ESRF	**Experiment title:** Effect of volatile-containing glass beads on sintering evolution of lunar regolith simulants	**Experiment number**: ES-1119
Beamline: ID22	**Date of experiment**: from: 01.03.22 to: 03.03.22	**Date of report**: 01.09.22
Shifts: 6	**Local contact(s)**: Andy Fitch	*Received at ESRF:*

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Report:

Measurements overview

Samples consisted of raw lunar simulant with varying quantities and compositions of imitated volcanic glasses, alongside the widely used space agency (ESA/ NASA) and natural (Hawaiian ash field) alternatives. The crystalline component was spiked with 10 wt% Pt to later determine sample temperature. Data was collected over a uniform heating and cooling cycle, with the mirror furnace programmed in 2 W intervals and held at maximum power for 30 minutes. Each measurement took around 4 hours. Due to tuning the maximum temperature, only the heating data of the first two samples is comparable with later measurements. The following summarises the completeness of sample datasets:

1. TUBS-M: heating only
2. TUBS-M 30wt% orange: heating only
3. TUBS-M 50wt% orange: complete cycle
4. TUBS-M 30wt% green: complete cycle
5. TUBS-M 50wt% green: complete cycle
6. TUBS-T: omitted due to time constraints
7. EAC-1A (ESA): complete cycle
8. JSC-1A (NASA): capillary (repeatedly) broke at high temperature
9. Hawaii ash: complete cycle but sample migration
10. Hawaii ash processed: dominated by single crystal reflection; broke at high temperature

Experimental challenges

By comparison of the raw simulant profile with earlier in-house measurements using the hot air blower, we were able to exceed the former's 1000 °C limit and observe increased melting. Using calibration tables, we aimed to peak at 1100 °C – at which the raw basaltic simulant retains little crystallinity. Although originally configured to reach 154 W, the maximum power had to be reduced to avoid capillary softening and drooping. Initial inference of temperature from the expansion of the Pt lattice constant using multiple peaks however proved inconsistent, although calculation of the idealised Pt lattice parameter at 134 W indicated a maximum temperature of 960 °C, supported by the retained crystallinity with 50 wt% glass (Fig.1). Consequently, the anticipated extensive melting was not observed.

The 1 mm quartz capillaries unexpectedly did not withstand high temperatures and chemically interacted with several of the sample compositions, melting out of the beam before 134 W. In all cases the capillary ruptured at the tip (Fig. 2), compromising the pseudo-inert sealed atmosphere (in reality some air may have been trapped between grains during filling, and subsequent evolved gas analysis has indicated substantial outgassing from the simulants over this temperature range). Vacuum conditions therefore appear unattainable with a sealed capillary and these samples.

Therefore, for future experiments at ID22 a more laborious sample preparation is envisioned using a large number of pre-sintered quenched samples and the automated sample feeder for high throughput. Measurements should then be less disrupted and although not tracking in real time, compositional effects can be deduced from the average of several samples, increasing reproducibility.

Results and interpretation

Sample state was observed during the heating as a function of glass content, from which sintering rates may be calculated considering the onset and extent of melting. Although peak broadening is observed at higher glass concentrations, both 50 wt% high temperature curves show a lower amorphous contribution than at ambient temperature (purple lines in Fig. 1), indicative of loss of sample through the line of sight of the beam. Therefore, a quantitative comparison using relative proportions via the fixed Pt component (assuming homogenous distribution) is sought. At the elevated holding temperature certain mineral phases are observed to grow preferentially from the melt. High temperature variations between simulants helps elucidate differences in material handling (rheology). Comparison after recrystallisation is limited, but discrepancies between samples may be used to inform preferred applied temperature rates and post-processing strategies.

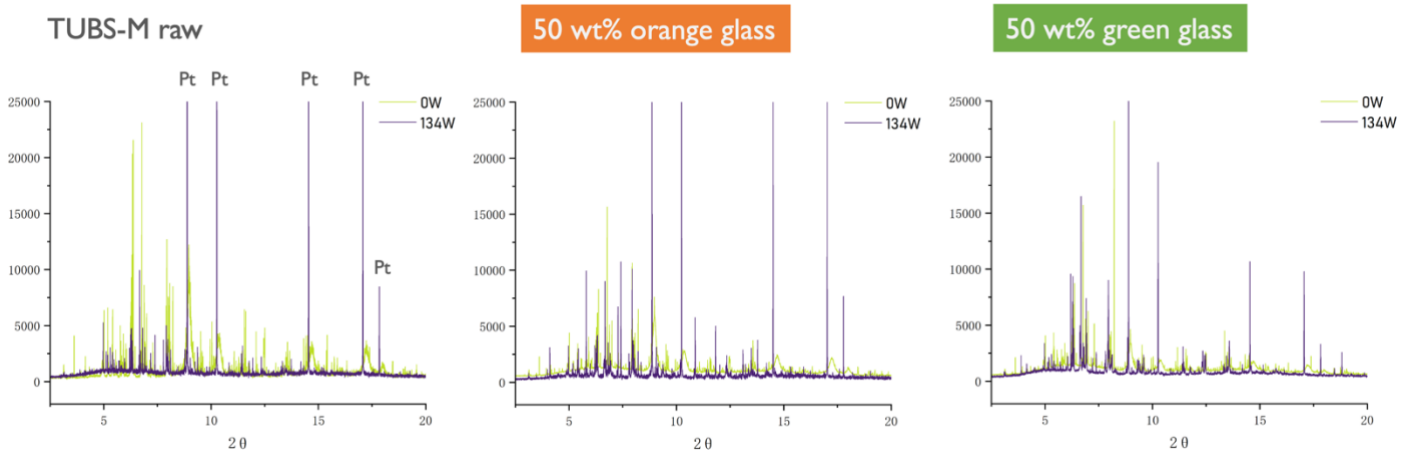

Fig. 1 (*above*)**.** XRD patterns of raw simulant TUBS-M, alongside 50 wt% volcanic glass fractions at ambient and maximum temperatures (134 W).

Fig. 2. (*right*)**.** Perforated capillary tip and escape of melt.