EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

Once completed, the report should be submitted electronically to the User Office using the
**Electronic Report Submission Application:**

*http://193.49.43.2:8080/smis/servlet/UserUtils?start*

***Reports supporting requests for additional beam time***

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| ESRF | **Experiment title:**<br>**In-situ SXRF determination of Pb partitioning in hydrothermal diamond anvil cell.** | **Experiment number**:<br>CH1267 |
|---|---|---|
| **Beamline**:<br>Id22 | **Date of experiment**:<br>from: April 24th 2002 to: April 30th 2002 | **Date of report**:<br>July 2002 |
| **Shifts:**<br>18 | **Local contact(s)**: Alexandre Simionovici, Andrea Somogyi. | *Received at ESRF:* |

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## Report:

Diamond Anvil Cell experiments at high pressures (0.3 – 1.2 GPa) and temperatures (25 – 800°C) were performed on the Id22 µFID beamline. In order to understand magma genesis in subduction zones, the fluid/melt partition coefficients of Pb were experimentally studied. The equilibrium between aqueous fluids and silicate melts before and after their complete miscibility, and the composition of the different components in these phases were characterized *in situ* at a micrometer scale for every stage of the high pressure and temperature experiments (Fig. 1).

The experimental setup comprised a Compound Refractive lens, ensuring the focusing of monochromatized X-rays at 15 keV, with a flux of $5.10^9$ photons per second. In order to restrict the scatter beam, a Pt/Cu pinhole of 5 µm diameter was positioned behind the lens, thus allowing to obtain a spacial resolution of 1.5*5 $\mu m^2$. The beam was monitored by silicon PIN-diodes. The incident beam irradiating the cell was normal to the diamond surface. An X-ray CCD camera was set in transmission geometry to observe the sample during the heating stages. X-ray fluorescence measurements were carried out with an energy dispersive solid state Si(Li) detector, set at 11.5° to the incident beam, in the horizontal plane. Experiments were carried out with an externally heated Bassett-modified Hydrothermal Diamond Anvil Cell, which allows a direct visual observation of the equilibrium between components at high pressures and temperatures. The sample was contained in a rhenium gasket compressed by two diamond (sample chamber diameter 400-200 µm). Pressure medium in all experiments was an aqueous fluid and was calculated, at a given temperature, from the appropriate equation of state of the fluid phase.

Starting materials were a synthetic haplogranitic glass together with one aqueous fluid, either pure water or NaCl aqueous solution (15 g/l). Lead was alternatively present in solution or in synthetic glass. To monitor the compositions of coexisting fluids and melts during the heating stages and also in the vicinity of the miscibility, trace element distributions and concentrations were determined *in situ* during experiment

(Fig. 2 and 3). Single analyses were recorded with an acquisition time of 2 to 15 min. 2D scans of the cell were performed using a 30 s per step integration time.

The determination of Pb partition coefficient ($D^{Pb}_{fluid/melt}=C^{Pb}_{fluid}/C^{Pb}_{melt}$) between hydrous silicate melts and aqueous fluids in the P-T conditions corresponding to the upper mantle in subduction zones is currently in progress. Results were presented at two meetings (VII International Conference on X-Ray Microscopy, Grenoble July 2002; Diffusion Inélastique des Rayons X et Photoémission de Haute Energie à Soleil, Paris Juin 2002). They provide information on (1) the nature of the contaminant agent (a silicate melt or an aqueous fluid) responsible of the unique Pb signature in subduction zone magmas; (2) the potential role of chlorine in this contamination process.

