EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** *Does wadsleyite experience hydrolytic weakening?*	**Experiment number**: ES 637
Beamline: ID06-LVP	**Date of experiment**: from: 08/02/17 to: 12/02/17	**Date of report**:
Shifts: 12	**Local contact(s)**: Wilson Crichton	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Andrew R Thomson [1]*, John Brodholt [1], David Dobson[1] and Isra Ezad[1]*

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Report:

It has been repeatedly observed that the creep strength of nominally anhydrous minerals (NAMs) is significantly and systematically weakened in the presence of water (e.g. Karato and Jung 2003). These observations are explained by the increased concentration of lattice vacancies that accompany hydroxyl incorporation to maintain charge balance, promoting ionic diffusion and lowering mineral strength (Kohlstedt 2006). It is often assumed that the presence of 'water' generally will cause a weakening of mantle rheology and cause a significant feedback on mantle geodynamics (Crowley et al. 2011; Conrad 2013), despite the lack of experimental evidence for the majority of high-pressure mantle mineralogies.

The transition zone, between approximately 410 and 660 km depth, has the potential to be Earth's largest volatile reservoir because of the capacity for wadsleyite and ringwoodite to hold up to ~ 3 wt.% H_2O in their structure (Smyth 1987; 1994; Bercovici and Karato 2003). Wadsleyite is unlike other NAMs because hydrogen has a specific crystallographic site, attached to an underbonded oxygen (O1) in the crystal structure (Smyth 1987; 1994). It is unclear whether or not hydrolytic weakening should occur in wadsleyite at all and existing experimental data does not address this issue. Therefore, we proposed to perform deformation experiments on ID06-LVP to directly measure the creep behaviour of wadsleyite samples with water contents varying by over ~ 2 orders of magnitude. Prior to our allocated beamtime at the ESRF we successfully synthesised sintered wadsleyite samples of $(Mg_{0.9}Fe_{0.1})_2SiO_4$ composition with water contents from << 1000 ppm to > 15,000 ppm, suitable for deformation in a D-DIA assembly.

The deformation experiments proposed on ID06-LVP required conditons of 13-16 GPa and 1200-1700 K to be achieved. Whilst these conditions should be well within the capabilities of ID06, they significantly exceed pressures used previously during deformation at the ESRF. To allow for the high pressure conditions required in this and future studies we set out to develop a self-aligning 6/6 deformation setup (see report ES-466). Modifications were made to address issues encountered during experiment ES-466, and pressure tests during in-house beamtime indicated that success of the proposed experiment may be possible.

During the course of 12 shifts of beamtime we performed five attempted experiments in 6/4 (6 mm pressure media and 4 mm truncated anvils) and 5/3 cells. Experimental assemblies consisted of a sintered (Mg,Co)O cubic ressure media, with ZrO_2 thermal

insulation, a Ni furnace, MgO internal parts and Al_2O_3 pistons, very similar to cell designs of Kawazoe et al (2016). All cells used pyrophyllite gaskets of 1.0 x 1.4 mm dimensions. Sintered olivine samples of $(Mg_{0.9}Fe_{0.1})_2SiO_4$ were loaded into experiments as starting materials, which would rapidly transform to wadsleyite upon heating at suitable high-pressure conditions. Figure 1 demonstrates that pressure conditions of 14 GPa were comfortably achieved in the experiments, and the pressure-force curves compared with those for the smaller experimental assemblies employed by Kawazoe et al. (2010; 2016). The larger assemblies used here allow for the study of larger samples, allowing significantly improved stress-strain resolution.

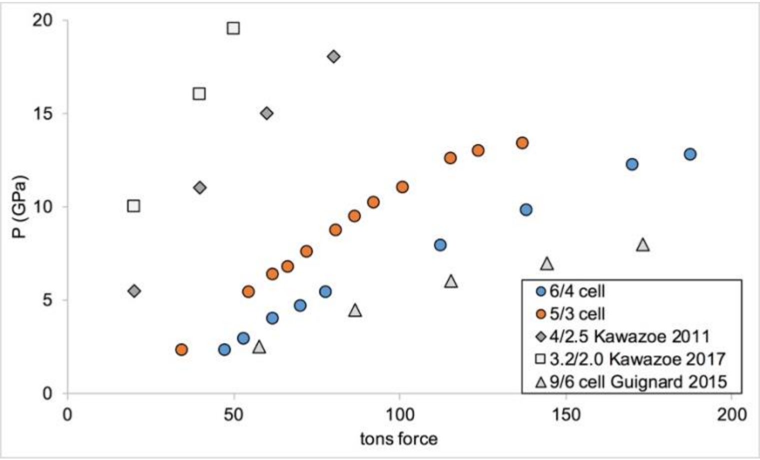

Figure 1: Ambient temperature pressure vs force curves for experiments in this study compared to those of previous studies.

In initial experiments attempted using two downstream diamond anvils, once arriving at pressure suitable diffraction was not available from the sample environment to allow for stress measurements to be performed (figure 2a). It is observed that the azimuthal diffraction is only complete at small 2theta, or limited to This issue could not have been forseen in advance without *in-situ* testing. Modifications made during beamtime to the press setup, and the use of three transparent diamond anvils (one upstream and two downstream) allowed significant improvements, which were optimised by our fith and final attempt. They allowed 180° azimuthal diffraction of the sample out to ~ 6.5 – 8° 2theta depending on the press rotation (figure 2b). This access would be suitable for stress quantification.

