EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

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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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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.

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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:** ***The density of wet lunar melts: critical data for lunar evolution models***	**Experiment number**: ES/578
Beamline:	**Date of experiment**: from: 15/06/2017 to: 20/06/2017	**Date of report**: September 5th, 2017
Shifts:	**Local contact(s)**: David SIFRE	*Received at ESRF:*

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ESRF

Introduction

The goal of the experiments was to measure the densities at lunar relevant *P-T* conditions of two hydrous primitive end-member lunar melts, namely the low-Ti Apollo 15C green glass and the high-Ti Apollo 14B black glass. During the allocated beamtime, experiments were performed using x-ray diffraction techniques previously developed for the Paris-Edinburgh press at ESRF [1]. The assembly consisted of a boron epoxy gasket, a cylindrical graphite furnace and a diamond capsule (Fig. 1). Diamond capsules ($Ø_{in}$ = 0.5 mm, $Ø_{out}$ = 1.5 mm and height = 1.0 mm) were used because of their incompressibility, high melting point, lack of reaction with the sample and because of its X-ray transparency. The synthetic equivalents of low-Ti Apollo 15C green glass and Apollo 14B black glass, both containing the appropriate amounts of brucite or $Mg(OH)_2$ to generate 4 wt.% H_2O upon melting, were packed in the diamond capsules. These mixtures were also doped with 500 ppm Cs to measure potential H_2O loss *after* the experiment, using the initial and post-experimental H/Cs ratios. Starting mixtures were analyzed with EPMA prior to density measurements and were observed to closely resemble the desired compositions. To prevent H_2O loss during the experiments, Pt end caps were used on both sides. These end caps both act as effective seals to prevent volatile loss and are efficient pressure transfer media during pressurization (Fig. 2). Capsules were enclosed in a hBN cylinder and hBN caps were placed on both ends. The furnace assembly was then fitted into a 7mm boron gasket. A ~50-100 µm diameter hole was drilled in the side of the BN sleeve surrounding the diamond capsule, which was filled with pressure markers hBN and Pt powders. These powders are required to determine the *P-T* conditions within the assembly using the equations of state of hBN and Pt.

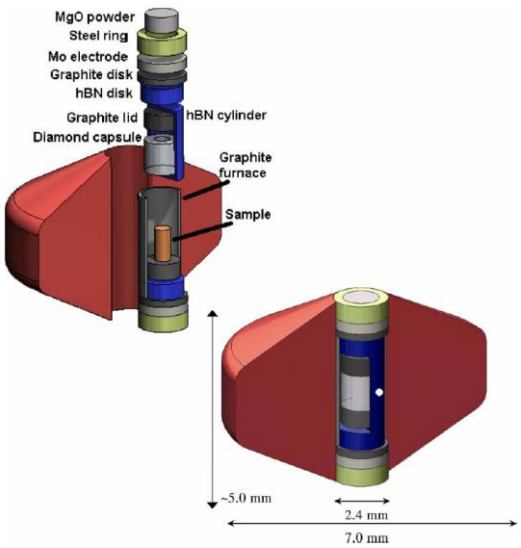

Fig. 1 Assembly used for *in situ* density measurements [1]

Observation and results

A total of 10 cell assemblies were prepared and loaded in the Paris-Edinburgh press at ID27. Five experiments were performed for each composition (Table 1) at different *P-T* conditions. At the desired peak oil pressure, experiments were rapidly heated (100-200 K/min) to minimize diffusion of H out of the sample. Diffraction spectra were collected until there were no signs of crystals remaining, i.e. the sample was completely molten (Fig. 3). In some cases the power used directly resulted in complete melting of the sample; power was then gradually decreased to pin-point the liquidus *T* using the technique outlined above (Table 1). This does not only yield multiple density measurements for the same sample at different *T*, but are also the first *in-situ* liquidus curves of hydrous lunar low- and high-Ti melts. Preliminary comparisons with anhydrous data for the same compositions and identical *P-T* conditions suggests that the liquidus *T* of both lunar melts with 4 wt.% H_2O are >150-200 K lower. After collection of the diffraction spectra, samples were rapidly quenched and additional measurements were obtained at room *P* by rotating the assembly upside-down by 180 degrees. Several run products showed clear evidence absence of quench crystals, which is very promising given the fact that it will significantly decrease errors on subsequent fitting of absorption spectra to obtain densities. Initial fitting of the hBN-Pt calibrations yielded *P-T* conditions that compared well with previous calibrations [1].

