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ESRF

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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** ***In situ XRD study of the effect of phase transitions on plastic properties of major Earth`s mantle constituents: implications for seismic anisotropy and deep seismic earthquakes***	**Experiment number**: HS-4765
Beamline: ID11	**Date of experiment**: from: 7th Feb 2013 to: 12th Feb 2013	**Date of report**: 14th Feb.2013
Shifts: 15	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

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Report:

The series of phase transitions in the dominant upper mantle constituent $Mg_{1.9}Fe_{0.1}SiO_4$ [olivine α – phase] to its high-pressure polymorphs wadsleyite [β- phase] and ringwoodite [γ-phase] play an essential role for large scale geodynamical processes and for the physical structure of the Earth's mantle (*Ringwood and Major*, 1966; *Weidner and Wang*, 2000). The comprehension of the underlying microscopic mechanisms and micromechanical properties during this phase transitions will therefore provide an overall understanding of the convective properties of the Earth's mantle (*Nakagawa et al.*, 2009*).*The goal of the present experiment was to conduct *in situ* three dimensional-X-ray diffraction (3D-XRD) experiments, combined with resistive heated diamond anvil cell (DAC) techniques, in order to track the orientations and positions of grains inside a sample undergoing those series of phase transformations. This experiment is very challenging because temperature should be maintained at 1300 K during the whole increase of pressure (up to 30 GPa) while performing *in situ* 3D-XRD measurements.

During the allocated beam time at ID11, we carried out simultaneous high-pressure and high-temperature 3D-XRD experiments up to 22 GPa and 800 K. Hydrous Mg-end member olivine crystals (Mg_2SiO_4) with ~500 ppm H_2O (synthesized prior to the experiment in a multi-anvil press at ETH Zürich) were used as starting materials to increase the phase transformation kinetics (*e.g., Perrillat et al.*, 2013). The beamline was tuned to a 10(H) x 5(V) μm^2 focused monochromatic X-ray beam of 0.2953 Å (41.98 keV). X-ray diffraction (XRD) patterns were collected using a Frelon4M detector with an active area of 1048 x 1048 pixels at a distance of 165.33 mm from the sample. A LaB_6 standard used to obtain precise calibration parameters of the sample to detector distance, detector tilt, instrument broadening and pixel size ratios.

The prototype of resistive heated DAC, developed at ID27, along with all necessary components (power supplies, vacuum vessel, vacuum pump, cooling jacket ...) was tested and used at ID11 for the first time. Three high PT assemblies were prepared, with olivine as a sample, and platinum and gold as pressure markers. Temperature was monitored using S-type thermocouples in contact with the diamonds. Unfortunately, because of technical restrictions of the power supplies and failing of thermocouple temperature readings during the run, several samples failed and only a maximal temperature of ~850 K could be reached on the best sample of this beam time. 3D-XRD images were collected at ambient temperature and upon compression to 3 GPa to precisely determine and track the initial micromechanical properties of the sample. The sample was then heated to ~850 K and pressurized to 22 GPa in pressure steps of 3-5 GPa. During this process 3D-XRD images were collected at each PT step to monitor the micromechanical properties of the sample and the onset of the phase transformation. The onset of the α-β transformation has

been successfully observed in one of the three attempts at 17 GPa. On the best sample, 8 pressure points were collected up 22 GPa and 850 K. The complete transformation of olivine to wadsleyite could not be reached due to the sluggish transformation kinetics at this relatively low temperature (> 7 hours at 850 K) (*Perillat et al.*, 2013). Data processing is currently in progress. The collected diffraction images (Figure 1) are of excellent quality for 3D-XRD studies and indexing. They will allow extracting the micromechanical properties of single-crystals of olivine inside a bulk aggregate (crystal size distribution, orientation statistics and the location of the grain in the sample chamber upon compression and heating) using the FABLE package (e.g. *Odderschede et al.*, 2010; *Nisr et al.*, 2012). In addition, preliminary results on the transformation textures between the α-β phase transformations can be extracted from the obtained data set. The results of this experiment will therefore provide important information on the plastic deformation mechanisms and micromechanical properties of olivine at the high-PT conditions relevant for the Earth mantle. Ultimately, these results will allow placing preliminary constrains on the plausible contribution of transition textures upon deformation in the olivine polymorphs to observed seismic anisotropy in subducted slabs.

In the coming months, we will apply for additional beam time to complete the study at higher temperatures (> 1000 K) using an improved resistive heating DAC, modified with K-type thermocouples, a larger vacuum vessel, and proper power supplies. Ultimately, we are aiming to investigate the complete range of phase transformations textures and mechanisms in the olivine systems at the relevant PT conditions of the Earth's mantle (1000-1300 K and 30 GPa), including the wadsleyite to ringwoodite transformation and the breakdown of ringwoodite to perovskite ([Mg,Fe]SiO_3) and periclase ([Mg,Fe]O).

