EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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 via the User Portal: *https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do*

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** Experimental constraints on the origin of volatile elements on Earth.	**Experiment number**: ES-1306
Beamline: ID16B	**Date of experiment**: from: 21/06/2023 to: 26/06/2023	**Date of report**: 07/09/2023
Shifts: 15	**Local contact(s)**: Valentina Bonino (email: valentina.bonino@esrf.fr)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Julien Siebert* (IPGP)

Lucas M. Calvo* (IPGP)

Ingrid Blanchard* (IMPMC)

Report:

This project focuses on understanding the most significant geochemical differentiation process occuring early on Earth: the segregation of the core from the bulk silicate Earth during the so called magma ocean stage. In this context, chemical elements are distributed between metal (core) and silicate (mantle) according to their affinity for metallic phases defined as the siderophility of elements. Some siderophile elements can also be volatile elements (i.e. low condensation temperature) and their depletions in the bulk silicate Earth with respect to solar composition (Wood et al., 2006) can be accordingly explained either through partitioning into metal phases or by volatilisation. Furthermore, the observed distribution of certain elements on Earth are assumed to be produced by the late delivery of extraterrestrial material after core formation ceased (Righter et al., 2017), the so-called 'late veneer' hypothesis. Sulfur (S), selenium (Se), and tellurium (Te), are siderophile and volatile elements which share similar condensation temperature (~700 K) and depletions in silicate Earth. The experimental study of the partitioning of these elements between metal and silicate can accordingly be used to decipher in between an early arrival of volatile elements on Earth, which abundances would be affected by core formation, or a late arrival from late accretion only.

In this research, we aim to determine for the first time the partitioning of Se, Te and S between metal and silicate phases at direct pressure and temperature of Earth's core formation (P >40 GPa, T >3000 K). By performing laser-heating diamond anvil cell (LH-DAC) experiments, we equilibrated at extreme P-T conditions metal carrying Se, Te and S with a silicate of peridotitic composition, similar to the primitive magma ocean. The twelve DAC samples, and one glassy standard (synthesised using a piston-cylinder apparatus) were prepared for analyses in FIB thick sections between 2 and 4 µm (Fig. 1). X-ray Fluorescence at the ID16B beamline offers both the analytical and spatial resolution to measure trace abundances of Se and Te (few tens ppm) in the small quenched silicate phase (< few microns) of the DAC samples which are well below the detection limits and spatial resolution achievable with traditional analytical techniques (e.g. electron probe microanalyses). The X-ray Fluorescence maps performed during the 15 shifts at the beamline ID16B seem promising thanks to the help of the local contact present at ESRF, Valentina Bonino, despite we faced unpredictible issues such as electric storms or server connection failures for which the data collection was interrupted, or troubles with the holder glue, which unsticked during some long measurements. Consequently, fewer maps than expected were collected.

The nanoXRF maps were performed mostly at high resolution (100 nm) with long exposure time (500 ms) for acquisition of good quality data. In the analysed run products, we collected quantitative XRF maps for Se and Te encompassing regions at the interface in between metal and silicate phases. In order to quantify the elemental concentration on melts, we define Regions Of Interest (ROIs) which are analysed by using the software PyMCA, developed at ESRF (Solé et al., 2007). With these measurements, we demonstrated that the low concentrations of Se and Te in the silicate phases can be detected. This will allow us to constrain for the first time the partitioning of these elements at direct conditions of Earth's core formation in a deep magma ocean.

Finally, we can argue that the beamtime was very productive even though the issues hampered measurements on critical samples as well. We expect to retrieve further data from the XRF maps in the upcoming days in order to determine whether the observed abundance of Se and Te in the silicate Earth should be explained through core equilibration processes or if they were delivered in a later stage. Due to technical issues on the beamline, we could not perform the measurements on all our samples and additional beamtime will be requested to finalize this study.

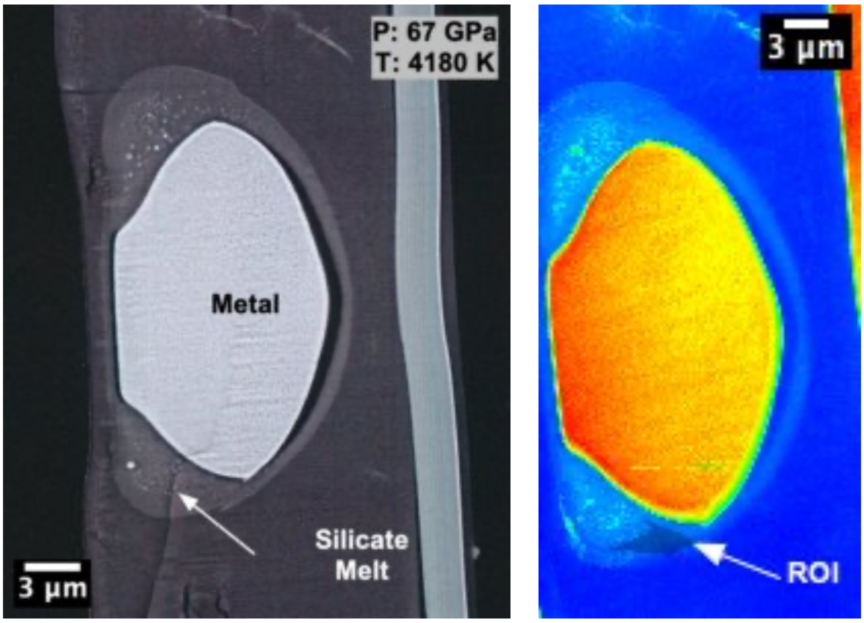

Figure 1: On the left, the backscattered image of one of the samples synthesised using LH-DAC experiments at 67 GPa and 4180 K where metal and silicate liquids are chemically equilibrated. On the right, the nano-XRF map collected on the silicate melt (lighter blue) will allow quantification of the partitioning of Se and Te during accretion and core formation of the Earth.

References:

- Righter, K., Pando, K., Marin, N., Ross, D. K., Righter, M., Danielson, L., et al. (2017). Volatile element signatures in the mantles of Earth, Moon, and Mars: Core formation fingerprints from Bi, Cd, In, and Sn. Meteoritics & Planetary Sciences.
- Solé, V. A., Papillon, E., Cotte, M., Walter, P., & Susini, J. (2007). A multiplatform code for the analysis of energy-dispersive X-ray fluorescence spectra. Spectrochimica Acta Part B: Atomic Spectroscopy, 62(1), 63-68.
- Wood, B. J., Walter, M. J., & Wade, J. (2006). Accretion of the Earth and segregation of its core. Nature.