

## Viscosity measurements of selected lunar regolith simulants

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### ABSTRACT

In the context of evaluating lunar construction options, this study focuses on characterizing the viscosities and glass transition properties of lunar regolith simulants to support the development of additive manufacturing processes using molten regolith. Employing the modular TUBS lunar regolith simulant system, we measured the viscosities of different simulants through high-temperature experiments conducted between 1051 and 1490 °C using concentric cylinder viscometry in air. Additionally, differential scanning calorimetry (DSC) was utilized to evaluate the glass transition temperatures, which were in the range between 689 and 815 °C. The measured viscosity data were parameterized by the Vogel-Fulcher-Tammann (VFT) equation, which is adept at describing the viscosities and related properties of silicate liquids. The measured viscosities were compared with the predicted values of six viscosity models. The model by Sehlke and Whittington (2016) best predicts the viscosities of the tested lunar regolith simulants at superliquidus temperatures, and no model adequately predicts viscosities at the glass transition temperature, indicating a need for further research in this area. We infer that 3D printing technologies based on molten lunar regolith are, viscosity-wise, best constrained to highland regions. The reduced environment on the Moon influences the 3D printing process in a positive manner.

**Keywords:** Viscosity, glass transition, fragility, lunar regolith simulant, rheology, differential scanning calorimetry, concentric cylinder, silicate melts, Moon, TUBS-M, TUBS-T

### INTRODUCTION

The return to the Moon and the associated construction of a permanent and self-sustaining research station on the lunar surface is currently the focus of numerous space agencies and private companies (Crawford 2004; Hufenbach et al. 2014). Due to its near-ubiquitous presence, the lunar regolith offers a potentially limitless supply of raw materials for the production of large surface structures (Johnson and Chua 1993; Wilhelm and Curbach 2014).

The fabrication of components and structures using lunar regolith simulants on Earth has already been demonstrated using, for example, selective laser melting (Gerdes et al. 2018; Reitz et al. 2021; Ginés-Palomares et al. 2023b), microwave sintering (Taylor and Meek 2005; Kim et al. 2021), solar sintering (Meurisse et al. 2018; Fateri et al. 2019b) and D-Shape technology (Cesaretti et al. 2014). Additive manufacturing with molten lunar regolith simulants for the production of structural components is currently being pursued at Technische Universität Berlin (TU Berlin) using a process in which the regolith is electrically melted in a crucible, flows by gravity through a nozzle at the bottom of the crucible, and is then applied in layers to form a solid component after cooling (Stapperfend et al. 2023). As no material additives are foreseen for this process, the material transport effort from Earth can be

minimized, reducing costs enormously, especially for large construction projects. The main driving factor for this technology is the melt viscosity ( $\eta$ ), which describes a fluid's internal resistance to flow. The viscosity of silicate melts is mainly dependent on the chemical composition, temperature, volatile, and crystal content and has been intensively investigated in the past (e.g., Romano et al. 2003; Lavallée et al. 2007; Giordano et al. 2008; Villeneuve et al. 2008; Di Genova et al. 2013).

Due to the geological diversity of previous landing sites on the Moon and those initially foreseen in the context of the Artemis program, continued characterization of lunar regolith material—particularly under elevated temperature conditions—is required to inform future site selection and the optimization of additive manufacturing technologies. Inherent to high-temperature technologies involving melt manipulation is its flow throughout the printing process. Stresses involved in melt flow will be dictated at superliquidus conditions via the equilibrium Newtonian shear viscosity of the molten regolith.

Against this backdrop of potential large-scale technologies and planetary-scale geological complexity, it may come as a surprise that the high-temperature behavior of lunar regolith and its terrestrial simulants remains largely unexplored. General inferences can be made regarding the potential variation of melt properties from parameterization programs that have been developed following decades of experimental work in the geosciences (Richet 1984; Lange and Carmichael 1987, 1990; Persikov et al. 1990; Dingwell 2006; Neuville 2006; Sehlke et al. 2014). This

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basis, together with the knowledge of the wide chemical range of terrestrial magmatic rocks, as well as of lunar rock and regolith known to us, leads to the expectation that the physical properties of molten regolith will vary widely with both temperature and chemical composition. Although there is some preliminary work on glass production from lunar regolith (Magoffin and Garvey 1990; Mackenzie and Claridge 1979; Schleppi et al. 2019, 2021) and high-temperature behavior of lunar regolith and its simulants (Ray et al. 2010; Street et al. 2010; Morrison et al. 2019; Fateri et al. 2019a; Sehlke and Whittington 2020; Zocca et al. 2020; Farries et al. 2021; Whittington and Parsapoor 2022; Ginés-Palomares et al. 2023a), additional experimental measurements of the high-temperature properties of chemically accurate lunar regolith simulants are necessary to better constrain the role of glass in manufacturing.

In this study, the superliquidus Newtonian shear viscosity of liquids aimed at mimicking returned Apollo compositions was determined, which were generated by the combination of lunar regolith simulants based on the TUBS system developed at the Technische Universität Braunschweig in Germany (Linke et al. 2018, 2020). Viscosities just above the glass transition temperature were determined by applying the “Shift Factor” to the calorimetric results to generate the Vogel-Fulcher-Tammann equation. This contribution is intended as the launch of a campaign to characterize the high-temperature flow behavior of remelted lunar regolith simulants over an adequate range of their likely initial chemistry, mineralogy, and aggregate components, first introduced by Sutherland et al. (2022).

