ESRF	**Experiment title:** Incorporation of Xenon and Krypton in crust relevant minerals: a high pressure and temperature XAS study	**Experiment number**: ES-442
Beamline: BM23 (C04)	**Date of experiment**: from: 2/11/2016 to: 8/11/2016	**Date of report**: 19/01/2017
Shifts: 18	**Local contact(s)**: Angelika Rosa	*Received at ESRF:*

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Report:

The aim of this experiment was to characterize the structural environments of Kr and Xe (noble gases) in silicates at high pressures and temperatures. Indeed elemental Xe is missing in the current atmosphere compared to primitive meteorites (which is not the case for Kr) and storage of Xe in silicate at depth (crust and upper-mantle) has been recently proposed. Present experiments were dedicated to test this hypothesis and study possibility and mechanisms of Xe incorporation in silicates. Kr was investigated to discriminate a Xe-specific behaviour. XAS measurements were performed on BM23 using Paris Edinburgh Press (PEP) and nano-polycrystalline diamonds in order to avoid glitches in the XAS spectra at the Kr K-edge (14326 eV) and at the Xe K-edge (34561 eV) in transmission and fluorescence modes on haplogranite (a proxy of the continental crust) and sanidine (high temperature feldspar). Xe and Kr gas standards were analyzed throughout beamtime to correct for eventual shift. Summary of exploitable measurements is given in Table 1. To date, there is no published data on X-ray absorption spectroscopy of Xe in silicates and only one study on XAS of Kr in SiO_2 glass at ambient conditions [1]. In the course of experiment we successfully retrieved EXAFS signals of Kr and Xe in sanidine glass at around 2-3 GPa and different temperatures. Spectra presented in figure 1 are currently analyzed and should enable to better understand Xe and Kr incorporation in silicate glass. At around 1300°C (after recrystallization of the glass), we observed a dramatic change in

absorption spectrum of Xe in sanidine glass (see figure 2) which may be due to Xe incorporation in minerals. However EXAFS signal is not exploitable, probably due to low Xe content in minerals compared to the glass. Xe content in haplogranite (0.5 wt % Xe) was too low to exploit EXAFS signal (despite 10 hours analysis in fluorescence mode). However careful analysis of XANES signal indicates a 1 eV shift between starting glass and compressed melt due to Xe oxydation in the haplogranite melt.

Cell3 (oil pressure : 200 bar)		
Glass of haplogranite (0.5 wt% Xe)		
Xe K-edge (fluorescence and transmission modes)		
P (GPa)	**T (°C)**	
1.6	905	rec.
1.6	1100	rec.
1.8	1460	melt
Cell1 (oil pressure : 300 bar)		
Glass of sanidine (2.6 wt% Xe, 1.7 wt% Kr)		
Xe K-edge (transmission mode)		
P (GPa)	**T (°C)**	
1.6	20	glass
1.7	200	glass
1.8	305	glass
1.9	390	glass
2.0	500	glass
2.1	585	glass
2.3	765	glass
2.5	985	glass
2.7	1165	glass
2.9	1330	glass / rec. ?
3.0	1415	rec.
2.8	1310	rec.
2.7	1195	rec.
(2.7 ?)	1040	rec.
(2.7 ?)	20	rec.
Cell1 (oil pressure : 300 bar)		
Glass of sanidine (2.6 wt% Xe, 1.7 wt% Kr)		
Kr K-edge (transmission mode)		
P (GPa)	**T (°C)**	
0.0	20	glass
0.0	20	glass
2.6	920	glass
2.7	1085	glass
2.0	706	glass
2.0	505	glass
1.4	20	glass

Table 1: summary of measurements performed during beamtime. P was determined using calibration of h-BN (? = no calibration available for this data point) and T based on calibration from [2]. rec. = recrystalized sample.

