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:** **Mineralogy, phase transitions and mineral densities for a subsolidus pyrolite mantle between 15 and 21 GPa and 1300 and 1600˚C**	**Experiment number**: ES1361
Beamline:	**Date of experiment**: from: 26/09/2023 to: 02/10/2023	**Date of report**: 19/12/2023
Shifts:	**Local contact(s)**: Wilson Crichton	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): Rondi Davies (Queensborough Community College) Ekaterina Kiseeva (University of Oxford) Anja Rosenthal (ESRF)		

Report:
The goal of our work at ID06 was to conduct a high-pressure multi-anvil experiment using a natural fertile mantle starting material (KLB-1) to recreate the pressure-temperature conditions of the mantle and transition zone at sub-solidus conditions (16-21 GPa and 1300-1600˚C) while making simultaneous X-ray diffraction and density measurements to determine the phases present, their densities, and constrain the P-T conditions of phase transitions.

During 18 shifts of beamtime and with the excellent support of the ESRF team at ID06 (Wilson Crichton and Anja Rosenthal), four high-pressure multi-anvil experiments using natural and synthetic starting materials were successfully conducted. Each experiment was pressurized, run at temperature for about six hours, and depressurized over a span of approximately 30 hours. Experiments were monitored during the run-time to observe phase growth and changes to phase stabilities with changes to pressure and temperature. X-ray data collected over the course of the experiment was carefully documented and described with the assistance of Dr. Crichton.

The starting materials, pressure-temperature conditions at quench, and minerals produced are listed in Table 1.

Table 1.

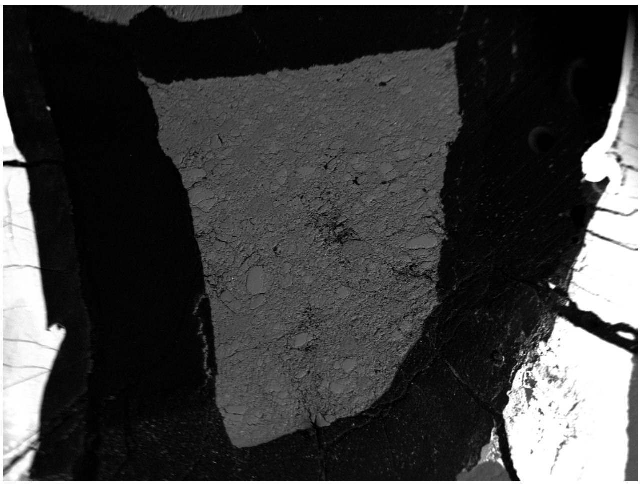
Experiment	Starting materials	Pressure (GPa), Temperature (K) at quench	Minerals synthesized
Run 1	KLB-1 natural	10 GPa, 1600K	Diopside, clinoenstatite, garnet, olivine
Run 2	KLB-1 natural	15 GPa, 1700K	Diopside, clinoenstatite, garnet, wadsleyite

Run 3	KLB-1 natural	15.4 GPa, 1700-1850K	Diopside, clinoenstatite, garnet, wadsleyite
Run 4	KLB-1 synthetic	17 GPa, 2000	Majorite, wadsleyite

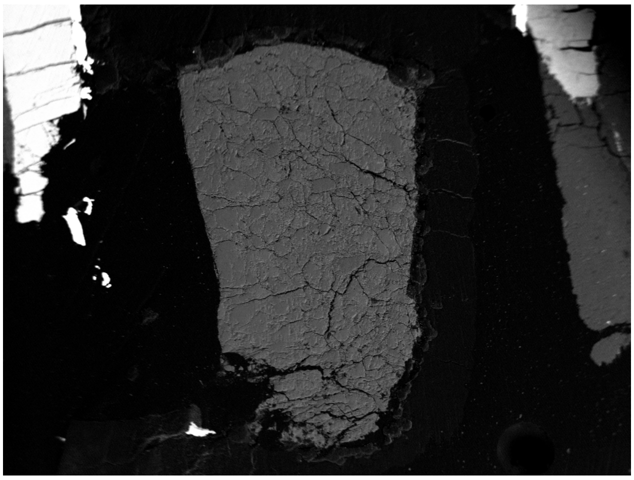
The four run products were taken to the American Museum of Natural History in New York for mounting, polishing, and chemical analysis using the electron microprobe (EMPA). The mineral phases present have large, well-formed crystals (Fig. 1a-d; SEM images) and EMPA confirms homogenous and well-equilibrated compositions. However, garnet grains have not equilibrated well, likely because of heating times were too short to allow for this. Run 4, using the synthetic starting material, resulted in small grain sizes and differences in composition to the natural counterparts. This experiment will require further investigation to understand why differences exist between the natural and synthetic starting compositions.

Figure 1. SEM images from EMPA showing all experiments withing a graphite capsule.

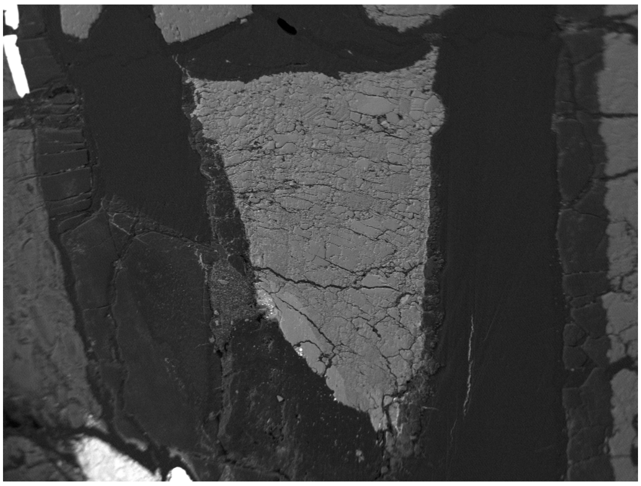
a. Run 1



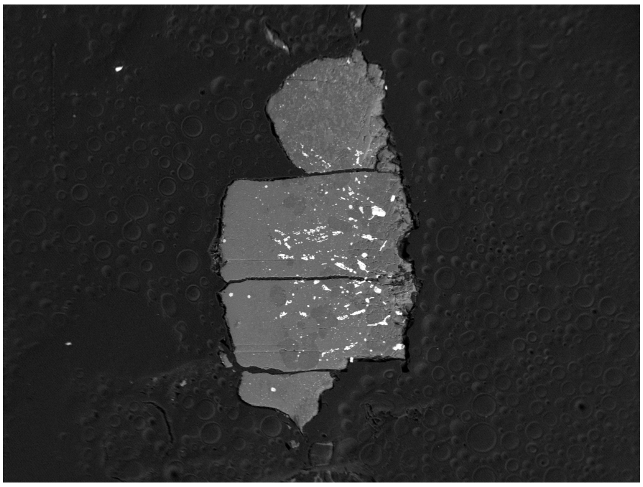
1b. Run 2



1c. Run 3



1b. Run 4



The findings from these experiments support previous unpublished work we have done using a natural KLB-1 starting mixture. This work is currently being written up for publication and we will incorporate the study conducted at ESRF into this work. We continue to discuss the results with ESRF scientists Crichton and Rosenthal to help interpret the results and develop ideas for additional publication and collaboration related to this work. The data will be used to re-evaluate the phase diagram for the transition zone, particularly where clinoenstatite transforms to a garnet structure, with implications for seismological observations of the mantle, thermodynamic models for phase equilibria, and the paragenetic relationships and depths of origin for sublithospheric diamonds.

The PI is deeply grateful to ESRF for this amazing opportunity and in particular to the ID06 for their scientific and technical support and collaboration.