## MATERIALS AND METHODS

### Lunar regolith simulants

For this work, simulants from the modular lunar simulant system TUBS were used. The base simulants TUBS-M and TUBS-T correspond to the average chemical composition of the lunar mare and the lunar highland regions, respectively, and were mineralogically and chemically characterized by Linke et al. (2020). An intermediate mixture of the two base simulants, named TUBS-I, was also investigated. Based on the modular simulant system, chemically adapted simulants (CAS) of specific samples from Apollo 17 were created for another research project at TU Berlin (Linke 2022). The CAS were created by mixing the base simulants with raw minerals to chemically mimic specific sampling locations.

The following CAS were generated by Linke (2022) in preparation for a possible lunar technology demonstration of the MOONRISE laser melting payload (Linke et al. 2022):

- Station-5: Mean value from Apollo 17 sample 75061 and 75081
- LRV-6: Apollo 17 sample 74121
- LRV-7/8: Mean value from Apollo 17 sample 75111 (LRV-7) and 75121 (LRV-8)

### Concentric cylinder viscometer

The lunar regolith simulant samples prepared at TU Berlin were transported to the Ludwig-Maximilians-Universität München in the form of powders. Approximately 150–200 g of each powder was melted in a thin-walled iron-saturated Pt crucible under direct heating at 1500 °C in a Nabertherm box furnace with Superkanthal MoSi<sub>2</sub> heating elements and ZrO<sub>2</sub> fiber inner insulation walls in air. A plate of dense ZrO<sub>2</sub> was laid under the crucible to protect the furnace against any melt spills. The fully melted samples were held at 1500 °C at 1 atm for 2 h. Next, they were removed rapidly from the furnace, and the melt was poured from the thin-walled Pt synthesis crucible directly into a Pt<sub>80</sub>Rh<sub>20</sub> viscometry measurement crucible (2.54 cm inner diameter, 5.08 cm inner height, and 1 mm wall thickness), leaving around 5 mm space at the top of the crucible to accommodate the volume of insertion of a viscometry spindle. Two spindles were employed in the present study: a high-viscosity spindle (“Pthv,” 3 mm diameter, 43 mm length) and a low-viscosity spindle (“Ptlv,” 14.4 mm diameter, 33.2 mm length). The high-viscosity spindle was only used for the TUBS-T simulant, while the low-viscosity spindle was used for all other melts. Both were tapered 45° at the top and bottom to reduce edge effects (Dingwell 1986, 1989; Dingwell and Virgo 1988).

The filled viscometry crucible was transferred directly to a bottom-loading viscometry furnace (Deltech) operating at 1500 °C in air with Superkanthal heating elements and electronic temperature and power management through in-house viscometer and temperature control software. The viscometry crucible sits in a cradle machined from a mullite-based ceramic brick. The viscometry furnace is operated with a Type B (Pt<sub>70</sub>Rh<sub>30</sub>/Pt<sub>94</sub>Rh<sub>6</sub>) thermocouple, which resides within an Al<sub>2</sub>O<sub>3</sub> sheath. Sample temperature is calibrated by inserting a Pt-sheathed Type S (Pt/Pt<sub>90</sub>Rh<sub>10</sub>) measuring thermocouple into the center of the melt in the viscometry crucible, and a temperature correction is obtained. The spindle(s) are suspended from a Brookfield DIII viscometry head (0.7187 mNm full-scale torque), which is controlled and recorded using our software. The samples were stirred for several hours at 1500 °C to remove potential bubbles and to ensure chemical homogeneity. The entire system was calibrated against the soda lime glass viscosity standard DGG-1 of the Deutsche Glastechnische Gesellschaft with an accurately known viscosity-temperature relationship (Meerlender 1974).

The viscometry experiments were initiated at 1500 °C (held for 2 h) and then at successive 25 °C steps of decreasing temperature. This temperature sequence was terminated either by melt crystallization (as indicated by a substantial upward drift of torque at constant temperature) or viscometer instrument limits. By systematic software-driven halving of the viscometer speed upon the obtainment of 100% torque (full-scale reading), a rotation speed over a range of 40 to 0.1 rpm was varied. This, combined with the two spindles available, yields a dynamic range of 6 log units of viscosity determinations from 10<sup>2</sup>–10<sup>5</sup> Pa s. The dynamic viscosities were calculated by:

$$\log(\eta) = \log(m \cdot M + n)$$

where  $m$  and  $n$  are parameters depending on the rpm and spindle used, and  $M$  is the torque of the spindle. The calibration data of the Ptlv and Pthv spindles are shown in Table 1. All torque values lower than 10% and higher than 90% were omitted from the data set.