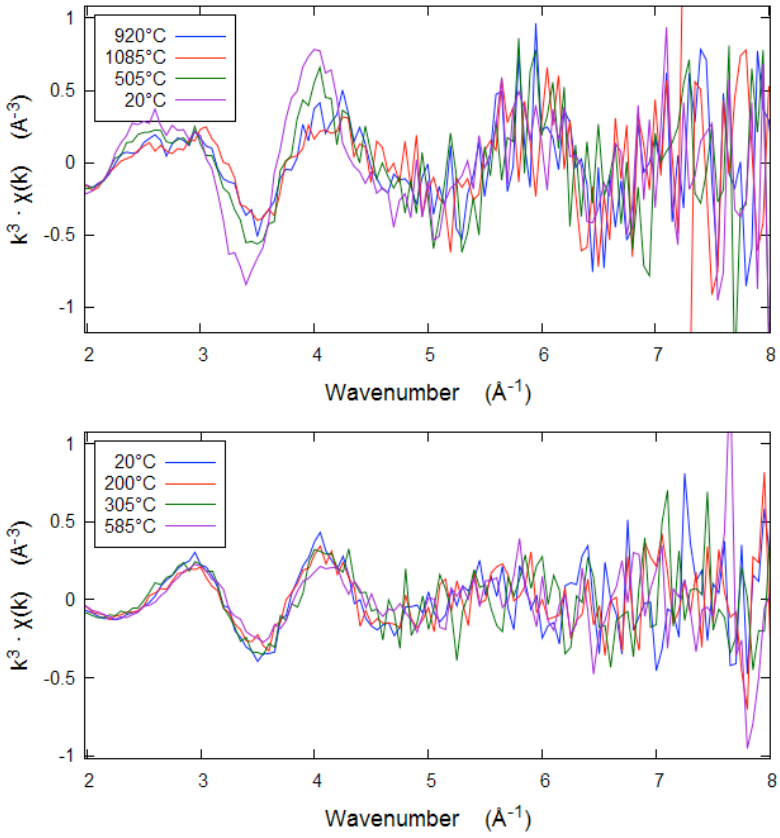

Figure 1: EXAFS k^3 weighted $\chi(k)$ function of the merged experimental data. Top: Kr K-edge, bottom Xe K-edge. See pressures in Table 1.

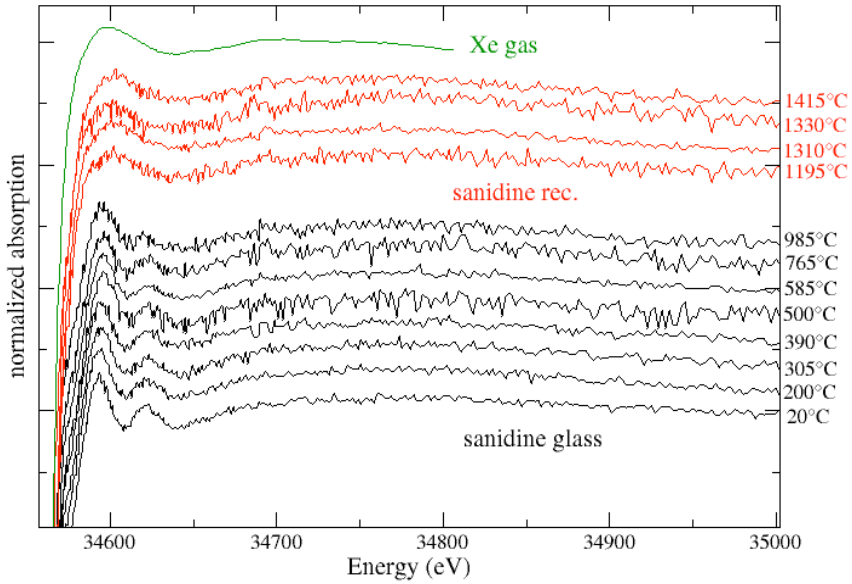

Figure 2: normalized absorption spectra at 2-3 GPa retrieved in sanidine at Xe K-edge in transmission mode. (merge of scans are represented)

References: [1] Wulf et al, *Am. Mineral.*, 84, 1999; [2] Kono et al, *Phys. Earth Planet. Inter.* 228, 2014