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:** Looking at the mantle transition zone through diamond inclusions	**Experiment number**:
Beamline:	**Date of experiment**: from: 26.09.2016 to: 01.10.2016	**Date of report**:
Shifts:	**Local contact(s)**: Valerio Cerantola	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Kiseeva Ekaterina (University of Oxford)

Vasiukov Denis (Universitaet Bayreuth)

Report:

During this experiment, the PI and collaborators with the help of beam scientists Valerio Cerantola and Alexander Chumakov used synchrotron Mössbauer source spectroscopy to analyse a set of 13 diamond inclusions from the Jagersfontein Mine in South Africa.

These inclusions ranged from 50 to 150 microns in size and derived from subcratonic depths, roughly between 250 and 500 km. These samples were unique in that most of diamond inclusions are coming from depths <250 km. Additionally, due to the small sample size, this was a pioneering study that could not be conducted using any other facility in the world.

As a result, the garnets contained higher percentages of the most oxidised form of iron the deeper they formed in the mantle. Garnets from 500 kilometres, for example, had twice as much oxidised iron as garnets from the shallow mantle. One of the reasons for that could be in oxidised material brought into the deep mantle by subduction. Carbon compounds in fluids or melted rock may be responsible for this surprisingly high level of oxidation deep in the mantle. It is possible to consider the reaction between CO_2 and Fe^{2+}, resulting in the oxidation of Fe^{2+} to Fe^{3+} and reduction of carbon to diamond.

The results of this experiment were published in Nature Geoscience (Kiseeva et al., 2018), and also featured in the ESRF Highlights in 2018, with an article entitled “Oxidised garnets from the Earth’s transition zone”.

Below is the abstract of the publication and a figure outlining data produced at ERSF.

Oxidized iron in garnets from the mantle transition zone. The oxidation state of iron in Earth’s mantle is well known to depths of approximately 200 km, but has not been characterized in samples from the lowermost upper mantle (200–410 km depth) or the transition zone (410–660 km depth). Natural samples from the deep (>200 km) mantle are extremely rare, and are usually only found as inclusions in diamonds. Here we use synchrotron Mössbauer source spectroscopy complemented by single-crystal X-ray diffraction to measure the oxidation state of Fe in inclusions of ultra-high pressure majoritic garnet in diamond. The garnets show a pronounced increase in oxidation state with depth, with $Fe^{3+}/(Fe^{3+}+ Fe^{2+})$ increasing from 0.08 at

approximately 240 km depth to 0.30 at approximately 500 km depth. The latter majorites, which come from pyroxenitic bulk compositions, are twice as rich in Fe^{3+} as the most oxidized garnets from the shallow mantle. Corresponding oxygen fugacities are above the upper stability limit of Fe metal. This implies that the increase in oxidation state is unconnected to disproportionation of Fe^{2+} to Fe^{3+} plus Fe^{0}. Instead, the Fe^{3+} increase with depth is consistent with the hypothesis that carbonated fluids or melts are the oxidizing agents responsible for the high Fe^{3+} contents of the inclusions.

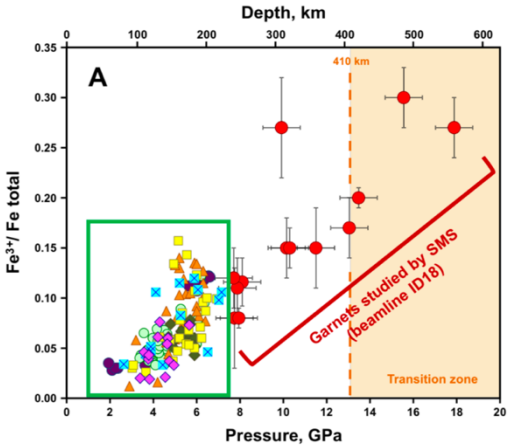

Figure 1. A. Ferric iron contents of high-pressure eclogitic garnets compared to lithospheric (<200 km) garnets from peridotite xenoliths. Data produced at ERSF are in red circles.

The PI thanks the SMS team for the amazing opportunity to conduct this research.