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:** **Acoustic velocites of $(Mg_xFe_{1-x})_2SiO_4$ ringwoodite**	**Experiment number**: ES-1212
Beamline: ID06-LVP	**Date of experiment**: from: 08/11/2022 to: 14/11/2022	**Date of report**: 05/03/2023
Shifts: 18	**Local contact(s)**: Dr D. Druzhbin & Dr. W. Crichton	*Received at ESRF:*

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Report:

ES-1212 was performed in Nov, 2022. The polycrystalline wadsleyite (Wads) samples with different Fe concentrations (X_{Fe}=0, 0.1, 0.2) and one ringwoodite (Rw) sample (Fo90) were successfully pre-synthesized at UCL using multi-anvil press at 14-20 GPa and 1473-1673 K and double polished as the starting materials for the ultrasonic measurements at ESRF. During this beam time, seven multi-anvil runs were conducted using the 10/4 assembly with a newly developed X-ray transparent TiC-MgO composite heater. The composition of samples and the maximum *PT* conditions for each run are listed in Table 1. In each run, we first compressed the sample to the targeted pressure (~10-20 GPa) depending on its composition, and then heated the sample to 800-1000 °C to release the stress in the sample during compression. After that, the temperature was decreased and increased in cycles and X-ray diffraction (XRD), imaging and MHz frequency ultrasonic measurements were made during the heating loops to determine the structure, length and acoustic velocities. The temperature was monitored by the thermocouple and pressure was determined using the peaks of NaCl by real-time XRD to make our best not to go out of the Wads stability field. The new diffraction free gaskets were used through the X-ray windows in all experiments.

Table 1: summary of experimental runs performed in ES-1135.

Experiments	Starting material	Max *PT* conditions	Notes
Run_1	Mg_2SiO_4 Wads	10 GPa, 723 K	-TC works well -blow out during compression at 723 K during the first heating cycle
Run_2	$(Mg_{0.9}Fe_{0.1})_2SiO_4$ Wads	15.51 GPa, 540 W, ~1453 K	-TC breaks during compression at 873 K in the first heating cycle -Good ultrasonics signal for both P and S wave

			- US and XRD data were collected for three heating cycles between room temperature and 1453 K at pressures between 10.1 and 15.5 GPa to stay in the stability field of Fo90 Wads and also during decompression to room pressure at ambient temperature
Run_3	Mg_2SiO_4 Wads	16.44 GPa, 504 W (1630 K)	-TC works well -Only P wave ultrasonic signal was observed, high noise - US and XRD data were collected for three heating cycles between room temperature and 1630 K at pressures between 11.43 and 16.44 GPa to stay in the stability field of Fo100 Wads and also during decompression to room pressure at ambient temperature
Run_4	$(Mg_{0.9}Fe_{0.1})_2SiO_4$ Rw	20.1 GPa, 570 W (1636 K)	-TC works well - Good ultrasonics signal for both P and S wave -US and XRD data were collected for three heating cycles between room temperature and 1632 K at pressures between 16.2 and 20.0 GPa -US and XRD data were also collected during decompression at room temperature and heating up to 873 K at some pressure points to collect high T data -The sample back transformed to olivine at ~ 873 K and ~ 6 GPa during decompression
Run_5	Mg_2SiO_4 Wads		-blow out during compression at 86 bar
Run_6	Mg_2SiO_4 Wads	14.41 GPa, 270 W (937 K)	-TC works well -ultrasonic signal not so good from ambient condition, high noise -blow out at 14.4 GPa and 937 K during the first heating cycle
Run_7	$(Mg_{0.8}Fe_{0.2})_2SiO_4$ Wads	16.97 GPa, 620 W (~1623 K)	-TC works but flipped -Peaks of NaCl lost at high pressure and high temperature due to intrusion of thermocouple into soft backing plate -sample transformed to olivine during heating and not able to make it transform back due to the narrow stability of Fo80 Wads (<1 GPa at 1400 K) and inability to accurately determine pressure and temperature