Figures:

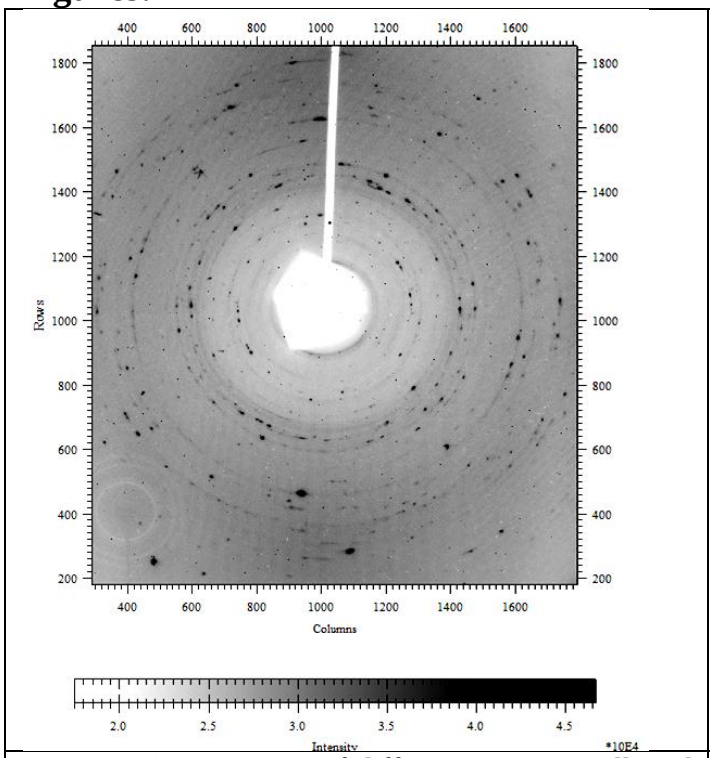

Figure 1 a) Summation of diffraction images collected over 42 ° in omega with a step size of 0.25 ° **at 2 GPa**. Refelctions of several single grains corresponding to the olivine α–phase are well seperate and intense. A priliminary indexing allowed to indentify **about 150 grains**.

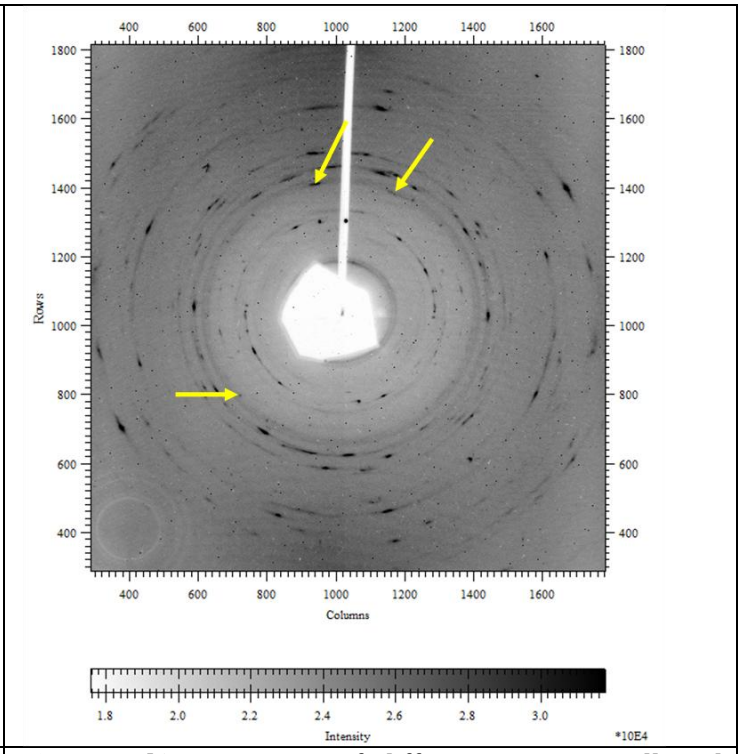

Figure 1 b) Summation of diffraction images collected over 42 ° in omega with a step size of 0.25 ° at **17 GPa and 850 K**. The diffraction images shows the texturing (non random distribution of reflections) and recrystallisation (braodening of peakes) of single grains of the olivine α – phase. A priliminary indexing allowed to indentify **about 80 grains**. New reflections and a weak new diffraction ring corresponding to wadsleyite (β-phase) are highlithed by yellow arrows.

References: **Nakagawa**, T., et al., (2009), Incorporating self-consistently calculated mineral physics into thermochemical mantle convection simulations in a 3-D spherical shell and its influence on seismic anomalies in Earth's mantle, Geochem. Geophys. Geosyst., 10, *Q03004*,. **Nisr**, C, et al., (2012), High resolution three-dimensional x-ray diffraction study of dislocations in grains of $MgGeO_3$ post-perovskite at 90 GPa, J. Geophys. Res.,117, B03201. **Oddershede**, et al., (2010) Determining grain resolved stresses in polycrystalline materials using three-dimensional X-ray diffraction, J. Appl. Cryst., 43, 539-549. **Perrillat**, J. P., et al. (2013), Mechanism and kinetics of the α–β transition in San Carlos olivine $Mg_{1.8}Fe_{0.2}SiO_4$, Geophys. Res. Let., 118, 1–10. **Ringwood**, A. E. and A. Major (1966), Some high pressure transformations in olivines and pyroxenes, J. Geophys. Res., 71(18), 4448. **Weidner**, D.J. and Y. Wang (2000) Phase transformations: implications for mantle structure, Geophys. Monogr., 117, 215-235.