Following viscosity determinations, the spindle was retracted vertically from the crucible, quenched, and cleaned with concentrated hydrofluoric acid. The crucible was then removed from the furnace through the bottom door, and the melt

**TABLE 1.** Calibration parameters for the rotational viscometer concentric cylinder method

Ptlv (low-viscosity platinum)			Pthv (high-viscosity platinum)		
$\eta = m \cdot M + n$			$\eta = m \cdot M + n$		
Speed (rpm)	m	n	Speed (rpm)	m	n
40	0.0636	0.3999	40	1.205	-0.7067
20	0.1285	0.2824	20	2.4391	-2.0242
10	0.2569	0.2556	10	4.9607	-5.4643
5	0.5073	0.1167	5	10.242	-19.186
2.5	1.0261	-0.5969	2.5	20.858	-38.912
1.2	2.1444	-3.0637	1.2	44.43	-81.229
0.6	4.3789	-8.2706	0.6	90.176	-166.8
0.3	8.8122	-16.0980	0.3	179.59	-230.97
0.2	13.3390	-25.7250	0.2	295.68	-1157.4
0.1	27.6430	-70.1370	0.1	603.15	-1945.7
0.05	55.9680	-126.9400	0.05	1200.4	-2559.7

was poured from the hot crucible onto a stainless-steel plate for cooling. More viscous melts were left to cool in the viscometry crucible and then drilled from the crucible using diamond corer drills. Comparative measurements of the LRV-6 CAS and LRV-7/8 CAS compositions with similar protocols.

**Electron probe microanalysis (EPMA)**

The chemical compositions of the glass samples quenched from the viscosity measurements were determined using a Cameca SX-100 electron microprobe at the Department for Earth and Environmental Sciences, LMU Munich. To meet the requirements of the beam-sensitive glass, the following conditions were applied: 15 kV accelerating voltage, 5 nA beam current, and a 10 μm defocused beam. Calibration was performed using the following silicate and oxide standards: albite (Na, Si), orthoclase (K, Al), Fe<sub>2</sub>O<sub>3</sub> (Fe), periclase (Mg), wollastonite (Ca), rutile (Ti), bustamite (Mn), Cr<sub>2</sub>O<sub>3</sub> (Cr), apatite (P). Only results with totals between 98.5–101.5 wt% were accepted for evaluation. The relative error of the measurements is <2%.

**Magnetization measurements**

To detect and quantify possible magnetic crystals that formed during the quenching of melts, magnetic hysteresis has been measured on a 3900 MicroMag Vibrating Sample Magnetometer at the LMU München. This is performed by exposing a sample to an external magnetic field. Simultaneously, pickup coils measure the magnetic moment of the vibrating sample as a function of the external field and the magnetization history, which is called hysteresis loop.

**Differential scanning calorimetry**

The quenched glasses were investigated using differential scanning calorimetry (Netzsch DSC 404 C/3/F1 Pegasus) to determine their glass transition temperatures (*T<sub>g</sub>*). The calorimeter was calibrated for temperature and sensitivity using the melting temperature of indium and gold to within ±2 °C. For the measurement of the glass transition temperature, ~20 mg of the post-viscometry glass was transferred to a Pt crucible and then analyzed under a dynamic argon (20 cm<sup>3</sup>/min) atmosphere. To structurally relax the glasses, the samples were heated at 10 °C/min to about 50 °C above the peak of the glass transition temperature. The samples were then cooled to room temperature at 10 °C/min and again heated at 10 °C/min to obtain *T<sub>g</sub>* defined as the peak temperature. A cooling and heating rate of 10 K/min is sufficient to produce a homogeneous and practically crystal-free glass. Comparative measurements of the LRV-6 CAS and LRV-7/8 CAS compositions were performed with similar protocols.

**RESULTS**

**Chemical composition of remelted simulants**

Table 2 displays the chemical compositions of all simulants. The SO<sub>3</sub> and Cl concentrations in all samples are below the detection threshold of 0.13 and 0.06 wt%, respectively, and are therefore not mentioned. Notably, the measured Fe<sub>2</sub>O<sub>3</sub> (Total) and TiO<sub>2</sub> concentrations in the CAS samples differ from those reported by (Linke 2022). As the redox state was not measured in this work, the iron oxidation state was calculated using the model by Kress and Carmichael (1991). The melt’s structure can be quantified using parameters such as the number of non-bridging oxygen atoms per tetrahedron (NBO/T) (Mysen 1988) and the structure modifier content (SM) (Giordano and Dingwell 2003). NBO/T indicates the degree of polymerization within the melt, where higher values suggest less-polymerized structures. SM represents the sum of network modifier oxides in mol%:  $SM = x_{FeO} + x_{MgO} + x_{MnO} + x_{CaO} + x_{Na_2O} + x_{K_2O}$  (Giordano and Dingwell 2003). In the superliquidus range, the absolute viscosity increases with decreasing NBO/T. The calculated NBO/T and SM of the measured samples are displayed in Table 4. For all compositions, *f*<sub>O<sub>2</sub></sub> was adjusted to a fixed value of 0.21 (air), corresponding to atmospheric conditions.

