EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: High temperature sintering evolution of site-specific lunar regolith simulants	**Experiment number**: ES-1197
Beamline: ID22	**Date of experiment**: from: 27.01.2023 to: 30.01.2023	**Date of report**: 07.03.2023
Shifts: 6	**Local contact(s)**: Andy Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Ms. Jennifer Sutherland *

Dr Thomas C. Hansen

Prof. Dr. Donald Dingwell

Dr. Stefan Linke

Report:

The beamtime can be split into two approaches, which were completed with varying success.

Automatic sample changer

The robot performed extremely reliably to enable 2 x 10.5 minutes counting time of 120 pre-prepared samples under ambient temperature conditions. Samples consisted of 12 different simulant geologies, pre-quenched at the high temperature melt labs at LMU at intervals between 1000 °C and 1300 °C (basalt/ 'lunar mare') or 1500 °C (anorthositic/ 'lunar highland'). Intervals were at 25 °C or 50 °C steps, depending on the pre-informed range of interest determined by earlier D20 (ILL) and TGA measurements. This time resolution was fine enough to discern key periods of melting. Most interestingly of all was the observation that TUBS-M +50 wt% synthesised orange glass retained crystallinity at 1300 °C, whereas the fully crystalline TUBS-M on its own did not (plots omitted for confidentiality). This result is contrary to that observed from the addition of sodalime as a simple placeholder to observe the effects of added glass, which is prolific on the Moon but a missing component in many simulants. Further investigation will consider the chemical interaction during melting between the orange glass and regolith.

Despite not taking advantage of the in situ heating capabilities at the beamline, the reliability of the sample changer enabled a complete set of data collection, which had not been achieved in earlier beamtime allocation due to furnace complexities (and, previously limited to ~1050 °C). Additionally, reproducibility concerns could be addressed by the 'new' sample between temperature increments. Inspection of groups of patterns per simulant enabled outliers to be identified (possible preferential grain alignment) and capillary re-filling to test replicability of trends.

Samples were loaded into 1.5 mm diameter Kapton capillaries, which mostly encompassed the particle size distribution of the simulants pre-heating. This width was essential to avoid capillary blocking, given the large number of samples to prepare, as well as to achieve a representative cross section of the multiphase components (assisted by crushing in a zirconia mortar and pestle). Kapton also has advantage of being more forgiving/ flexible on spin realignment than the 1 mm borosilicate glass alternative.

<u>Induction heater</u>

We borrowed the induction heater from sample environment pool with the intention of doing four in situ measurements for comparison. The furnace ostensibly had a regulation temperature of 1250 °C (calibrated as 1158 °C at the thermocouple). Samples were dehydrated for 2h at 300°C in the ID22 oven to avert a build-up of gas pressure inside the sealed capillaries. The available Pt capillaries were too narrow (0.57 mm inner diameter offering the largest volume to wall thickness ratio), whereas the MgO showed many parasitic peaks at the level of the sample intensity. Therefore, we opted for 1 mm Al2O3.

The (sole) thermocouple broke on the second temperature cycle, so we had to abandon this line of measurements. However, for continuing at a later date, some experimental issues were observed:

- The difference in Al2O3 peak intensities with and without a sample doesn’t seem proportional to the absolute intensity, nor any wavelength dependence. Background subtraction therefore does not appear possible. Could try with boron nitride.
- There is a broad hump (pink line 18°+, **Fig. 1**) in the RT pattern taken outside the furnace compared to within, possibly originating from the Kapton walls. The beamline scientist has observed previously when the beam spends too much time in the air outside the nozzle just before the sample, but questionable why not observed in other patterns.
- The measurements within the furnace also have a prominent bump ~25° - possibly parasitic scattering from the surrounding graphite.

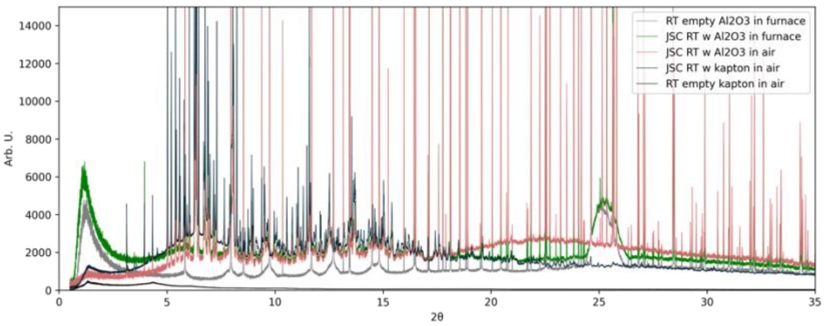

Fig. 1. Parasitic signals observed in the data above 18° (pink) and ~25° (furnace) data.