THERMAL PROPERTIES AND MICROSTRUCTURE OF REMELTED LUNAR REGOLITH SIMULANT (OPRL2NT).

Anis Parsapoor and Alan Whittington[1], [1]Department of Earth and Planetary Sciences, The University of Texas at San Antonio, One UTSA Circle, San Antonio TX 78249, Email:anis.parsapoor@utsa.edu

Introduction: Surface-based exploration of the Moon will require the construction of landing and launch pads to mitigate dust hazards, and of robust habitats to protect lunarnauts from temperature extremes and micrometeorite hazards. Because of the limited supply of lunar regolith on the Earth, analog materials (simulants) of lunar regolith are required as they allow more comprehensive studies [1]. Texture and grain size play a dominant role in controlling mechanical properties; therefore, knowledge of crystal nucleation and growth rates is important when optimizing the conditions of melting and cooling rate of molten regolith.

Methods and Materials: We focus on effects of different cooling rate on microstructural and thermal properties of a high-Ti mare lunar simulant, OPRL2NT. This consists of 10% anorthosite, 75.6% basalt, 14.4% ilmenite developed by Off Planet Research, LLC. The in situ differential scanning calorimetry (DSC) technique was used to investigate the solidification paths of OPRL2NT lunar mare simulant. Samples were heated to 1500 °C at 30 °C/min in PtRh pans under an Ar atmosphere, in a Netzsch DSC-404 F1 Pegasus. The samples were then cooled at different rates. The heat flow curves as a function of temperature were recorded three times for each experiment: first with an empty pan (baseline), second with a sapphire reference material (standard), and third with the sample under investigation. Data for the three curves can be combined using the Netzsch Proteus software to calculate apparent heat capacity for each cooling rate. Calorimetric measurements were combined with textural analysis conducted on basaltic melt cooled from liquidus to solidus conditions at rates of 10, 30, 40, 60, and 80 °C/min. The recovered run products were mounted in resin and polished for microscopic investigations.

Results: The heat capacity of OPRL2NT powder slowly increases from ~0.8 to ~1.0 $Jg^{-1}K^{-1}$, until the glass transition, with a maximum at about 670 ˚C ($T_{g,peak}$). This is followed by a trough in apparent heat capacity from ~750 ˚C to ~950 ˚C, extending to an apparent heat capacity of ~0.1 $Jg^{-1}K^{-1}$ (Fig. 1a). The area of this trough corresponds to the enthalpy of crystallization of about -40 Jg^{-1}, where the negative sign indicates the exothermic nature of crystallization. The melting peak, beginning at ~1014 ˚C ($T_{m,onset}$), reaching a maximum at ~1163˚C ($T_{m,peak}$), with the heat capacity then falling and leveling off at ~1.1 $Jg^{-1}K^{-1}$ at ~1400 ˚C (Fig. 1a). The area under this peak represents the enthalpy of melting, about +407 Jg^{-1} in this case, where the positive sign indicates that melting is endothermic. For this experiment, 1440 ˚C marks the effective liquidus temperature (T_{liq}). During cooling of OPRL2NT from 1500˚C, heat capacity of the liquid is about 1.2 $Jg^{-1}K^{-1}$. When cooling at 30˚C/min, crystallization begins around 1200˚C and peaks at ~1078˚C and appears to cease at around 875˚C (Fig. 1b). The enthalpy of crystallization is ~105 J/g, suggesting about 25% crystallization. The glass transition begins at ~730˚C and is complete by ~670˚C.

Cooling rates play a crucial role in the microstructural evolution of textures, crystal size distributions and growth rates of crystallized minerals. To reveal the growth process and changing morphology of Fe-Ti crystals during the cooling experiments, we recovered the samples from the DSC, mounted them in resin and polished for microscopic investigations. At a cooling rate of 80 °C/min, a few patches suggest incipient crystallization, but no well-formed crystals were observed Fig. 2a). At slower cooling rates of 60 and 40 °C/min, small feathery crystals were observed which may have nucleated heterogeneously on the PtRh pan (Fig. 2b). At cooling rates of 30 and 10 °C/min, acicular euhedral rutile and ilmenite crystals were clearly visible (Fig. 2c and d). These crystals are arranged in sub-parallel mats, which intersect the orientation of crystals in other mats at high angles. At a cooling rate of 10 °C/min, the crystals grew mainly via continuous growth, but above 30 °C/min, the main growth way was lateral growth and eventually forms a quadrilateral. When the cooling rate is fast, the viscosity of the melt increases quickly, inhibiting the diffusion of titanium [2]. This leads to rutile crystals being smaller at faster cooling rates. The sample crystallizes extensively when cooled at 30 and 10°C/min (ilmenite, rutile and glass), but only slightly at 40, 60 and 80°C/min. With decreasing cooling rate from 80 to 10 °C/min, the temperature of peak latent heat release increases from 822 to 1164°C, and the total enthalpy of crystallization increases from -17 to -105 J/g.

Conclusion: For OPRL2NT, depending on the cooling rate, the major crystallizing phase were rutile and ilmenite. The molten simulant quenches to glass containing <5% crystals when cooled faster than 30 °C/min. At cooling rates of 10-30 ˚C/minute there is about 25% crystallization.