**TABLE 2.** Chemical compositions measured by electron microprobe analysis of lunar simulants after viscometry measurements and original data by Linke (2022)

	TUBS-M		TUBS-T		TUBS-I		Station-5 CAS		LRV-6 CAS		LRV-7/8 CAS		b) Det. Lim.
	This work <sup>b</sup>	(Linke 2022)	This work <sup>b</sup>	(Linke 2022)	This work <sup>b</sup>	(Linke 2022)	This work <sup>b</sup>	(Linke 2022)	This work <sup>b</sup>	(Linke 2022)	This work <sup>b</sup>	(Linke 2022)	
SiO <sub>2</sub>	49.07	48.61	48.92	48.71	49.04	48.66	42.75	41.88	45.65	45.34	46.55	46.04	0.15
TiO <sub>2</sub>	2.47	2.29	0.17	0.12	1.38	1.205	10.62	8.18	5.43	4.12	5.58	4.33	0.13
Al <sub>2</sub> O <sub>3</sub>	13.55	13.29	30.16	30.33	21.66	21.805	13.14	12.92	20.26	20.20	15.18	15.12	0.13
Fe <sub>2</sub> O <sub>3</sub> (T)	10.93		1.46		6.44		12.65		7.70		10.83		0.22
FeO(T)		10.14		1.05		5.595		15.08		8.41		11.06	
Na <sub>2</sub> O	3.97	3.67	3.09	3.05	3.44	3.36	3.11	3.07	3.13	3.049	3.41	3.38	0.13
K <sub>2</sub> O	1.82	1.71	0.29	0.22	1.02	0.965	1.36	1.32	0.89	0.85	1.47	1.40	0.08
MgO	8.28	8.73	0.89	0.57	4.70	4.65	7.30	7.17	5.28	5.14	6.97	7.06	0.09
CaO	8.25	8.31	13.91	14.57	11.22	11.44	7.76	7.61	10.37	10.52	8.75	8.78	0.11
MnO	0.17	0.18	<dl.	0.02	0.10	0.10	0.28	0.19	0.17	0.11	0.21	0.16	0.06
Cr <sub>2</sub> O <sub>3</sub>	<dl.	0.04	<dl.	0.00	<dl.	0.02	0.17	0.03	0.10	0.02	0.09	0.03	0.07
P <sub>2</sub> O <sub>5</sub>	0.53	0.51	0.04	0.00	0.28	0.255	0.42	0.39	0.26	0.22	0.43	0.41	0.04
<b>Total</b>	<b>99.04</b>	<b>97.48</b>	<b>98.93</b>	<b>98.64</b>	<b>99.27</b>	<b>98.055</b>	<b>99.56</b>	<b>97.84</b>	<b>99.22</b>	<b>97.979</b>	<b>99.48</b>	<b>97.77</b>	
Fe <sub>2</sub> O <sub>3</sub> <sup>a</sup>	7.39	7.91	3.98	3.98	7.51	7.51	4.67	4.67	6.57	6.57	6.57	6.57	
FeO <sup>a</sup>	4.30	5.51	2.27	2.27	4.76	4.76	2.81	2.81	3.93	3.93	3.93	3.93	
Fe <sup>3+</sup> /Fe <sub>tot</sub> <sup>a</sup> (molar%)	0.61	0.62	0.62	0.61	0.61	0.59	0.59	0.60	0.60	0.60	0.60	0.60	
Fe <sup>2+</sup> /Fe <sub>tot</sub> <sup>a</sup> (molar%)	0.39	0.38	0.38	0.39	0.39	0.41	0.41	0.40	0.40	0.40	0.40	0.40	

Notes: The relative error for the electron microprobe analysis is <2%. All values given in wt%.

<sup>a</sup> Calculated using the method by Kress and Carmichael (1991).

<sup>b</sup> Detection limit (Det. Lim. applies for only the data of this work).

**TABLE 3.** High-temperature viscosity data

$T_{\text{real}}$ (°C)	10 000/ $T$ (K <sup>-1</sup> )	$\log_{10} \eta$ (Pa s)					
		TUBS-M	TUBS-T	TUBS-I	Station-5 CAS	LRV-6 CAS	LRV-7/8 CAS
1490	5.67	0.38	1.17	0.77	0.09	0.45	0.30
1465	5.75	0.45	1.30	0.86	0.14	0.53	0.36
1441	5.83	0.52	1.44	0.96	0.20	0.62	0.44
1417	5.92	0.60	1.58	1.07	0.26	0.71	0.51
1392	6.00	0.69	1.73	1.18	0.32		0.60
1368	6.09	0.78	1.88	1.29	0.39	0.90	0.69
1344	6.18	0.88		1.41	0.47	1.01	0.78
1319	6.28	0.98	2.20	1.54		1.13	0.88
1295	6.38					1.26	0.99
1271	6.48	1.21	2.57	1.82			
1246	6.58	1.33	2.77			1.52	
1222	6.69	1.46		2.13			
1198	6.80	1.60	3.21				
1173	6.91		3.46	2.50			
1149	7.03		3.72	2.70			
1125	7.15		4.00				
1100	7.28		4.32	3.15			
1076	7.41		4.66				
1051	7.55			3.66			

Note: The error is  $\pm \log 0.02$  Pa s.

### High-temperature viscosity

The high-temperature viscosities measured in air are presented in Table 3. All simulants investigated showed the characteristic decreasing viscosity of silicate melts with increasing temperature. The highland simulant TUBS-T has the highest viscosity values throughout the whole temperature range, which is expected to be due to the largest amount of network formers of all tested melts and results in low NBO/T and SM values.

### Glass homogeneity

Glass homogeneity was assessed using an electron probe microanalyzer (EPMA) with a backscattered electron (BSE) detector. This analysis revealed subtle heterogeneity in the form of possible phase separation in the post-viscometry samples of the Station-5 simulant. Due to the limited scope of this study, further exploration of this heterogeneity was not feasible. Consequently, only the high-temperature viscosity measurements for the Station-5 simulant were used, and differential scanning calorimetry (DSC) data of this sample were excluded from further investigation.

