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.

	Experiment title: *Does wadsleyite experience hydrolytic weakening?*	Experiment number: ES 466
Beamline: ID06-LVP	**Date of experiment**: from: 19/11/16 to: 22/11/16	**Date of report**:
Shifts: 9	**Local contact(s)**: Wilson Crichton	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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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_2SiO_4 and $(Mg_{0.9}Fe_{0.1})_2SiO_4$ 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 14-16 GPa and 1200-1700 K to be achieved, which although well within the capability of the setup on

ID06, significantly exceeded pressures used previously during deformation. 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 figure 1a), similar to that employed by Nishiyama et al. (Nishiyama et al. 2008). Implementation of this setup will improve alignment and stability of the DIA setup with small assemblies, which will ultimatley allow for more routine deformation experiments at pressures of 12 – 20 GPa.

Prior to attempting the intended wadsleyite deformation experiments, we tested the alignment of the new anvil guide using Al cubes and subsequently investigated potential pressure media and gasketting. Fig 1d demonstrates the success of the new anvil guide at improving alignment. Boron epoxy, pyropphyllite and Cr-doped MgO cubes were all tested as pressure-media. However, all test experiments failed due to anvil breakage at 35 ± 5 bars press load. Inspection of the broken deformation anvils and primary rams after several failures identified significant deformation of the carbide primary rams. Figure 1b and c show that the failure of the anvils extended from the centre of the truncation as a ring fracture, expanding towards the rear anvil surface. Combined with the deformation on the primary rams this is indicative of a lack of support behind the secondary deformation anvils. This issue is a result of the primary carbide rams being too “soft”, causing them to deform significantly more than the secondary anvils. Unfortunately, this issue could not be solved during the allocated beamtime, and thus none of the planned experiments were attampted. The issue could be solved by either replacing the carbide primary rams with a harder grade, or designing harder carbide spacers that can be inserted between the existing primaries and the secondary deformation anvils. Once these alteration have been made, we anticipate the new anvil guide setup will prove highly successful on ID06-LVP.

Figure 1: (a) photograph of anvil guide as recovered from test experiment, with deformed Al cube inside. (b) 3 broken secondary anvils, demonstrating the failures eminating from the centre of the deformation truncation. (c) 2 broken anvils, demonstrating the expanding ring crack at the rear-face of the anvil. (d) The recovered experiments: (from left to right) 2 Al cubes, fired pyrophyllite, boron epoxy with gaskets, boron epoxy with no gaskets, boron epoxy with smaller gaskets.

References

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