Experimental report

05/11/2022

Proposal: **EASY-760** **Council:** 4/2020

Title: Lunar regolith simulants TUBS-M and TUBS-S phase characterisation andthermal sintering behaviour

Research area: Chemistry

This proposal is a new proposal

Main proposer: **Thomas HANSEN**

Experimental team:

Local contacts: Thomas HANSEN

Samples: (Ca,Mg)x(Si,Al)yO

Instrument	Requested days	Allocated days	From	To
D20	20	20	01/02/2021	02/02/2021

Abstract:

InnovaXN PhD thesis at ILL: Jennifer SUTHERLAND

We want to take neutron diffraction patterns at good resolution from the two main lunar regolith simulants, developed at TU Braunschweig (TUBS), TUBS-M and TUBS-S. For later collaboration with space agencies (ESA) it is of importance to take room temperature diffraction patterns under vacuum and in air in different sample containers (vanadium, quartz, aluminium).
After this we will slowly heat the two regolith simulants under vacuum on a linear temperature ramp up to 1000C to study the phase transitions during the sintering process. This needs to be known, as lunar regolith is to be used as construction material for shelters and habitacles for a manned lunar base. Additive manufacturing through melting or sintering lunar regolith is the envisaged processed.

The aim of experiments INTER-525 and INTER-561 was the initial in-situ characterisation of lunar regolith simulants TUBS-M and TUBS-T, representing mare and highland geologies respectively. During the beamtime – conducted at D20 over several days spanning cycles 212 and 213 – we tracked the sintering (incomplete melting) behaviour as a function of temperature, whilst also covering the role of a basic but well-characterised soda-lime glass additive as a sintering agent.

We obtained a good sequential refinement (Fig. 1) on the as-received multi-phase simulants up to 1100 °C in vacuum (10^{-4} mbar). This maximum temperature induces almost complete melting of the basaltic mare powder at the experimental limit of D20's own furnace, used in preference to a pooled furnace for more agile and responsive sample changeover during this initial characterisation.

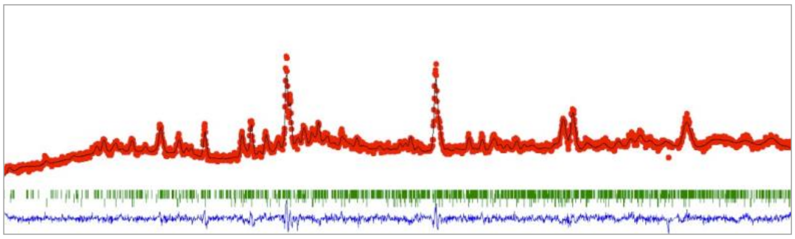

Fig. 1. Fitted NPD pattern of simulant TUBS-M without glass at 1060°C following sequential refinement (chi2 = 1.80).

The principal phases identified within TUBS-M are plagioclase (labradorite), pyroxene (augite) and olivine (forsterite), with minor proportions (<3 vol%) of titanomagnetite; anorthoclase identified in thin section (<<1 vol%) could not be confirmed. TUBS-T meanwhile is dominated by plagioclase (bytownite), with minor rutile (TiO_2). Notably, hydrous phases from weathered secondary minerals are present (<3 vol%) in both samples, presented as a strong background level diminishing by 440 °C in TUBS-M, and at staggered intervals up to 800 °C in TUBS-T. Water-bearing minerals are problematic for imitating lunar processes as they are not encountered on the Moon's surface; dehydration of the samples is therefore essential to avoid foaming or even sample container rupture, driven by the expulsion of gas across a wide viscosity range. Such material handling deductions are essential to the space hardware community to ensure that developed surface processes will function as anticipated, once deployed remotely. A further takeaway is the observed melt migration up the walls of the sample can, consistent with [1], leaving a central void and thus varying the sample volume encountered by the beam. Extraction of crystalline to amorphous fraction is thus only appropriate in a relative sense.