The homogeneity of the glasses was also assessed by rock magnetic analysis. Figure 1 shows the weight-normalized hysteresis loops of 6 samples after adjustment for dia-/paramagnetic contributions. An original hysteresis loop of a sample that contains only dia-/paramagnetic, and no ferromagnetic particles would look like a straight line with a specific slope. If ferromagnetic particles are present in the sample, their contribution will overlay the line in a limited field range until saturation is reached, and magnetization will develop linearly with the external field. By subtracting the linear slope of the high-field part of the original hysteresis loop from the complete curve, the corrected hysteresis loop, which only shows the ferromagnetic fraction, can be obtained. As an example, for sample LRV-7/8, in addition to the corrected hysteresis loop, the paramagnetic part is also shown, as well as the original curve that represents the sum of the para- and ferromagnetic curves.

Samples TUBS-M, TUBS-T, and TUBS-I exhibit no magnetic remanence. Their original hysteresis loops resemble

more or less two straight lines with positive slopes and no opening indicative of paramagnetic magnetic contributors. The shape of the corrected hysteresis loop, covering mainly the upper left and the lower right quadrant, is not a real physical result and excludes the presence of an amount of ferromagnetic crystals like (titano)magnetite or -hematite exceeding our detection limit, which we estimate below the order of 0.01 wt%.

Samples Station-5, LRV-6, and LRV-7/8 also show closed hysteresis loops; in the case of Station-5 curvature is not seen, indicating the absence of a significant amount of (titano)magnetite or -hematite. The hysteresis loops of LRV-6 and LRV-7/8 are also closed but curved. Therefore, it is assumed that these samples contain (titano)magnetite of a superparamagnetic grain size [ $< \sim 30$  nm; e.g., (Dunlop 1973; Dunlop and Özdemir 1997)]. LRV-6 and LRV-7/8 reach a saturation magnetization of 27.5 and 319.7 mAm<sup>2</sup>/kg, respectively. Using the value of 96 mAm<sup>2</sup>/kg for pure magnetite, this translates into a weight percentage of (titano)magnetite of 0.0287 and 0.333%, respectively, which can be considered insignificant.

### Glass transition temperature

The DSC measurements near the glass transition temperature are shown in Figure 2. The calorimetric glass transition temperature ( $T_{g,DSC}$ ) depends on the cooling rate of the samples in the DSC and, therefore, does not necessarily correspond to the viscometric glass transition temperature (here denoted as  $T_{g,12}$ ), defined as the temperature where the viscosity of the sample is at  $10^{12}$  Pa s (e.g., Richet 1984; Dingwell and Webb 1989; Webb and Dingwell 1990). The viscosity at the calorimetric glass transition temperature can be calculated by using the so-called "Shift Factor," which is dependent on the composition of the samples (Gottsmann et al. 2002). The "Shift Factor" has been determined by using the mol% of excess oxides ( $x$ ), which is a compositional parameter calculated by summation of molar percentages of network modifying oxides (FeO, MnO, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, H<sub>2</sub>O), from which the molar

TABLE 4. Chemical and VFT parameters of discussed compositions after viscosity measurement

	NBO/T <sup>a</sup>	SM <sup>b</sup> (mol%)	A <sub>VFT</sub> (Pa s)	Standard error <sup>c</sup>	B <sub>VFT</sub> (K)	Standard error <sup>c</sup>	C <sub>VFT</sub> (K)	Standard error <sup>c</sup>	T <sub>g,12</sub> <sup>c</sup> (K)	T <sub>g,DSC</sub> <sup>d</sup> (K)	Excess oxides(mol%)	K <sup>e</sup> (Pa K)	log η at T <sub>g,DSC</sub> (Pa s)	m <sub>12</sub> <sup>f</sup>
TUBS-M	0.53	32.22	-2.82	0.02	3349	27	711	1.6	937	962	20.20	9.79	10.57	61
TUBS-T	0.05	22.77	-3.97	0.02	5307	28	732	1.4	1064	1088	2.20	10.20	10.9	51
TUBS-H	0.26	27.47	-3.80	0.06	5150	73	631	3.8	957	987	11.68	9.89	10.67	46
Station-5 CAS	0.47	30.16									18.88			
LRV-6 CAS	0.30	28.00	-2.96	0.02	3458	27	782	1.6	977	1001	12.86	9.87	10.65	63
LRV-7/8 CAS	0.44	30.20	-2.53	0.02	2765	21	711	1.3	972	993	17.91	9.81	10.60	74

<sup>a</sup> Non-bridging oxygen per tetrahedra on a molar basis (Mysen 1988).

<sup>b</sup> Sum of structure modifier oxides (normalized mol%); SM = x<sub>FeO</sub> + x<sub>MgO</sub> + x<sub>MnO</sub> + x<sub>CaO</sub> + x<sub>Na<sub>2</sub>O</sub> + x<sub>K<sub>2</sub>O</sub> according to Giordano and Dingwell (2003).

<sup>c</sup> Viscometric glass transition temperature (here denoted as T<sub>g,12</sub>) at a viscosity of 10<sup>12</sup> Pa s derived from VFT-fit parameters.

<sup>d</sup> Corrected calorimetric glass transition temperature (peak).

