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Introduction: Knowledge of silicate magma viscosity is of fundamental importance in decoding the magmatic processes driving the evolution of planetary bodies. The viscosities of terrestrial magmas are relatively well investigated and documented, but this is not the case for lunar magmas, which have compositions outside the terrestrial range. The viscosity of silicate melts is known to be strongly related to melt structure, composition and pressure-temperature (P - T) conditions. Here we aimed 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 P - T conditions relevant to the evolution of the lunar interior.

Experimental techniques: We used an *in situ* falling-sphere viscometry technique recently developed at ESRF beamline ID27, previously used to measure the viscosity of FeS liquid at high pressure [1]. The cell assembly used is shown in Figure 1, and is based on our previous ESRF *in situ* magma density determination studies on the same lunar compositions [2,3]. The cell consists of a boron epoxy gasket, a cylindrical graphite furnace and a CVD single crystal diamond cylinder sample container. Starting materials consisted of synthetic analogues of the Apollo 14 ‘black glass’ and the Apollo 15C ‘green glass’. Spheres of tungsten carbide were used in the experiments (Figure 2) because of their relatively high density of 15.63 g/cm^3 , high melting point of $2870 \text{ }^\circ\text{C}$, and expected low reactivity with the silicate magmas during the experiments. Spheres with diameters ranging between 60 and $140 \text{ }\mu\text{m}$ in diameter, as determined by optical microscopy, were used. A single sphere was carefully placed in the top one third of the sample and centred relative to the capsule diameter.

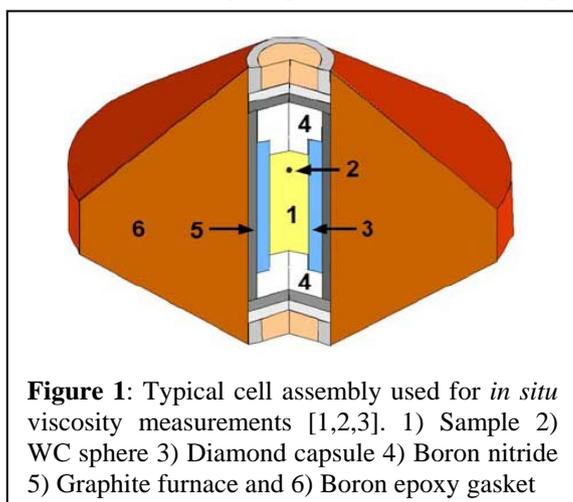


Figure 1: Typical cell assembly used for *in situ* viscosity measurements [1,2,3]. 1) Sample 2) WC sphere 3) Diamond capsule 4) Boron nitride 5) Graphite furnace and 6) Boron epoxy gasket

A total of fifteen *in situ* experiments were conducted. The cells were initially cold-compressed. Temperature was raised in a stepped manner with heating to the final temperature being done very rapidly to promote fast and full melting of the sample. Radiographic images were acquired every 20 ms during the final temperature increase to document the position of the WC sphere with time as soon as the sample was molten. After radiographic observations, pressure and temperature were held constant and diffraction patterns of the sample and the P - T calibrants were recorded. P - T conditions were subsequently determined from the diffraction pattern of hBN using the PVT equation of state of Godec et al. [4].

Observations and results: The failure rate of our experiments was exceptionally high, with only one successful run out of 15. There were many instances of blow-outs during heating, due to the abrupt flowing of gasket material. This very high blow-out rate was completely unexpected as we successfully used the exact same assembly at identical P - T conditions at ESRF for previous density measurements on the same samples [2,3]. Apart from a new supplier of boron epoxy gaskets we cannot think of a reason for this, and our number of experiments is too small to come to definitive conclusions.

In some instances, during heating radiography showed that the edges of the WC sphere started to get blurry due to reaction with the silicate melt. In one case the WC sphere was found to dissolve completely in the high-Ti black glass composition. Clearly WC is more reactive with these lunar magmas than expected. Future experiments will use molybdenum spheres. Previous *ex situ* experiments have shown that these show minimal interaction with lunar magmas during high P - T experiments [5].

