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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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

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Reports can now 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.

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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;
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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:** Deformation mechanisms of hydrous magnesium silicate phase D at high pressure by X-ray radial diffraction: implications for seismic anisotropy in deep subducted slabs	**Experiment number**: HS/4059
Beamline: ID09A	**Date of experiment**: from: 22/07/2010 to: 27/07/2010	**Date of report**: 1.Sept.2010
Shifts: 15	**Local contact(s)**: Evans Shaun	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Phase D, $MgSi_2H_2O_6$ a dense hydrous magnesium silicate is possibly the ultimate water carrier down to lower mantel regions. It is stable at the uppermost lower mantle in cold subducting slabs. At those depth, phase D could account for more than 40 modal percent of water saturated peridotite [1]. This abundance and the potential of the layered structure to align in a non-hydrostatic stress field make of phase D a strong candidate to contribute to the seismic anomalies observed in deep subduction zones (shear wave splitting with SH>SV, bulk velocity reductions, deep focused earthquakes triggered by the dehydration Phase D) [2-5].
In order to interpret seismic observations in deep subduction zones, in terms of mineralogy, deformation state and the degree of hydration, precisely determined rheology data combined with elasticity data of candidate phases at relevant pressure and temperature conditions are needed to establish realistic models for this geodynamical and geochemical complex regions.

During the allocated beamtime at ESRF ID09A, high-pressure *in situ* radial x-ray diffraction experiments were conducted using panoramic diamond anvil cells up to 45 GPa to investigate the deformation mechanisms of Phase D [6]. Experiments were conducted in phase D samples synthesized at 19 GPa and 1100 °C using a multi-anvil apparatus at ETH Zurich before the beamtime. The samples were confirmed to be Phase D by single-crystal x-ray diffraction and Raman spectroscopy.
The experimental set-up at ID09A comprised a monochromatic x-ray beam tuned to 0.413857 Å (29.96 keV) focused down to $30x30\mu m^2$. X-ray diffraction patterns were collected using a Mar555 image-plate detector with a 430 x 350 mm active area located at 400 mm from the sample. Sample to detector distance, detector tilt, and pixel size ratios were calibrated using a silicon standard before the experiments. All experiments were conducted using panoramic diamond anvil cells especially designed for X-ray radial diffraction experiments. Amorphous-boron gaskets with a thickness of 50 µm and a hole of 80 µm were used as pressure chambers to avoid x-ray absorption. The phase D crystals were reduced to fine-grained powders and loaded in the experimental volume without pressure-transmitting medium to enhance plastic deformation. Chips of gold and ruby were added to the sample chamber to serve as pressure standards. Angular-dispersive X-ray diffraction measurements were conduct with typical pressure steps of 3-5 GPa from ambient conditions up to 45 GPa. At each pressure, diffraction spectra were collected alternatively in the sample and the Au standard with typical exposure times of 20 seconds. Pressure was determined from the variations of lattice parameters of gold EOS [7]. Before and after data collection the pressure was cross-checked to ensure pressure stability during measurements by ruby florescence method using a laser spectrometer installed at ID09A.

During the beamtime, Phase D samples with three different compositions, including pure Mg-Phase D, Fe-bearing and Fe-Al-bearing Phase D, were pressurized and deformed to constrain the effect of cation substitution on the deformation mechanism. Upon compression, the development of strong textures were observed from intensity variations of Debye rings and substantial variation of diffraction peak position with orientation relative to the compression direction in all samples (see. Fig. 1-3a,b).

