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.

	Experiment title:	Experiment number:
	Oxidation state of the mantle through eclogite xenoliths	
Beamline:	**Date of experiment**: from: 13.06.2017 to: 17.06.2017	**Date of report**:
Shifts:	**Local contact(s)**: Valerio Cerantola	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Kiseeva Ekaterina (University of Oxford)

Wood Bernard (University of Oxford)

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 8 mantle xenoliths and ~10 cratonic inclusions in diamonds from varies locations worldwide.

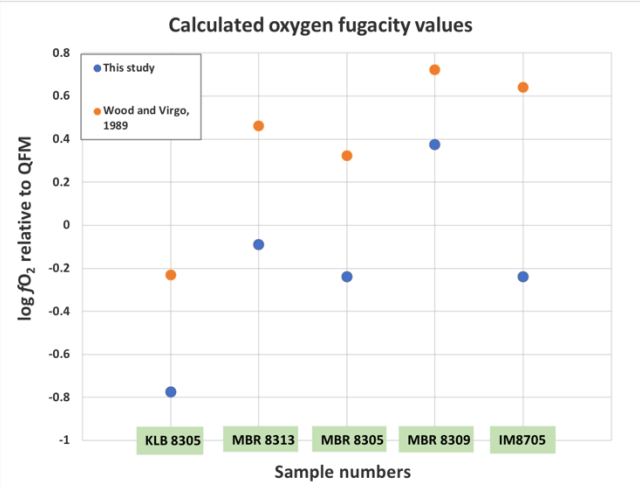
Mantle xenoliths comprised of 5 low-pressure spinel peridotites containing individual grains of spinel, orthopyroxene, clinopyroxene and olivine and 3 eclogitic samples containing individual grains of garnets and clinopyroxenes. Additionally, we analysed ~10 garnet inclusions in diamonds from Siberia and South Africa.

These data were subdivided into two studies: (1) study of mantle xenoliths and reproducibility of classical Mossbauer data and (2) study of eclogitic inclusions in diamond of cratonic origin.

This study (1) is still in progress with preliminary results having been reported at Goldschmidt 2019 in Barcelona. The conference abstract and figures are shown below.

Ferric ferrous ratios in mantle xenoliths by Synchrotron Mössbauer Source Spectroscopy.

Synchrotron Mössbauer Source (SMS) spectroscopy (ESRF, Grenoble, France) has high spatial resolution (~20 microns) and has been successfully applied to measuring Fe^{3+} concentrations in diamond inclusions [1,2]. Over the last few decades a number of studies have been conducted on individual minerals from mantle xenoliths in order to determine the oxidation state of the upper mantle. These studies were conducted using ≥50 mg of handpicked grains as opposed to individual crystals. In this study, we applied SMS to measure ferric iron contents of individual spinels, orthopyroxenes, clinopyroxenes and garnets from 5 spinel peridotite xenoliths and 1 pyroxenite and 2 eclogite xenoliths. Spinel xenoliths derive from Kilbourne hole, Mont Briançon and Ichinomegata. Spinels from these xenoliths were previously analysed by Mössbauer spectroscopy on bulk separates. Eclogite xenoliths (UAS 1055, UAS 1525) and pyroxenite xenolith (UAS 510) were obtained from Udachnaya kimberlite pipe in Siberia. In spinel peridotites measured ratios range between 0.04- 0.14 Fe^{3+}/Fetot for Opx, 0.14-0.19 Fe^{3+}/Fe_{tot} for Cpx and between 0.15-0.23 for Spl. These values are broadly in agreement with previous measurements [3]. In eclogites and pyroxenite, the ratios range between 0.05-0.16 for garnet and 0.07-0.17 for Cpx, showing $D^{Grt/Cpx}$ for Fe^{3+} of 0.8-1.9.



Oxygen fugacities derived from the spinel-olivine-orthopyroxene oxybarometer are consistent with previous results for the continental lithosphere fO_2 of between -1 and +1 log units relative to the FMQ buffer. Nevertheless, we observe small differences between our results on individual grains and previous data on bulk separates.

