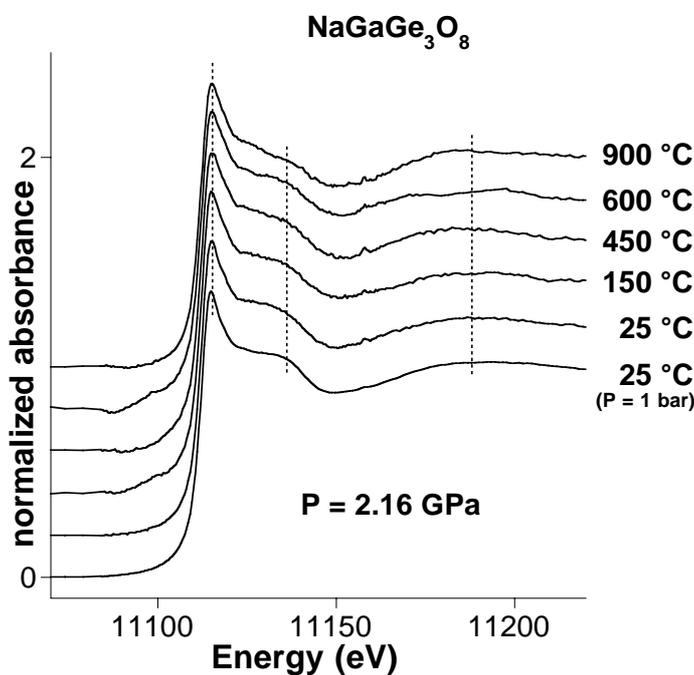


Fig. 1 - XANES spectra collected at the Ge K-edge, up to 2.16 GPa and 900 °C, in NaGaGe₃O₈ glass sample.

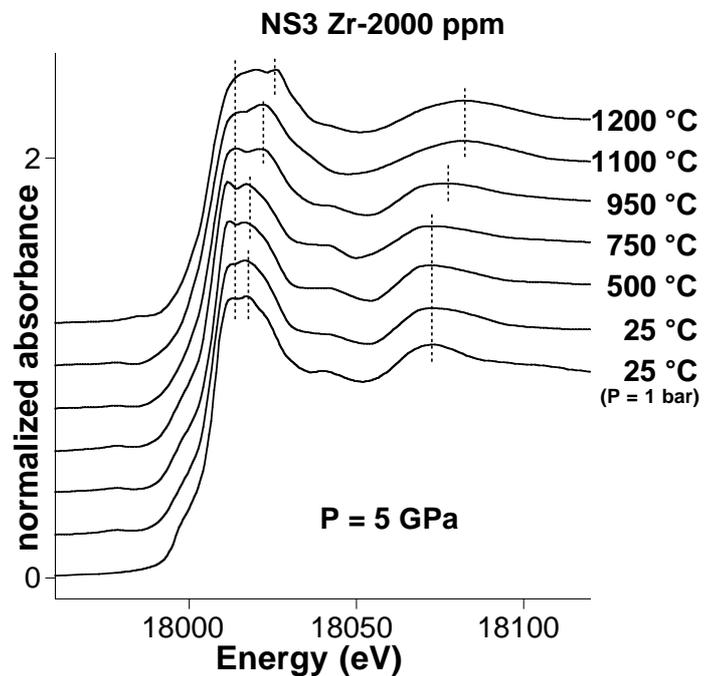


Fig. 2 - XANES spectra collected at the Zr K-edge, up to 5 GPa and 1200 °C, in NS3 Zr-2000 ppm glass sample.

Finally, we collected some series of quick-XANES spectra for an ultra-diluted sample, NS3 glass (Na₂Si₃O₇) containing 2000 ppm of zirconium, in which Zr is 6-coordinated. Zirconium K-edge XANES spectra, collected at 5 GPa and up to 1200 °C, are presented in Figure 2. No significant changes due to the pressure (5 GPa) can be observed at ambient temperature. However, at 5 GPa and around the glass transition temperature (\approx 950 °C), some changes appear in the XANES. Those changes are more easily identified in the melt-state (around 1200 °C). Particularly, one can observe the white-line that is divided in two features, and a significant shift toward the high energies for the first EXAFS oscillation. Such changes in the XANES spectra typically traduce some modifications of the short-range environment, suggesting Zr in a lower coordination number in the *in situ* state, which is in excellent agreement with our previous studies on nickel in silicate melts (Farges et al., 2001).