Experimental report

11/09/2023

Proposal: 5-25-285 **Council:** 10/2022

Title: Laser sintering process of lunar regolith simulants using an aerodynamic levitator by time- and temperature-resolved NPD

Research area: Materials

This proposal is a resubmission of 5-25-280

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Samples: Lunar regolith simulant (basaltic) NaCa2Mg2FeSi8Al3Ox
Lunar regolith simulant (anorthositic) Ca3Si8Al3Ox

Instrument	Requested days	Allocated days	From	To
D20	5	5	30/05/2023	04/06/2023

Abstract:

We aim to use neutron diffraction to generate the baseline fundamental description of lunar regolith simulants, with a particular emphasis on phase evolution during sintering and cooling. Samples consist of metal-oxides and mineral composites in chemical and shape concentrations according to returned Apollo specimens. Impact melt and agglutinated material is not yet widely present in available simulants for technology verification studies by the wider space community. 3D printed constructions, separation of oxygen and metal, and the extraction of volatiles (water) are application areas to be developed and refined. Heating is seen as the dominant modification process in all processing of regolith. Phase behaviour undergoing laser melting up to 1500 °C is proposed to be investigated, employing an aerodynamic levitator to remove gravity and nucleation influences. Comparisons are made to conventional vanadium can containment. A particular focus is on differences between leading highland simulant providers encompassing varying anorthosite/ agglutinate fractions, as represents the upcoming Artemis landing zone (2025) in contrast to earlier studies dominated by basaltic mare material.

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Programme

The proposal focused on the recrystallisation behaviour of lunar regolith simulants, incorporating an aerodynamic levitator to reach, contain, and quench higher sample temperatures (up to 1800 °C). The schedule aimed to investigate both the effects of starting geology (mare, mare with synthesised volcanic glass, and highland simulant); atmosphere (invert i.e., argon, vs. oxidised, i.e., air); and cooling rate (quench, 0.01%, and 0.05% laser power s^{-1}, based on preliminary testing out-of-beam) in a combinatorial approach. It had been anticipated that the middle glass-rich sample 'OG' (TUBS-M + 30 wt% orange glass) would crystallise best, as it is enriched in Ti and Mg.

Levitator installation

The levitator was constructed at the DLR Cologne and driven to site in Grenoble. Dismounting required a forklift to convey between the drop-off area and instrument. Installation at the beamline took around a day for full alignment. The square mounting board hosting the levitated sample was able to just fit within the arc of the ROC.

Signal quality

Although the high count rate on D20 is in general suited for quick time-resolved diffraction, owing to small sample size (~2 mm diameter) able to be supported by the gas stream, samples were instead measured for 3 hours post-cooling. 3-hour counts permitted delineation of small peaks from the noise; an overnight trial beyond this provided diminishing returns for an unacceptable restriction of the sample set. Given the prominent amorphous signal of the vanadium chamber shielding the laser-illuminated sample, it was removed during counting.

For the slowest cooling rates, in situ measurements were also acquired. However, count integration time (~20 min total from amorphous starting point) was insufficient to distinguish smaller peaks from the noise (Fig. 1).

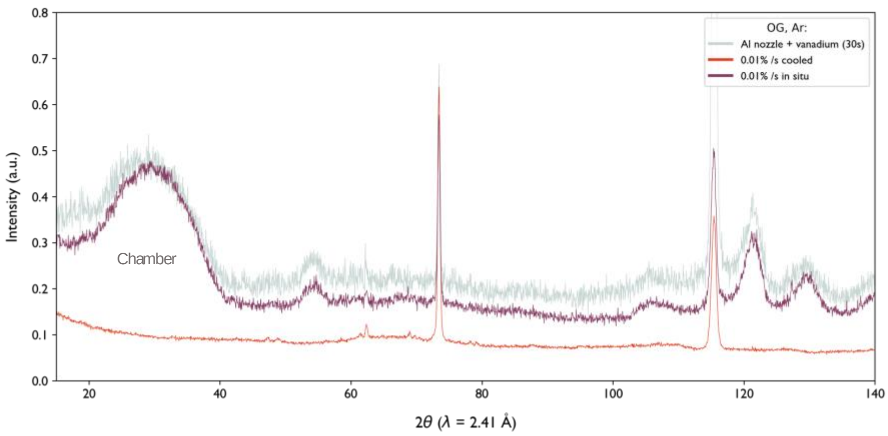

Fig 1. In situ (purple) vs. 3-hour post cooling (red) integrated counting for TUBS-M + 30 wt% orange glass during cooling from ~1500 °C. The grey line represents the background apparatus contribution.

<u>Preliminary results</u>

Main outcomes:

- TUBS-M + 30 wt% orange glass was the only sample to crystallise at the intermediate rate (0.05% s^{-1}), but the crystallisation of the highland simulant TUBS-T at 0.01% is much more pronounced (Fig. 2).
- When crystallisation occurred, it was only in the argon atmosphere.
- No crystallisation for any cooling rate with mare simulant TUBS-M.
- No crystallisation observed for any sample following apparent recalescence peak in temperature profiles (Fig. 3). Improved counting statistics in a future iteration would enable better pinpointing of temperatures associated with undercooling events.