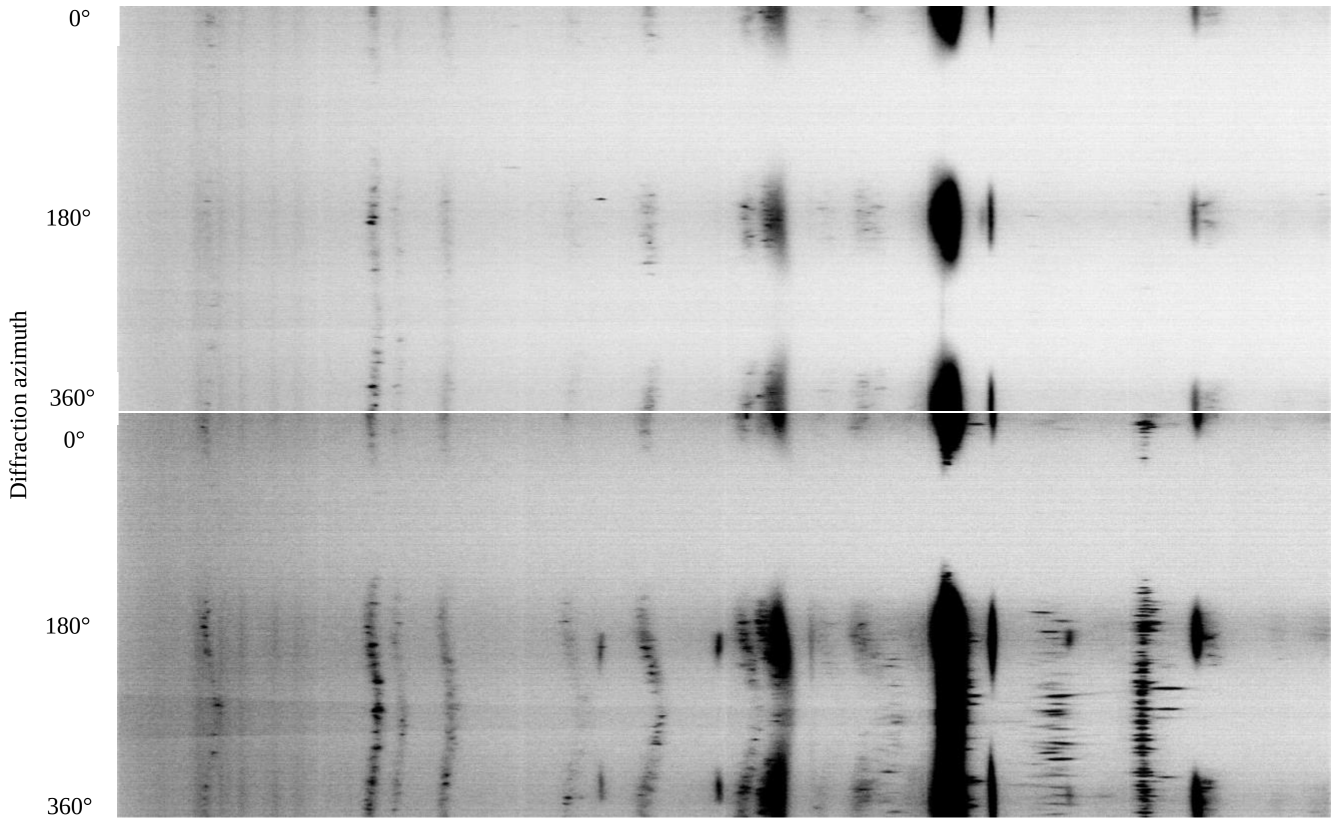

Figure 2: Recovered diffraction patterns at ~ 14 GPa in the (a) initial and (b) final attempted experiments of ES-367. The 2 theta range of plotted diffraction is from 2-8.5°.

Unfortuantely, as in many high-pressure experiments we experience blow-outs during compression in 2/5 attempted experiments. Additionally, in the final experiment which achieved suitable pressure conditions, an anvil broke during sample heating at ~ 150 W power (~ 300 °C). In an earlier experiment, sample temperatures of ~ 1000 °C (evidenced by the onset of nickel recrystallisation and sample conversion to wadsleyite) were achieved for a period of 30-45 minutes at > 14 GPa. These are record conditions for any 6-6 DIA experiment at the ESRF, and most likely outside of Japan. However, the cell blew out during conversion of olivine-wadsleyite, cutting it short before deformation could begin.

We are now extremely confident that 6-6 DIA experiments will be possible, and provide high-quality stress-strain measurements during deformation (see figure 3 for sample length image at 14 GPa in the final experiment). We propose to test various furnace assemblies in the press at ID06 both when there is no beam, or during in-house time prior to future user experiments. We are aware of the upcoming long shutdown, and have therefore re-submitted our proposal to the final call prior to this, in the hope of completing our proposed experiments.

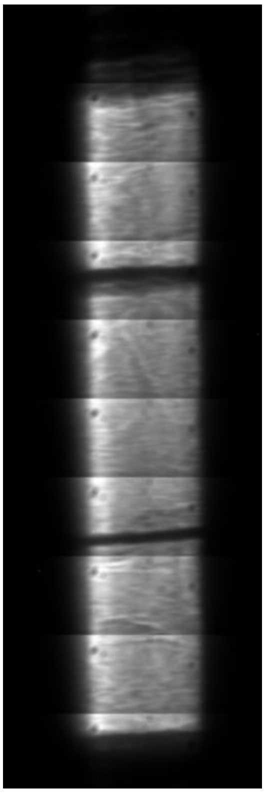

Figure 3: radiographic image of the sample at 14 GPa.

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