Despite these experimental issues, one experiment was successful in recording a falling WC sphere

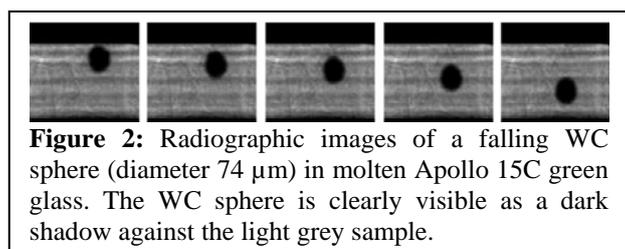


Figure 2: Radiographic images of a falling WC sphere (diameter 74 μm) in molten Apollo 15C green glass. The WC sphere is clearly visible as a dark shadow against the light grey sample.

through molten low-Ti ‘green glass’ in the absence of reactions (Figure 2). In this particular experiment, Apollo green glass was subjected to a hydraulic pressure of 150 bar (sample pressure \sim 1.3 GPa). The sphere started to sink when the input power was 375 w during final heating at 25 w / min.

Time-lapsed images of this experiment were analysed (Figure 3). The falling sphere method is based on

Stoke’s law which relates viscosity to the terminal velocity of the sinking sphere by the relation

$$\mu = \frac{2(\rho_s - \rho_m) \cdot a \cdot r^2}{9v} C_F$$

where μ is the viscosity (Pa s), $\rho_s - \rho_m$ the density difference between the sinking sphere and the melt (kg/m^3) – a term which is very well constrained in this case because of our previous *in situ* measurements of ρ_m , a is the gravitational constant, r the sphere radius (m), and v the sinking sphere velocity. C_F is the Faxen correction term which accounts for interaction between the sphere and the wall of the capsule.

The time versus falling distance profile constructed from the position of the sphere, tracked on each individual frame is shown in Figure 3. The terminal velocity determined from least-square regression of the linear part of the plot equals $18.896 \mu\text{s}^{-1}$.

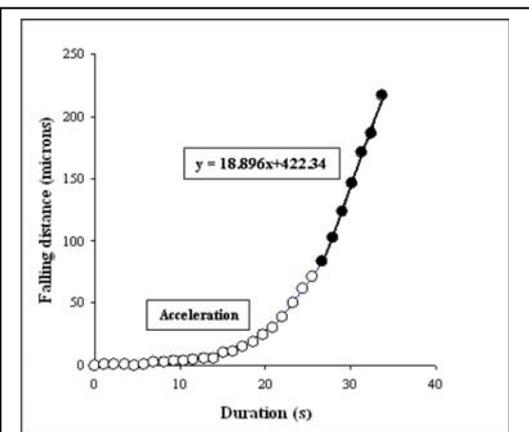


Figure 3: Time versus falling distance profile of the WC sphere in the Apollo 15C melt. This plot was used to determine the terminal velocity of the falling sphere. The slope of the least-squares best fit line through the seven points between 26 and 34 seconds is the velocity used to calculate the viscosity for this experiment.

The resulting viscosity coefficient obtained for the low-Ti Apollo 15C ‘green glass’ composition on the basis of the one successful experiment is \sim 48 Pa s. This value is orders of magnitude higher than the value of 0.64 Pa s previously reported by Maeda et al. [6] for a similar composition at similar conditions. The predicted viscosity calculated using the model of Giordano et al. [7] is also much lower, 0.34 Pa s at 1300 $^{\circ}\text{C}$. Additional data, in

particular electron microscope images of the run product, are required to rationalise this result. X-ray diffraction patterns of this experiment after the sphere had fallen suggest crystals were still present in the sample, but their proportion and sizes are not known. A sphere falling through a ‘crystal mush’ would obviously lead to higher than viscosities than expected from a fully molten liquid.

Summary and outlook: Despite unexpected experimental setbacks we have shown that *in situ* falling-sphere viscometry on silicate liquids is in principle feasible at ESRF. Future experiments should use Mo as a sphere to prevent sample contamination, full post-experiment characterisation is required to correctly interpret results, and the behaviour of the ‘new’ gaskets should be tested in comparison with the ‘old’ gaskets that were produced in-house.

[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] Van Kan Parker M. (2011) *PhD thesis*, VU University Amsterdam. [4] Godec Y.L. et al. (2000) *Sci. Tech. High Press.* University Press, 925-928. [5] Van Kan Parker et al. (2010) *Geochim. Cosmochim. Acta*, in press. [6] Maeda et al. (2000) *Japanese Geosciences Union*, abstract Pb-P004. [7] Giordano et al. (2008) *Earth Planet. Sci. Lett.* 271, 123-134.