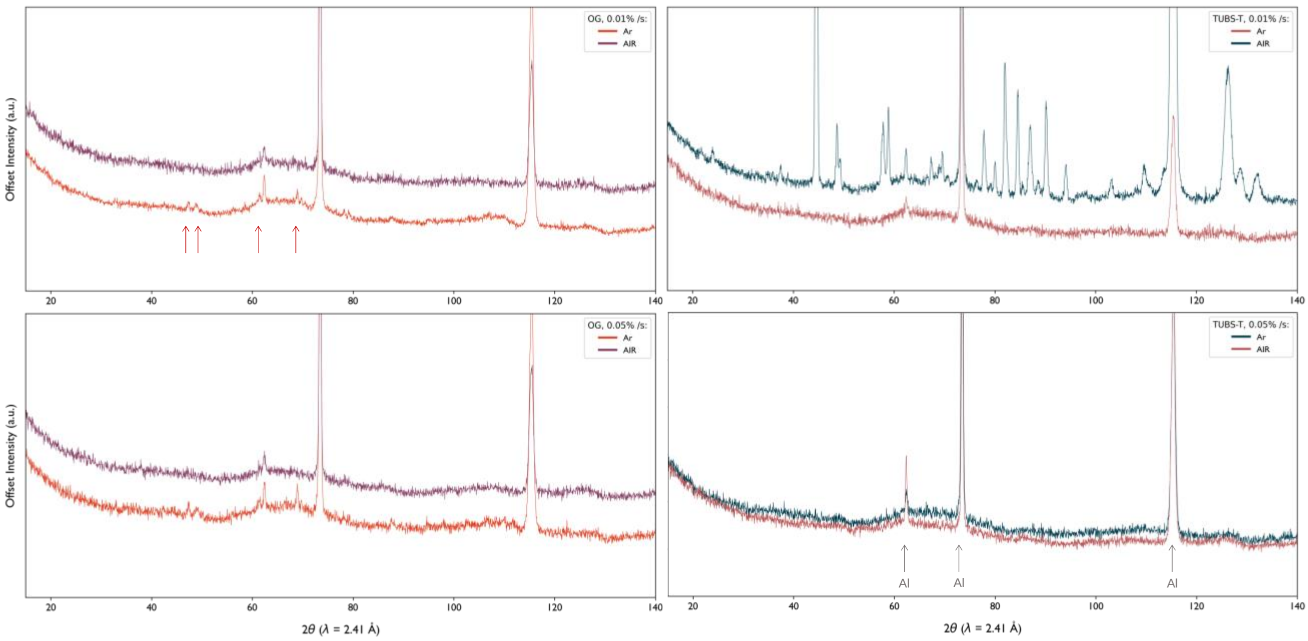

Fig 2. For TUBS-M + 30 wt% orange glass, minor crystallisation (to approx. same extent) is observed under Ar only, at both the slow and intermediate rates (left). Significant different between rates for TUBS-T (right).

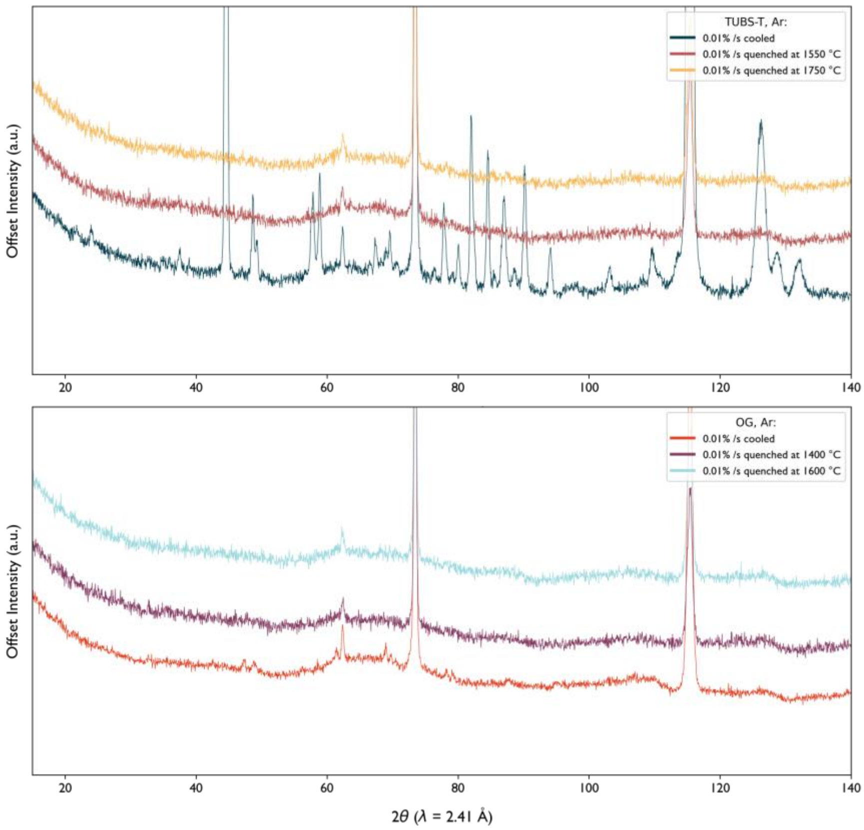

Fig 3. Following quenching from supposed recalescence peak in temperature profile, no crystallisation was observed in any of the samples. Improved sample stability for temperature calibration or continuous in situ counting would help narrow the event range.