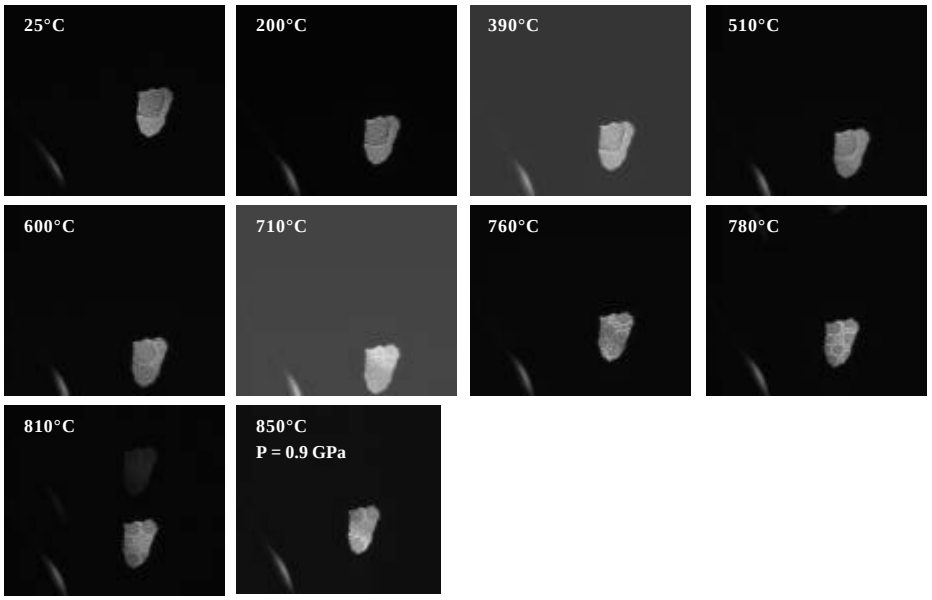


Figure 1: Globules of melt (dark grey) in the aqueous fluid (light grey) observed from CCD camera. These phase contrast images (0.1 µm spatial resolution) are obtained together with the fluorescence measurements at different temperatures.

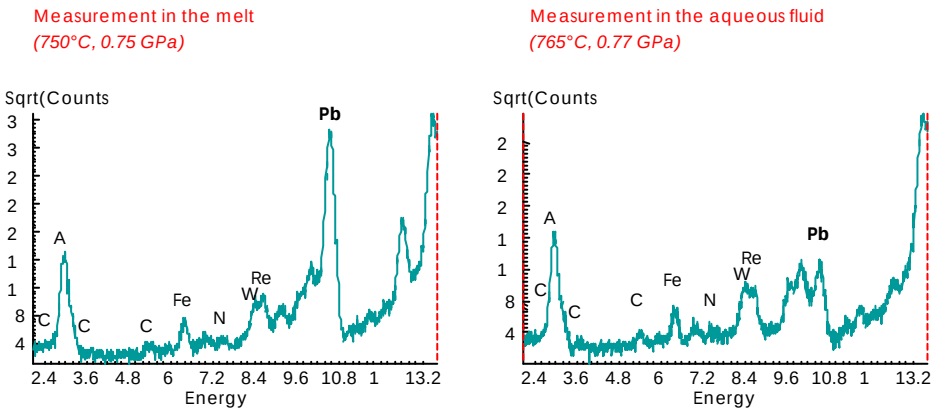


Figure 2: SXRF spectra obtained on globules of melt and fluids, respectively, with a 15 keV incident energy.

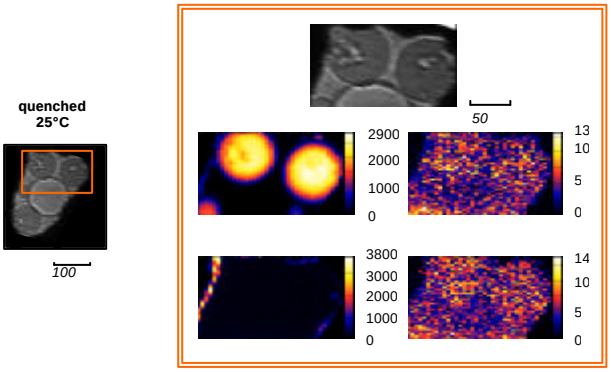


Figure 3: Elemental distribution (Pb, Re, Cl, Ca) in the different phases performed after the experimental quench.