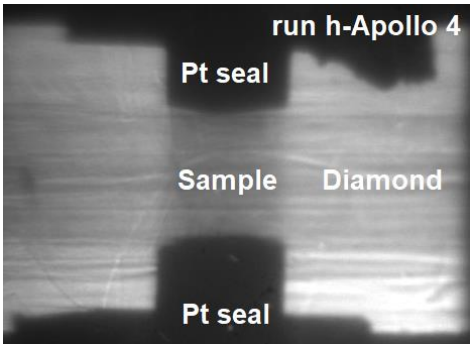

Fig. 2 Typical run product showing that Pt is an effictive seal to prevent water loss at high *P-T*. Significant water loss is also suggested from the much lower melting temperatures observed for the hydrous lunar melts relative to previous work for the same anhydrous lunar melts [3].

Outlook

Data analysis is still in progress. Chemical run products will be analyzed in the next months using an EPMA to quantify possible H_2O losses. These H_2O contents will be used to compare the melt densities obtained from absorption spectra fitting with density data from their anhydrous counterparts for a given pressure [2]. Our preliminary results show that 1) H_2O was retained during the experiments, given the much lower melting points of the hydrous compositions relative to their anhydrous counterparts; 2) most lunar melts quenched to a crystal-poor glass, which is expected to result in very accurate density measurements; 3) melting points of hydrous low-Ti and high-Ti lunar glasses are at least 150-200 K lower than their anhydrous counterparts. To summarize, we showed in experiment ES/578 that we can realibly obtain densities and liquidus relationships of hydrous lunar melts, and that significant H_2O losses did not occur. We therefore showed that this set-up is also suitable for *in-situ* determination of viscosities of hydrous lunar melts. Precise experimental determination of the density, viscosity and melting points of hydrous lunar melts and constraining the dependence of these variables on *P-T* and composition of the silicate melt is absolute key for modelling the generation and evolution of primitive lunar melts and or understanding the early evolution of the Moon. With further experiments, we aim to provide the first full *in situ* data set on both the density ***and*** viscosity of primitive hydrous lunar magma as a function of *P-T*.

References: [1] Van Kan Parker et al (2010) *High Press Res* 30, 332 [2] Van Kan Parker et al (2012) *Nat Geo* 5, 186

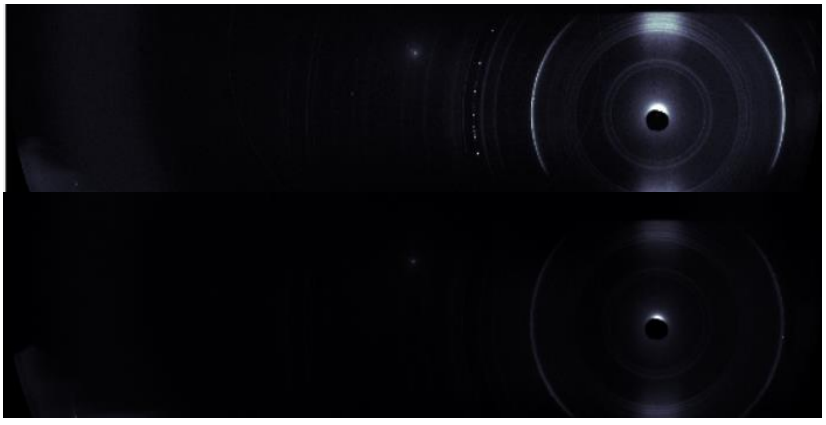

Table 1: Succesful xperiments performed during experiment ES/578. Preliminary *P-T* conditions were estimated based on calibrations of [1].

Run	*P* (bar; GPa)	*p* (watt)	T (K)
h-Apollo 1 (GG)	200; 1.5	315	1775
h-Apollo 2 (GG)	200; 1.5	350	1850
h-Apollo 3 (BG)	200; 1.5	315	1775
h-Apollo 4 (BG)	200; 1.5	350, 315	1850
h-Apollo 5 (GG)	320; 2.5	350, 315	1850
h-Apollo 6 (BG)	320; 2.5	350, 315	1850
h-Apollo 7 (GG)	320; 2.5	350, 315	1850
h-Apollo 8 (BG)	320; 2.5	350, 315	1850
h-Apollo 9 (GG)	450; 3.5	350, 315	1850
h-Apollo 10 (BG)	450; 3.5	350, 315	1850

Fig. 3: X-ray diffraction spectra for sample h-Apollo 2. Top image shows that the sample has not been fully molten, implied from the presence of distinct diffraction patterns. Also visble is the *P-T* calibrant. Lower image shows (near)-complete melting of the sample, showing only a small remainder of the crystal present in the top image.