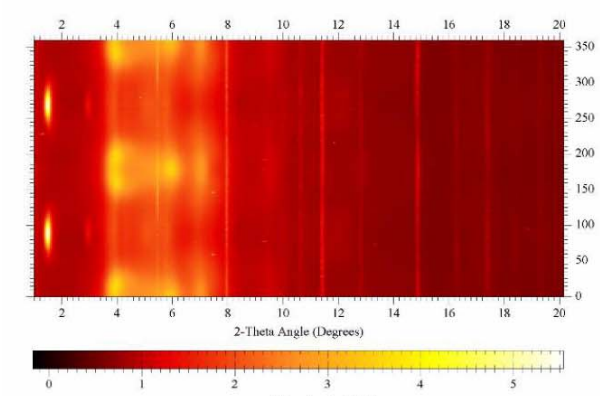

Fig. 1, 1.4GPa

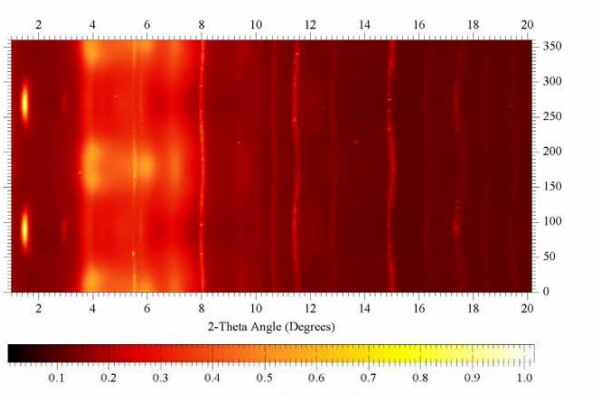

Fig. 2, 3.7GPa

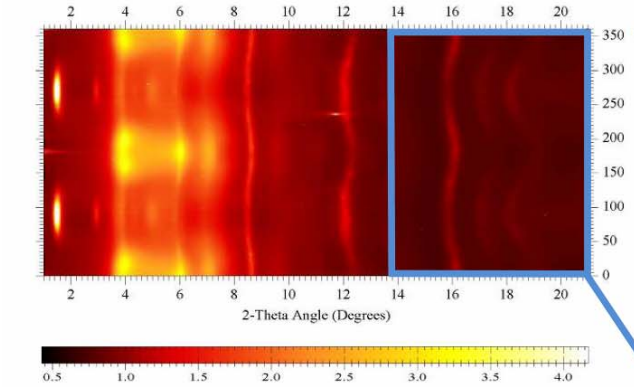

Fig. 3a, 45.3GPa

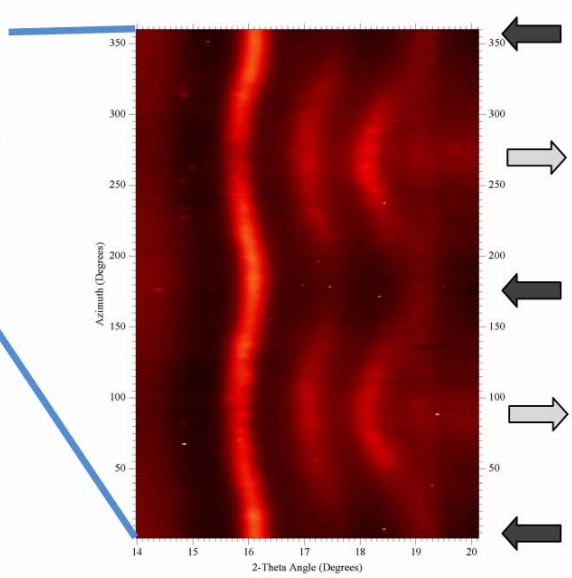

Fig. 3b, 45.3GPa

Figure 1-3a,b. Unrolled radial diffraction images of pure Magnesium Phase D at different pressures (see below each image) at room temperature collected for 20 seconds. The azimuth angle is plotted against 2Theta angle, intensity variations are given by red to yellow shading. At low pressures (Fig 1.) there is almost no observable differential stress and no texturing (straight lines = Debye ring for one lattice plane in diffraction image). With higher pressures (Fig. 2, and Fig 3a,b) the sample starts texturing (intensity variation for equal lattice planes depending on orientation to maximum and minimum stress direction, indicated by black and grey arrows) and becomes highly stressed (Fig. 3b) (variation of 2Theta position for equal lattice planes depending on orientation).

Data processing is currently in progress. All collected data are of high quality and will allow the determination of the cell parameters, texture and lattice strains using Rietveld method as implemented in the software package MAUD [6]. These data will be combined with single-crystal elastic properties for phase D to provide the first constrains on the deformation mechanism and rheological properties of Phase D at pressures relevant for the Earth mantle. These results will allow constraining better the origin of seismic anisotropy in subducted slabs and preliminary results of this study will be presented at the AGU Fall meeting 2010 in San Francisco, United States.

Given the interesting results already obtained during this first series of measurements and the probed feasability of deformation experiments in Phase D, we will request a continuation of HS-4059 to investigate the deformation mechanisms under high P-T conditions.

References:

[1] Iwamori, 2004, Earth Planet. Sci. Lett., 227, 57–71. [2] Laurence & Wysession, 2006a, Earth Planet. Sci. Lett, **241**, 962-971. [3] Laurence & Wysession, 2006b, AGU Monograph, 251-261. [4] Mainprice et al., 2007, Earth Planet. Sci. Lett, **259**, 283–296. [5] Chen & Brudzinski, 2003, Geophy. Res. Let., **30**, 1682-1686. [6] Merkel & Yagi, 2005, Rev. Sci. Instrum., **76**, 046109. [6] Lutterotti et al., 1997, J. Appl. Phys., **81**, 594. [7] Anderson et al., 1989, J. Appl. Phys., 65, 1534-15431. [8] Singh et al., 1998, J. Appl. Phys., **83**, 7567.