<sup>e</sup> "Shift Factor" according to Gottsmann et al. (2002).

<sup>f</sup> Fragility index with T<sub>g,12</sub> at 10<sup>12</sup> Pa s. Calculated by m<sub>12</sub> = B<sub>VFT</sub>/[T<sub>g,12</sub>(1 - [C<sub>VFT</sub>/T<sub>g,12</sub>]<sup>2</sup>)].

percentages of network forming oxides (Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and Fe<sub>2</sub>O<sub>3</sub>) are subtracted (Gottsmann et al. 2002). The excess oxides were calculated using the iron oxidation state derived using the model by Kress and Carmichael (1991).

The viscosity at the calorimetric glass transition temperature can then be calculated by applying the relationship:

$$\log_{10} \eta(\text{at } T_{g,DSC}) = K - \log_{10}|q|$$

where η is the dynamic viscosity at the glass transition (here T<sub>g,DSC</sub>) in Pa s, K the calculated "Shift Factor," and q the cooling rate in °C/s (here: 0.17 °C/s). The "Shift Factor" can be calculated by:

$$K = 10.321 - 0.175 \ln(x)$$

where x is the mol% of excess oxides. Note that K in Gottsmann et al. (2002) is defined at the peak of the glass transition temperature.

The low-viscosity data (at superliquidus temperatures) can then be combined with these additional high-viscosity data to allow for a VFT-fit. The measured glass transition temperatures and derived viscosity values are reported in Table 4. During the heating and cooling cycle of the DSC, no exothermic (crystallization) peaks were detected, meaning that there is no change in the homogeneity created by these measurements.

### Viscosity-temperature relationship

By grouping the viscosity at the calorimetric glass transition temperature with the experimental high-temperature viscosity data, the non-Arrhenian temperature-dependent viscosity can be approximated with the Vogel-Fulcher-Tammann (VFT) equation (Vogel 1921; Fulcher 1925; Tammann and Hesse 1926):

$$\log_{10} \eta = A_{VFT} + \frac{B_{VFT}}{T - C_{VFT}}$$

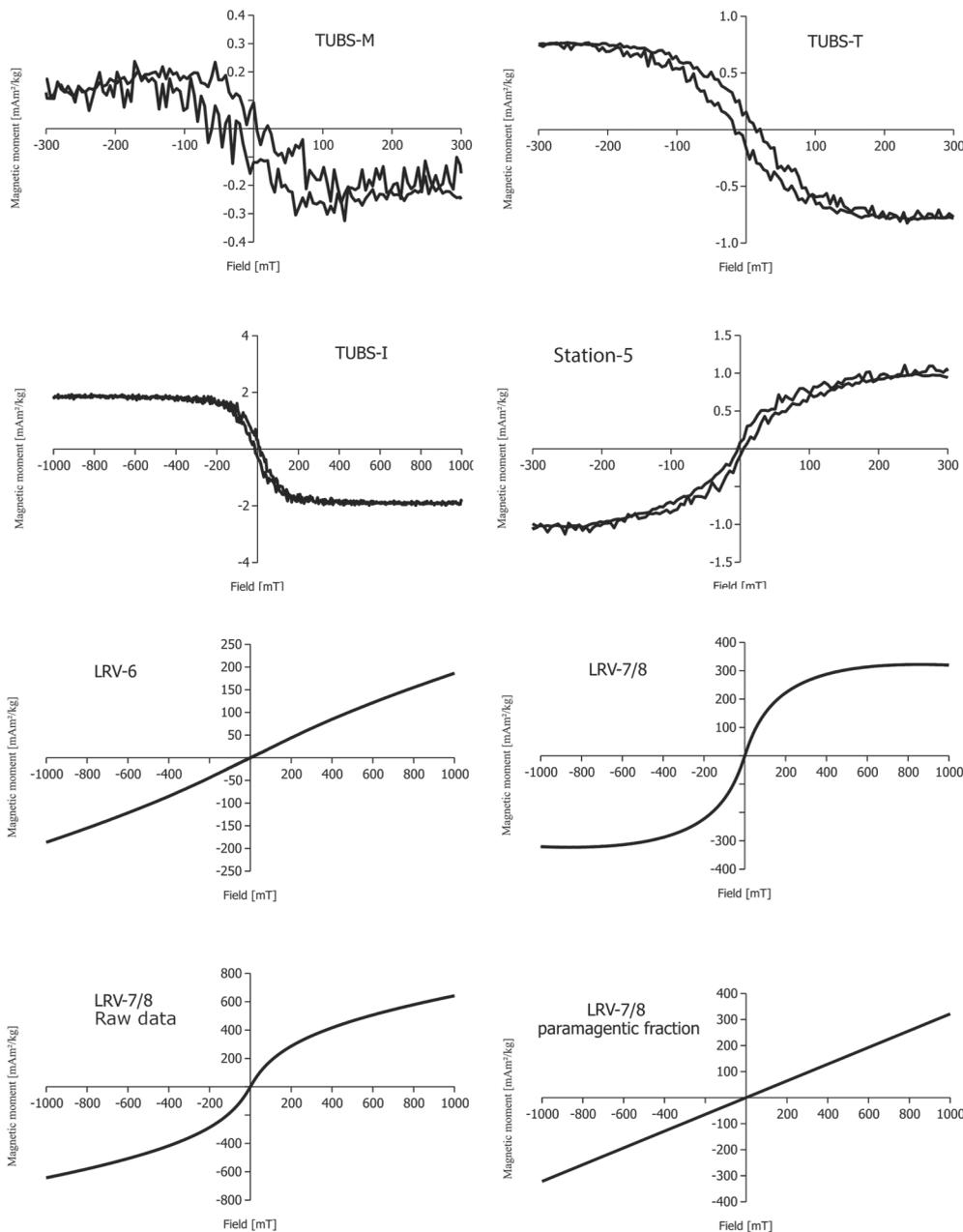
where the relationship between viscosity (η in Pa s) and temperature (T in K) depends on parameters A<sub>VFT</sub> (pre-exponential term), B<sub>VFT</sub>, and C<sub>VFT</sub> (Vogel temperature, sometimes denoted as T<sub>0</sub>) modeled from viscosity measurements in a wide range of geochemical compositions (Giordano et al. 2008). The VFT parameters were obtained by fitting the high-temperature viscosity data and the DSC-derived viscosity data at T<sub>g,DSC</sub> (peak) by the method of Gottsmann et al. (2002) to the Vogel-Fulcher-Tammann equation using nonlinear regression, ensuring an optimal representation of the temperature-dependent behavior of the viscosity.