With the addition of soda-lime glass spherules in sizes and proportions consistent with lunar impact and volcanic melt, increase in volume fraction was seen to affect onset of melting, degree of melting and extent of recrystallization (Fig. 2). Phases diminish in a chronology consistent with their individual melting temperatures. Note that above the glass transition (~ 570° C) of the soda-lime glass, additional crystalline peaks transiently emerge until the onset of partial melting.

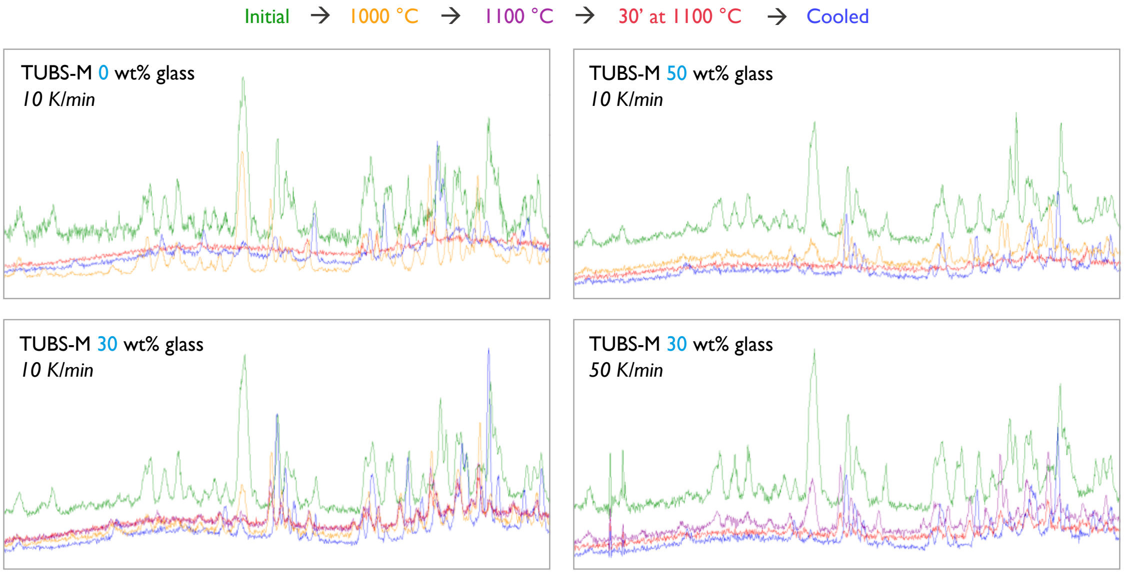

Fig. 2. Evolution of TUBS-M with temperature with varying soda-lime glass (45-90 µm) fraction.

Trialling a faster temperature ramp with 30 wt% soda-lime returns a sharp decrease in peaks after 1040 °C consistent with 0 wt% glass, but unlike the latter continues to melt at the holding temperature due to the thermal lag. Curiously, both 30 wt% glass patterns retain more crystallization than without any glass at all.

Reproducibility was also an issue considering the effect of soda-lime bead size, as initial results appeared to suggest differential melting. Verification measurements later demonstrated no obvious trend in the extent of melting, consistent with thermal equilibrium conditions. This glass size independence is useful going forward in imitating and incorporating more chemically rich and realistic glasses.

Partial melting of TUBS-T before 1100 °C was observed only with the aid of glass additives and on first impression is a less favourable, more energy intensive, construction material. The data provides the first comparative in situ results of representative mare and highland material processing and promotes a more conscious selection of simulant geology during wider technology demonstrations. The subsequent proposal 5-25-285 aims partly to retain a focus on highland compositions (coinciding with the recently defined landing zone for upcoming surface missions around the lunar South Pole) at elevated temperatures up to 1300 °C, to enable tracking of partial melting consistent with lab-based experiments over this range.

References

[1] Dominguez, J.A. and Whitlow, J., 2019. Upwards migration phenomenon on molten lunar regolith: New challenges and prospects for ISRU. *Advances in Space Research*, *63*(7)