The TiC heater worked well up to the highest temperature (1630 K) in our experiment and the sample was observed to be in the middle of the X-ray window after compression, which enables the measurement of sample length through X-ray imaging. The ultrasonic signal for both P and S wave are quite good for run-2 and run-4 during the whole experiments and were successfully measured up to 15.5 GPa, 1453 K and 20.1 GPa, 1636 K respectively. In run-3, only P wave signal was observed and measured up to 16.4 GPa, 1630 K and we were not able to identify the S wave signal due to the high noise-signal ratio. This poor signal-noise ratio was later found to be due to either (1) the negative thermal expansion of soldering material which partly pulled the transducer off during heating or (2)

the poor isolation of the earth wire with the signal wire which are quite close to each other. The XRD pattern were collected at the same time through several heating loops and therefore by refinement and fitting, we can get high pressure high temperature equation of state parameters of the corresponding phases. Compared with the traditional pyrophyllite gasket, the diffraction free amorphous gasket in the X-ray window would increase the chance of blow out, which lead to the failure of run-1, 5 and 6. In run-7, the Fo80 Wads was transformed to olivine upon heating due to it's narrow stability field (< 1 GPa at 1400 K).

Experiments all produced large volumes of XRD and ultrasonic data (typically ~ 8000 diffraction patterns per experiment). To process such large volume of data, new analyses procedures have been developed. An example of typical ultrasonic spectrum at 19.9 GPa and 1630 K was shown in Fig. 1 with (a) raw spectrum and (b) the cross-correlation of P wave signal.

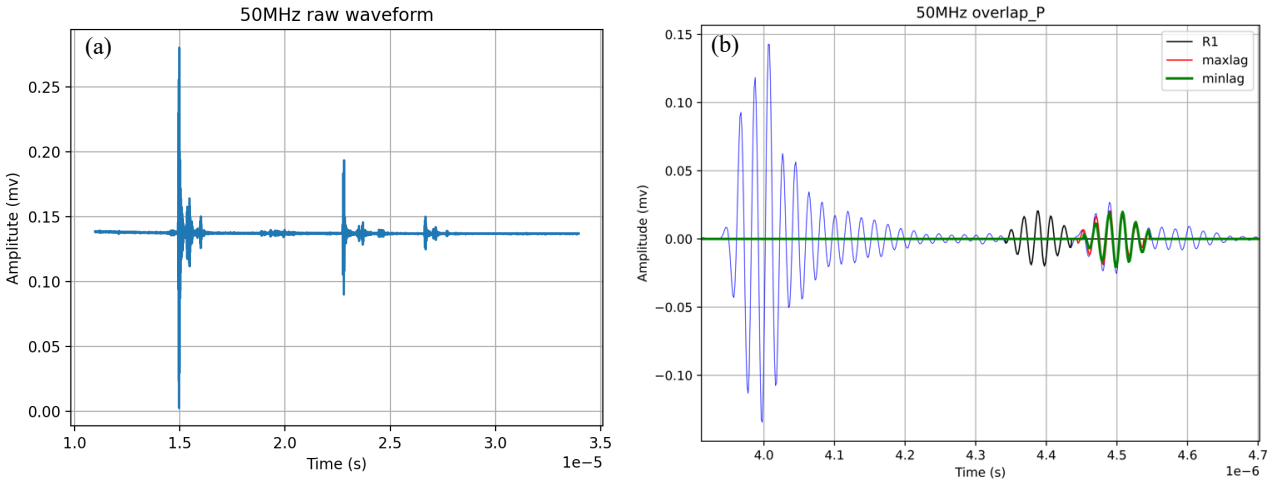

Figure 1: exemplar ultrasonic data signal. (a) raw data; (b) cross-correlation of P wave signal.

Due to complexity of experimental setup, we would like to request more beam time to continue and complete our study in the next step. The goal is to measure both P and S wave of Fo100 Wads simultaneously and measure the Fo90 Wads with thermocouple working to explore the strong dependence of elastic properties on temperatures. The thermocouple works for most of our experiments. For the ultrasonic measurement, we find a way of connecting signal wire and transducer without soldering so the transducer would have less chance to be pulled off. Also, we have learned to isolate the Earth wire and the signal wire very well. Despite the narrow stability field of Fo100 and Fo90 Wads, we were able to keep it in the range where Wads is stable during the heating path and measurement. So we believe that we will find the suitable way to measure the acoustic velocities of Wads at transition zone conditions. If having the chance, we would also try to measure Fo80 Wads which has a larger stability pressure range at higher temperatures (> 1600 K).