The measured high-temperature viscosity values, the viscosity at the glass transition temperature, and the fitted viscosity curve are displayed in Figure 3. The corresponding VFT-parameters are shown in Table 4.

## DISCUSSION

### Fragility

The concept of fragility, first introduced by Angell (1991), plays a pivotal role in understanding the departure of a liquid's viscosity from an Arrhenian temperature dependence. This property is a measure of the departure from Arrhenian



**FIGURE 1.** Weight normalized corrected hysteresis loops of the quenched simulant samples (top six graphs) as well as raw data and paramagnetic fraction data of the LRV-7/8 sample (bottom two graphs).

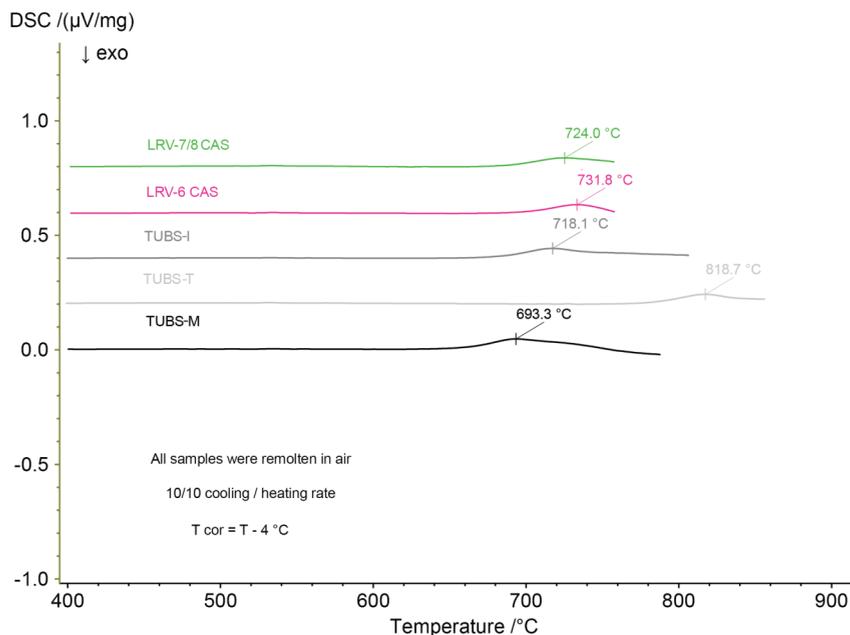
temperature dependence of viscosity. Fragility has been defined as the slope of the logarithmic viscosity curve at the viscometric glass transition temperature (here denoted as  $T_{g,12}$ ), when plotted on a reduced temperature scale. The fragility index ( $m$ ) can be calculated according to (Plazek and Ngai 1991; Böhmer and Angell 1992):

$$m = \left. \frac{d \log_{10} \eta}{d(T_{g,12}/T)} \right|_{T=T_{12}}$$

This parameter has been widely employed to differentiate various types of melts and has proven indispensable in this regard (Hess et al. 1996; Toplis et al. 1997; Avramov 2011; Dingwell et al. 2022). Glassmakers have used fragility as a concept that

helps to evaluate the time they have to shape the glass within the working range, which is typically between  $10^3$ – $10^{6.6}$  Pa s (Le Bourhis 2007; Zheng and Mauro 2017).

The degree of fragility within a melt is mostly influenced by its composition. As the degree of polymerization decreases, the fragility tends to increase (Hess et al. 1995; Giordano and Dingwell 2003; Giordano et al. 2009; Russell et al. 2022). Consequently, more fragile liquids exhibit heightened variations in viscosity with temperature changes, demonstrating low viscosity at high temperatures and high viscosity at low temperatures. On the other hand, stronger liquids exhibit higher viscosities at elevated temperatures than fragile liquids. These characteristics jointly serve to inhibit the nucleation



**FIGURE 2.** DSC measurements near the glass transition temperature of the tested lunar regolith simulants. Presented data are stacked for visualization with an offset of  $0.2 \mu\text{V/mg}$ . Data of TUBS-M not shifted.

and growth of crystal phases, thereby facilitating the process of glass formation of less-fragile melts (Kelton 2017). The fragility of the melts was calculated and is shown in Figure 4 with an Angell plot. All analyzed samples have high fragilities, ranging from 35 for the TUBS-I simulant up to 59 for the LRV-7/8 CAS.