Figure 1. Calculated oxygen fugacities for the same set of samples analysed by classical Mossbauer and SMS. Data from SMS is in blue circles.

In conclusion:

- We observed approximately 20% difference in Fe^{3+}/Fe_{tot} content of spinels analysed by SMS and classical Mossbauer on grain separates with SMS having lower values. This may be related to matrix correction applied at SMS or to single grain vs multiple grain analysis.
- Accounting for the ferric iron in the orthopyroxene in equilibrium with spinel, this difference in Fe^{3+}/Fe_{tot} values may translate into 0.5-0.8 log units of fO2.
- Given the uncertainties, even with the specified difference in fO2s, the two methods are in fairly good agreement.

The study (2) is also in progress. At the moment, it hasn't been independently reported at any of the conferences, however, the data obtained at ESRF will be combined with other petrological or geochemical studies prior to publication, due to the scarcity of data points. More inclusions in diamonds of eclogitic paragenesis and eclogitic xenoliths are required to be analysed for an individual publication.

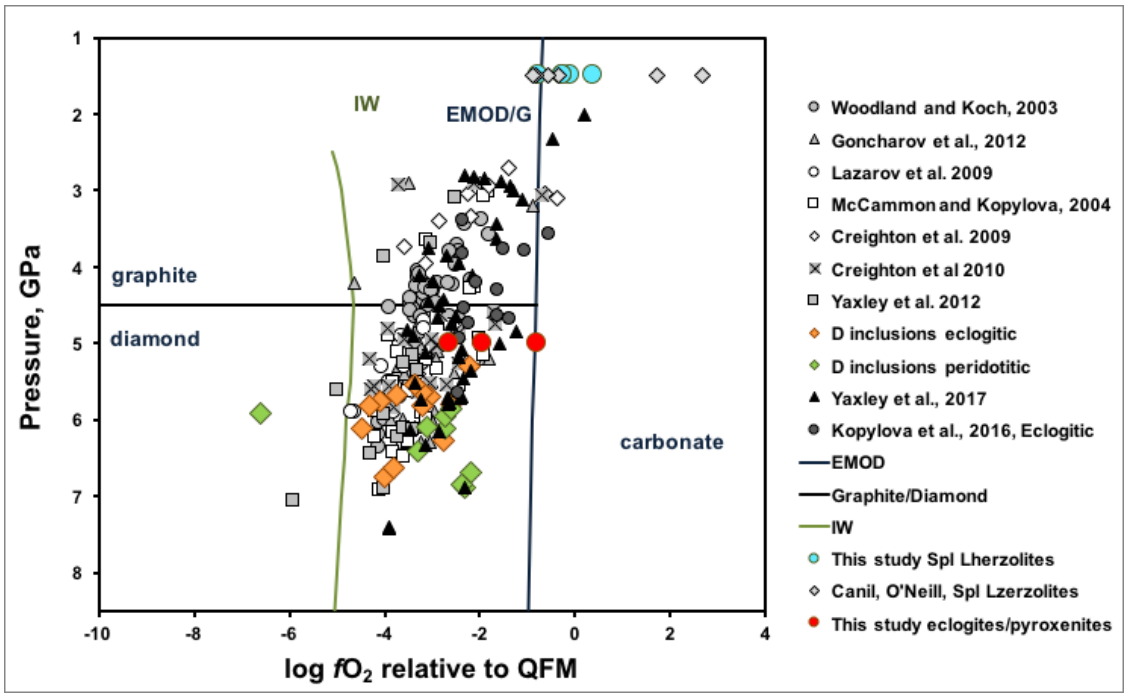


Figure 2. A compilation of calculated oxygen fugacities for mantle xenoliths and inclusions in diamonds. Inclusions in diamonds analysed by SMS are in orange and green diamonds. Mantle xenoliths analysed by ESRF are in light blue and red circles

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