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 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:

Investigation of thermal conductivity of iron-containing Earth’s lower mantle minerals: iron magnesium aluminium silicate perovskite and ferropericlase

Experiment number:

ES-479

Beamline: ID18	**Date of experiment**: from: 24 Jan 2017 to: 31 Jan 2017	**Date of report**: 10/09/2020
Shifts: 18	**Local contact(s)**: Valerio Cerantola	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Iron- and aluminum-bearing magnesium silicate perovskite (Mg,Fe,Si,Al)O_3 is likely the main component (75 vol. %) of the Earth’s lower mantle and ferropericlase (Fe, Mg)O is the second abundant phase (20 vol. %). Thermal conductivity of these minerals is a fundamental physical parameter in controlling the heat transfer in the Earth’s interior. However their thermal conductivity has long been one of the properties most unconstrained at extreme pressure and temperature conditions due to significant experimental difficulty. Some of the main difficulties of thermal conductivity measurements is the accurate temperature distribution estimation as well as the estimation of the thickness of the sample since for thin samples presicion errors contribute significantly to the calculations.

We used the previously implemented laser heating system installed at the Nuclear Resonance Beamline[1], and for the purposes of the experiment, we partially utilized a newly developed laser heating system[2,3] that allows more precise heating and temperature estimation. We conducted measurements on ferropericlase ($Fe_{0.25}Mg_{0.75}O$) samples loaded in Ne inside a DAC, however, we could not produce any data that would be useful for the calculation of the thermal conductivity of the sample. The X-ray beam focal size of the SMS set-up at ID 18 (H x V = 13 x 13 μm^2) was prohibiting for the temperature accuracy required for such measurements. Although the focal size of the beam was technically smaller than the laser spot, the probing area was comparable in size with the heated sample area (Figure 1). This did not allow a fully controlled temperature spatial distribution due to the probing of non-heated sample at the tails of the beam that was introducing uncertainties to the temperature estimation, both in the spectroradiometric measurements as well as the Moessbauer absorption (broadening of the absorption lines).

We continued with the testing of a time-resolved experimental setup that further improved our know-how into the development of a fully time-resolved SMS scheme for future experiments[1,4]. The allocated beamtime provided us with a clear idea of what needs to be improved to successfully achieve the goals of the proposal.

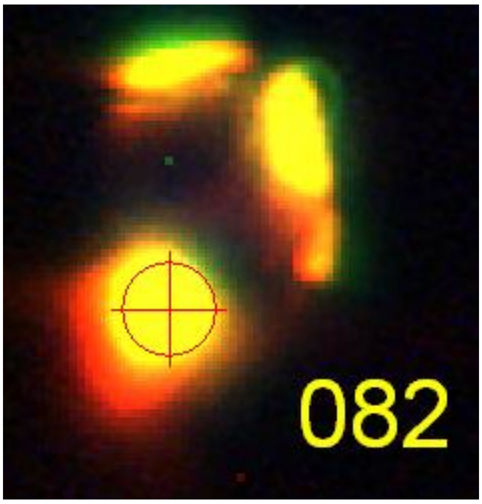

Figure 1. Microscope image of a laser heated sample inside the Diamond Anvil Cell. The red crosshair is calibrated to viualize the position of the beam as well as approximatly the focal size at FWHM (13 μm x 13 μm). The laser heated area is glowing yellow/orange showing the comparable size of the beam and the laser focus. The bright areas not under the crosshair are due to the backlight illumination visible through the transparent Ne pressure medium.

References:

1. Kupenko, I., Dubrovinsky, L., Dubrovinskaia, N., McCammon, C., Glazyrin, K., Bykova, E., Ballaran, T. B., Sinmyo, R., Chumakov, A. I., Potapkin, V., Kantor, A., Rüffer, R., Hanfland, M., Crichton, W., & Merlini, M. (2012). Portable double-sided laser-heating system for Mössbauer spectroscopy and X-ray diffraction experiments at synchrotron facilities with diamond anvil cells. *Review of Scientific Instruments* **83**(12), 124501. https://doi.org/10.1063/1.4772458
2. Kupenko, I., Strohm, C., McCammon, C., Cerantola, V., Glazyrin, K., Petitgirard, S., Vasiukov, D., Aprilis, G., Chumakov, A. I., Rüffer, R., & Dubrovinsky, L. (2015). Time differentiated nuclear resonance spectroscopy coupled with pulsed laser heating in diamond anvil cells. *Review of Scientific Instruments* **86**(11), 114501. https://doi.org/10.1063/1.4935304
3. Aprilis, G., Strohm, C., Kupenko, I., Linhardt, S., Laskin, A., Vasiukov, D. M., Cerantola, V., Koemets, E. G., McCammon, C., Kurnosov, A., Chumakov, A. I., Rüffer, R., Dubrovinskaia, N., & Dubrovinsky, L. (2017). Portable double-sided pulsed laser heating system for time-resolved geoscience and materials science applications. *Review of Scientific Instruments* **88**(8). https://doi.org/10.1063/1.4998985
4. Strohm, C., Aprilis, G., Kupenko, I., Vasiukov, D., Cerantola, V., Chumakov, A. I., Rüffer, R., McCammon, C., and Dubrovinsky, L. (2020). Fully time-resolved synchrotron Mössbauer spectroscopy for pulsed laser heating experiments in diamond anvil cell. *Under Revision at Journal of Synchrotron Radiation.*