References:

[1] Woods-Robinson R. Siegler M.A. Paige D.A. (2019) *JGR, 124*, 1989–2011. [2] Zhang W. Zhang L. Li Y. and Li X (2016) *High Temp. Mater. Proc. 35(8)*, 787-797.

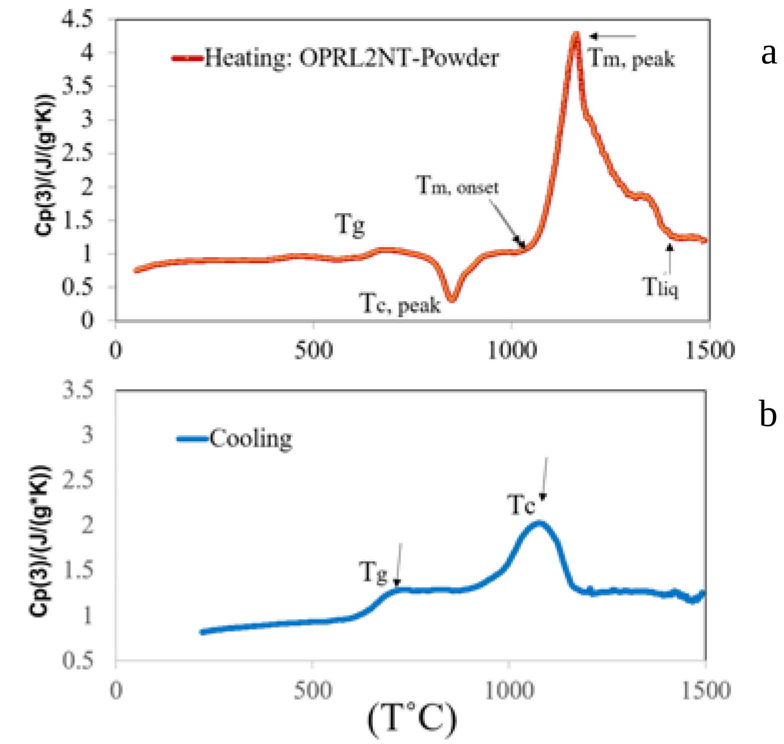

Fig.1. Apparent heat capacity vs temperature for the high Ti lunar simulant: OPRL2NT, heating and cooling rate 30 °C/min. The exothermic peaks in cooling stages represents crystallization of rutile. Cp: apparent heat capacity.

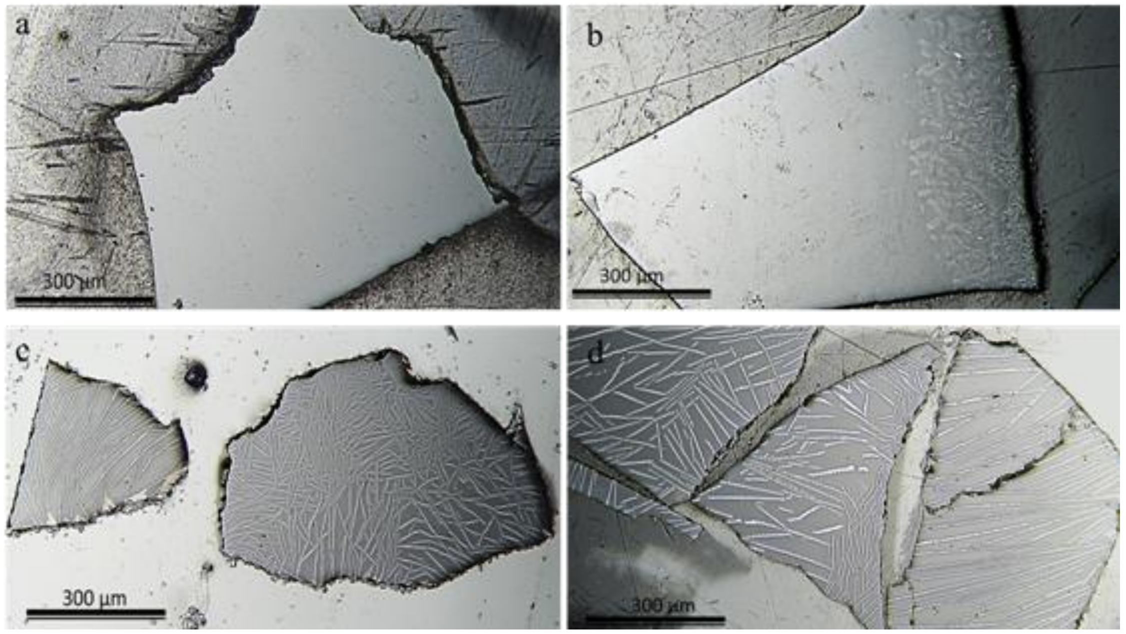

Fig. 2: Reflected light microscopic images of recovered products of OPRL2NT after crystallization at different cooling rates. OPRL2NT crystallization texture at (a) 80 ˚C/min, (b): 60 ˚C/min (c): 30 ˚C/min and (d): 10 ˚C/min. White needles represents rutile and light grey matrix phases are amorphous. (scale bar: 300 µm).

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