**E**UROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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 via the User Portal:

*https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do*

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

Reports can 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>Study of the crystallization forces during peridotite serpentinization and carbonation. | **Experiment number**:<br>ES-277 |
|---|---|---|
| **Beamline**:<br>19 | **Date of experiment**:<br>from:13 apr. 2015 8:00AM to: 14 apr. 2015 8:00AM<br>from: 08 jul. 2015 8:00AM to: 09 jul. 2015 8:00AM | **Date of report**:<br>September 9, 2015 |
| **Shifts:**<br>**3 x 2** | **Local contact(s)**: Alexander Rack (13-14 Apr. 2015) & Elodie Boller (8-9 Jul. 2015) | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

- 13-14 Avr. 2015: **M. Godard** (Géosciences Montpellier - GM)**, O. Rodriguez** (VOXAYA)**, S. Escario** (GM), **C. Ferrando** (GM)
- 8-9 Juil. 2015: **M. Godard** (GM)**, O. Rodriguez** (VOXAYA)**, S. Escario** (GM), **P. Smal** (GM)

## Report:

The objective of experiment ES-277 was to perform high-quality multi-resolution 3D observations of hydrothermally altered olivine-rich rocks in order to (a) map the redistribution of mineral phases resulting from water-rock interactions and (b) detect the development of cracks and/or evidences of stress-induced alteration. The proposed strategy was to analyse a series of olivine samples before and after hydrothermal flow-through experiments using a multi-scale approach: the samples were to be analysed at 2 high resolution setups (1.1µm and 0.18 µm) to track the onset of cracking resulting from water-rock reactions at mineral-mineral and water-mineral interfaces. In order to realize the HP-HT hydrothermal experiments, samples were to be enclosed in a thin titanium body (with low absorption for X-ray) specifically designed for the ESRF experiment. 5 shifts were requested to image (a) the unreacted samples (phase 1, 1 shift) and (b) the reacted samples using 2 different resolutions (phase 2, 4 shifts). The project was attributed 6 shifts : twice 3 shifts at a 3 month interval (April & July).

Upon approval of the project, we launched the fabrication of the Ti-jackets specifically designed for the ESRF experiments and contacted the ESRF local contact team to assess precisely the experiment. We rapidly identified a major yet unforseen impediment : a minimum thickness is needed to manufacture Ti-jackets and this thickness is too thick to allow <0.2µm resolution imaging of the olivine samples altered during the hydrothermal experiments. Multiresolution imaging of experimental samples was therefore precluded on these samples. With the help of the ESRF local contact team, we modified our strategy in order to reach our objectives. We divided the experiments as 2 sets of analyses using the following setups:

- Analyses of olivine aggregates in Ti-tubes before and after hydrothermal experiments (resolution 0.65µm Beam 55 KeV): 6 samples were prepared, 4 were analysed before experiments (only 3 in april and 1 in july) and 2 after experiments (july). The data are still being processed but analyses of snapshots show clear indications of dissolution and precipitation (e.g., Fig. 1).
- Multiresolution analyses of variously serpentinized and carbonated peridotites from the Oman ophiolite (Beam 19 KeV): several minicores (Length 5 mm – Diameter 1.7 mm) were analysed with a 0.65µm resolution (8 in april and 9 in july) and a subset of 10 were analysed with a 0.16µm resolution (5 in april and 5 in july) These tests showed the feasibility of the approach and its usefulness to address our scientific objectives (e.g., Fig. 2).

The preliminary analyses (data of the second phase received only a few days ago) of the dataset indicate that this approach is promising, but we can see now that it could be improved to better trace the studied process both in term of experimental procedure (in Montpellier) and image acquisition (ESRF). For instance we encountered some problems of registration for the handling and comparison of multi-resolution images and therefore for quantifying the physical parameters controling the reactivity of the rocks. According to our ESRF contact, a sequential multiresolution approach thus appears more adapted and we hope to implement this technique in a continuation project. Also, only 2 experimental samples were analysed and we must now finalize our research experimental program which requires exploring more samples to tackle the dependence of the mass transfers to the experimental conditions and measure the processes variability.

