ESRF	**Experiment title:** *The viscosity of lunar magmas at high pressure and temperature*	**Experiment number**: HD/534
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Introduction:

The goal of the experiments was to measure the viscosities of two compositional end members of primary lunar magma compositions, viz. the high titanium Apollo 14 black glass and the low titanium Apollo 15C green glass, at simultaneous high pressure and high temperature conditions relevant to the Moon. During the allocated beamtime, experiments were performed using the *in situ* falling-sphere viscometry technique that was recently developed at the beamline [1,2].

The cell assembly (Figure 1) consisted of a boron epoxy gasket, a cylindrical graphite furnace and a sample capsule. The original choice for the sample containers were CVD diamond cylinders because of their incompressibility, high melting point, the X-ray transparency and chemical inertia. For the capsule, $Ø_{in}$ = 0.5 mm, $Ø_{out}$ = 1.5 mm and height = 1.0 mm. Starting materials were synthetic analogues of the Apollo 14 black glass and Apollo 15C green glass, packed inside the sample containers. Two graphite caps, at both ends of the capsule, ensured compact sealing and pressure transfer to the sample. Capsules were enclosed in an hBN cylinder and hBN caps were placed on both ends. The furnace assembly was placed inside a standard 7 mm boron gasket. A small hole of ~50-100 μm diameter, drilled midway down the side of the BN sleeve surrounding the diamond capsule contained the pressure markers (hBN and Pt) used to determine the pressure and temperature by X-ray diffraction from their respective equations of state.

In previous experiment HD 417, WC spheres were observed to react with the high-Ti silicate melt during viscometry measurements, so we used rhenium spheres this time. These spheres were fabricated at VU University Amsterdam by applying 110 volts and 30 A to a strip of 25 micron thick rhenium foil. The spheres were then collected in a bowl of liquid nitrogen. Sphere diameters, determined by optical microscopy, ranged between 30 and 70 μm. A single sphere was carefully placed in the top one third of the sample and centred relative to the capsule walls.

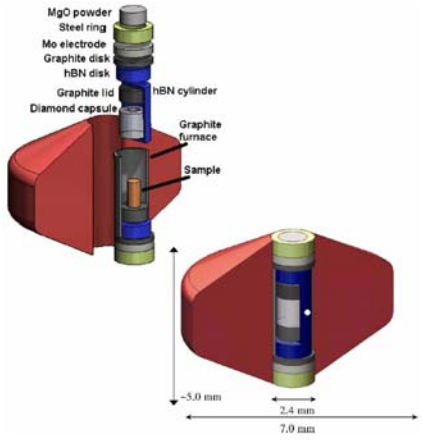

Figure 1: Typical cell assembly used for *in situ* viscosity measurements (from ref. [2])

Observations and results:

A total of 14 cell assemblies were prepared and loaded in the toroidal Paris-Edinburgh press at ID27. The first three experiments using the cell assembly shown in Figure 1 resulted in blowouts during heating, as observed in many experiments during

HD 417. With the goal of increasing our success rate, we made several modifications to the sample assembly. The modified approach included, in chronological order, (1) using a graphite capsule of similar dimensions as the original diamond capsule, (2) using a closed lid graphite capsule with $Ø_{in}$ > 0.5 mm instead of the diamond capsule, (3) using a Pt disk between the graphite lids and diamond/ graphite capsules to improve the seal and prevent sample material from escaping upon melting, (4) using a protective ring around the gasket material (as is usual in neutron diffraction experiments) to minimize flow of the gasket material. Failures during final heating continued until the final two experiments, that were successful in recording Re spheres falling in molten samples. The spheres did not react with the sample in these cases. For the last experiment (Fig. 2), we used a closed lid graphite capsule as the sample container and also used a protective ring around the gasket. At 150 bar oil pressure (corresponding to approximately 1.2 GPa), power to the furnace was first increased to 150 w at the rate of 8 w/min, followed by a 15 minute settling period. Temperature was subsequently raised very rapidly from 150 w to 340 w at the rate of 100 w/min. The Re sphere started to sink when the input power was 340 w (approximately 2156 K at *P* ~1.2 GPa as determined from the XRD patterns of Pt and hBN). The experiment was quenched and for obtaining a second measurement the press was rotated upside-down by 180 degrees. At 120 bar oil pressure the heating cycle was repeated and the Re sphere was again observed to fall at 340 w. There were a total of two successful measurements on the molten Apollo 15C Green glass sample at ~2150 K and 1 and 1.2 GPa respectively.

Results and outlook:

Data analysis is still in progress. The terminal velocity of the Re spheres was determined from the radiographic images, taken every 20 ms during the falling period (Figure 2). The viscosity coefficient obtained for the green glass composition is 1.4 Pa s.

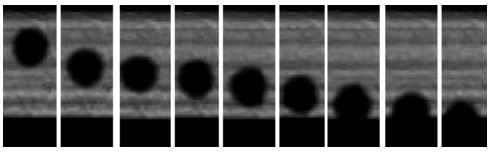

Figure 2: Snapshots of radiographic images of a falling Re sphere in molten Apollo 15C green glass. The Re sphere is clearly visible as a dark shadow against the light grey sample.

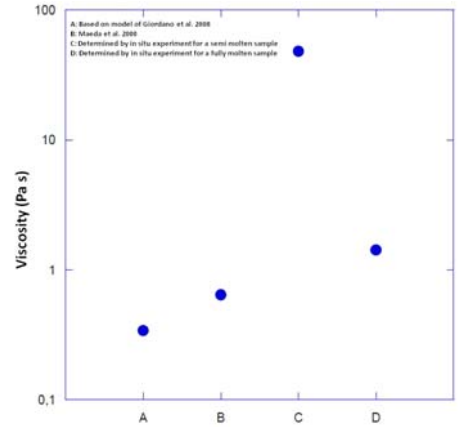

Figure 3: Comparison of viscosity of Apollo 15 Green glass (D) for a molten sample from this study at 1.2 GPa and 2156 K (D) to A) Model based calculation, B) from ref. [3] and C) value a measured on a semi molten sample in HD 417.

As illustrated in Figure 3, this value is much lower than 48 Pa s obtained from a semi-molten sample of this composition from an experiment during our previous ESRF beam time in February 2010 (HD 417). Our result is in much better agreement with both previous experiments using a different setup [3] and model predictions (0.34 Pa s, [4]). This illustrates that we have finally developed a setup that can provide reliable and reproducible viscosity data. We note that the improvements we tested were subsequently used at ID27 by the ETH Zurich group, who noted a significant increase in success rate in their experiments compared to previous experience using the original assembly depicted in Figure 1. This bodes well for the future of this technique.

Precise experimental determination of the viscosity of lunar melts and constraining its dependence on pressure, temperature and the composition of the silicate melt is a critical requirement to model the behavior of lunar melts, and the early evolution of the Moon. With further experiments, we aim to provide the first full *in situ* data set on the viscosity of primitive lunar magma as a function of pressure and temperature.

[1] Perrillat J.P. et al. (2010) *High Press. Res.*, in press. [2] Van Kan Parker M. et al. (2010) *High Press. Res.* 30, 332-341. [3] Maeda et al. (2000) *Japanese Geosciences Union*, abstract Pb-P004. [4] Giordano et al. (2008) *Earth Planet. Sci. Lett.* 271, 123-134.