	Experiment title: Dissolution of feldspars at upper mantle P-T conditions: an *in situ* Synchrotron X-ray fluorescence study.	**Experiment number**: HS-2099
Beamline: ID22	**Date of experiment**: from: 9/04/03 to: 17/04/03	**Date of report**:
Shifts: 21	**Local contact(s)**: Alexandre Simionovici	*Received at ESRF:*

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Report:

During the allocated beam time in ID22 (experiment HS-2099), a new experimental set-up for fluorescence measurements in a standard diamond anvil cell (DAC) has been developed. This setup allows to analyze elemental concentrations as low as 40 ppm in a fluid over the 10 GPa-500 ºC pressure-temperature range. Using this new experimental set-up, *in situ* measurements of the solubility of strontium feldspars ($SrAl_2Si_2O_8$) in high pressure-high temperature (HP-HT) aqueous fluids (up to 6 GPa and 450ºC) have been performed. These experiments are the continuation of the study that our group is carrying out from November 2001 (reports HS-1632, HS-1937), in order to constrain the reactivity of deep aqueous fluids toward mantle minerals in deep subduction zones in the Earth.

The experimental setup comprised a set of 98 parabolic Al Compound Refractive Lens (CRL), ensuring the focusing of the monochromatized X-rays at 23 keV in a spot size of 2x18 μm^2 with a flux of 5.10^9 photons. The intensity of the incident and transmitted beam was monitored by silicon PIN-diodes located before and after the sample. X-ray fluorescence was collected using an energy dispersive solid state Si(Li) detector of 150 eV resolution at 5.98 keV. As discussed in Ref [1], the angle for fluorescence collection imposed by the geometry of the standard DAC (experimental reports HS1632 and HS1937) is the major limiting factor for elemental analysis at very low concentration levels during the solubility processes at HP-HT conditions. The expected low solubility of feldspar minerals requires lower detection limits than those previously achieved [1]. In order to lower the detection limits, our DAC has been slightly modified: the diamond at the entrance of the beam, typically 2.2 mm in previous experiments

[1], was replaced by a 1.2-mm thick diamond. Reducing the X-rays path in the diamond by 1-mm allows to reduce both the scattering phenomena and the attenuation of the characteristic fluorescence lines emitted by the sample. Furthermore, reducing the thickness of the diamond window increases the optical aperture of the cell, hence much higher collection angles are possible. The Si(Li) detector was set at 30° from the incoming beam instead of the maximal 16º in previous works. Figure 1 illustrates the spectrum recorded in a solution containing 50 ppm of Rb and 40 ppm of Sr using the "modified cell" and 30° backscattering geometry (a). In the figure is also reported the spectrum recorded in the same solution using the 16° backscattering geometry and 2.2-mm diamond (b). This figure demonstrates that the elemental detection in a standard DAC can be lowered to 40 ppm without any reduction of the strength of the anvil, allowing to perform SXRF analysis up to 10 GPa and 600 ºC. These progress in quantitative elemental analysis in HP-HT fluids were presented at the 19th AIRAPT – 41st EHPRG meeting (International Conference on High Pressure science and technology, Bordeaux 2003) and a manuscript [2] will be published in the proceedings of the conference (Journal of Physics: Condensed matter).

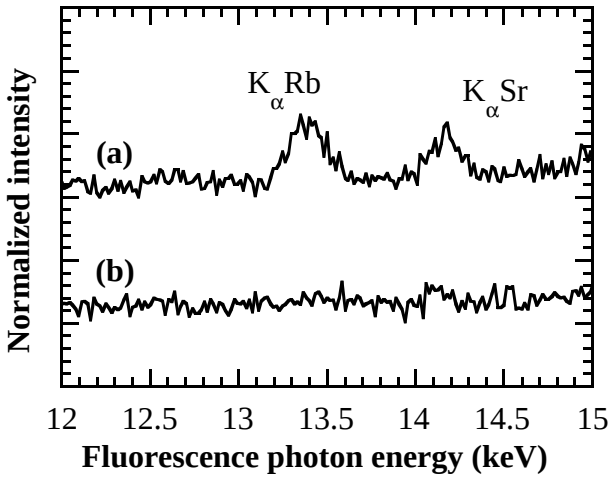

FIG. 1. X-ray fluorescence spectra collected in backscattering geometry in an aqueous solutions containing 50 ppm of Rb and 40 ppm of Sr: (a) 30º collection angle (1.2-mm thick diamond); (b) 16º collection angle (2.2-mm thick diamond).

The experimental setup described was then employed for *in situ* measurements of the solubility of strontium feldspars ($SrAl_2Si_2O_8$) up to 6 GPa and 450ºC. The DAC was equipped with an external resistif heater. The sample chambers were formed from 80-μm thick rhenium foils drilled with two separate holes: a large one (~150-200 μm) loaded with the $SrAl_2Si_2O_8$ crystals in an aqueous solution for solubility measurements, and a second one (~50 μm) loaded with a ruby chip in solid paraffin for pressure determination. The isolation of the ruby pressure gauge is required by the high chemical reactivity of the HP-HT aqueous solutions, resulting in the completely dissolution of the sensor up to 300 ºC. Then, fluid composition at different P-T conditions was analyzed by monitoring the fluorescence of Sr and Rb cations in the fluid, following the methodology developed in Ref [1]. The treatment of the data is currently in progress. The preliminary results show that $SrAl_2Si_2O_8$ dissolution is incongruent under the studied P-T conditions. Several secondary mineral phases precipitated during the process and they could not be identified by X-ray diffraction during the experiment. The presence of these heterogeneities in the solution makes difficult the interpretation of the data.

References

[1] Sanchez-Valle et al. 2003. Dissolution of strontianite at high P-T conditions: An *in situ* synchrotron X-ray fluorescence study. *American Mineralogist*, 88, 978-985.

[2] Sanchez-Valle et al. 2003. Progress in quantitative analysis of high P-T fluids using SXRF. In preparation for *Journal of Physics: Condensed matter*.