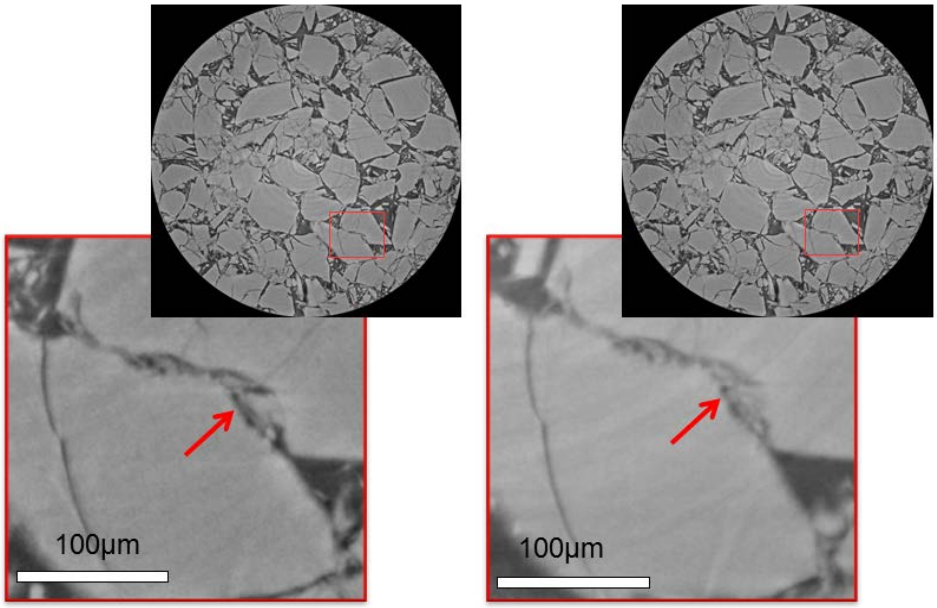


*Fig. 1: Olivine sample SC2_4 before (Left. Data April 2015 Slice 1838) and after (Right. Data July 2015 Slice 1495) reactive percolation experiments. Red arrow indicates zone of precipitation of secondary minerals in vein. Image resolution : 0.65 um. Beam 55 KeV. Image analyses in progress.*

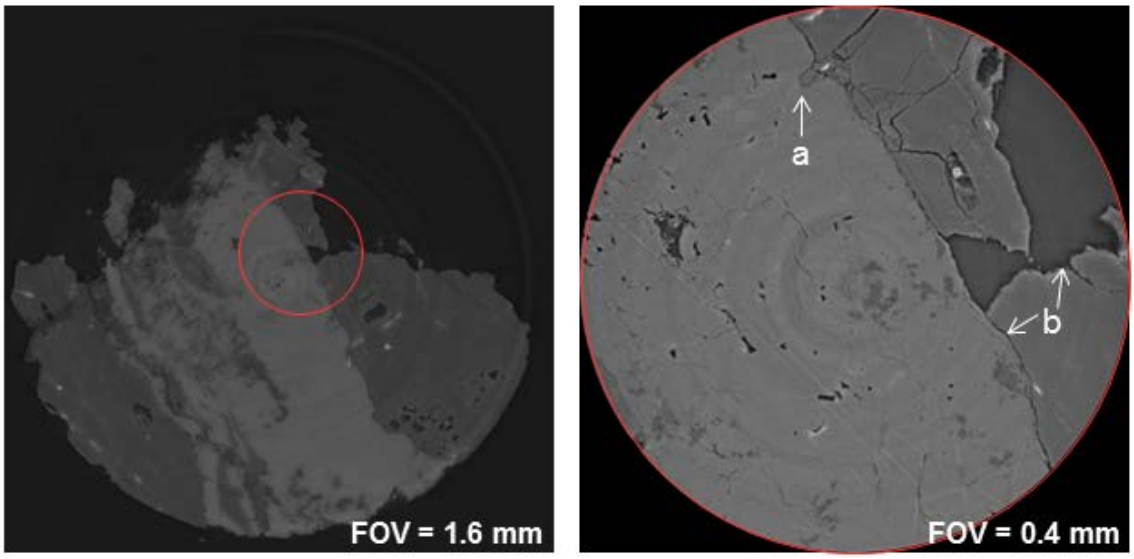


*Fig. 2: Harzburgite 15OS46 (Wadi Dima - Oman ophiolite) imaged with resolution of 0.65 um (Left) and zoom in (red circle) at resolution 0.16 um (Right). Arrows indicate (a) reactive dissolution-precipitation instability (fingering) between carbonate (light grey) & serpentine (grey) & (b )development of cracks at pore-serpentine-carbonate interfaces. Both features revealed by HR microtomography. Data July 2015 Beam 19KeV. Image analyses in progress.*