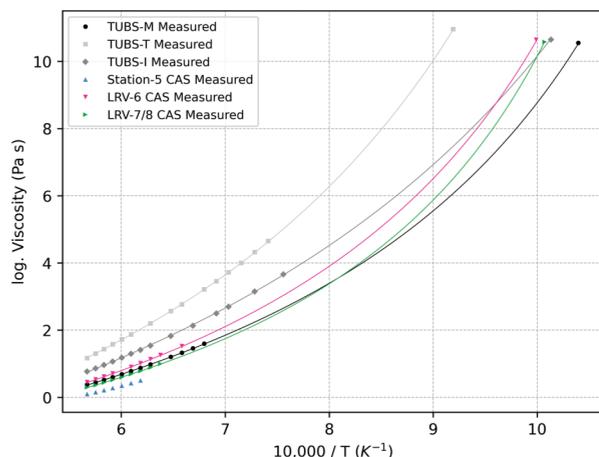
### Comparison with viscosity models

The measured viscosities of the regolith simulants were compared with the well-established viscosity models of Shaw (1972), Giordano et al. (2008) (hereafter GRD), Sehlke and Whittington (2016), and Duan (2014), as well as more recent models

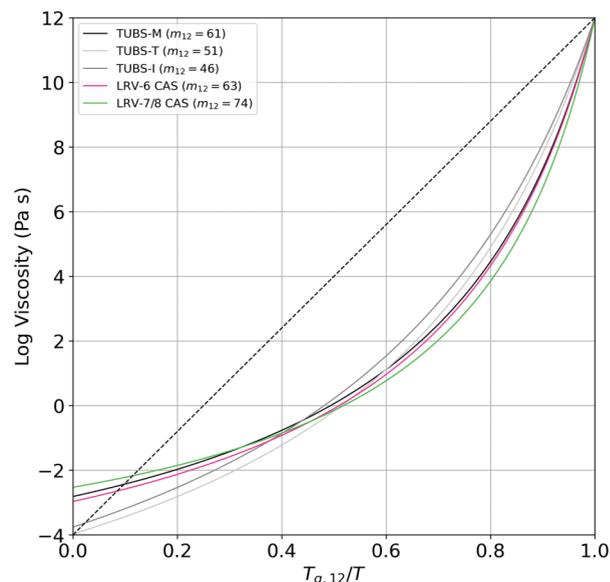
based on artificial neural networks: ViscCalc (Langhammer et al. 2022) and GlassNet (Cassar 2023).

The models were implemented as follows:

**Shaw (Shaw 1972):** The Shaw model predicts Arrhenian behavior, is based on Bottinga and Weill (1972) and is fitted on 2440 data points and compositions ranging from “lunar basalt” to “terrestrial rhyolites” and “hydrous granites of ternary minimum composition.” According to the original reference of the model, MnO and P<sub>2</sub>O<sub>5</sub> were excluded. Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are converted



**FIGURE 3.** Viscosity data of measured lunar regolith simulants. High-temperature viscosity data measured with concentric cylinder viscometer, low-temperature viscosity data derived from differential scanning calorimetry measurements. Solid line is the corresponding Vogel-Fulcher-Tammann equation.



**FIGURE 4.** Angell plot of the log viscosity as a function of the scaled inverse temperature.  $T_{g,12}$  is the temperature at which the shear viscosity reached  $10^{12}$  Pa s. The dashed line represents a strong liquid with ideal Arrhenian behavior.

to FeO and Al<sub>2</sub>O<sub>3</sub>, respectively, by doubling the moles of Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> as was done by Bottinga and Weill (1972). Total oxides (excluding MnO and P<sub>2</sub>O<sub>5</sub>) were normalized to 1 on a molar basis and used for further calculation of the mean slope, which represents the average contribution of each oxide's slope to the overall viscosity-temperature relationship. The dynamic viscosity can then be calculated (in Poise) by applying the formula:

$$\ln \eta = s \cdot \frac{10^4}{T} - 1.5 \cdot s - 6.4$$

where  $s$  is the characteristic slope for a given melt composition.

**GRD (Giordano et al. 2008):** The GRD model predicts VFT parameters and thus acknowledges the non-Arrhenian behavior of melts. It is fitted on 1774 experimental viscosity data points from 178 different melts ranging in composition from basanite through phonolite and trachyte, to dacite. All Fe<sub>2</sub>O<sub>3</sub> in this model is converted to FeO, resulting in:

$$\text{FeO}(T) = \text{Fe}_2\text{O}_3(T) \cdot 0.9$$

Total oxides were normalized to 1 on a molar basis and used for further calculation of the VFT parameters.

**Duan (Duan 2014):** The model by Duan incorporates pressure and the redox state for natural iron-bearing melts (from ultramafic, mafic to silicic). It is calibrated on ~200 experiments, measurements performed on 19 basaltic melt compositions, and relies on the VFT equation with a fixed "A" value of -4.753468 to capture the non-Arrhenian temperature-dependent viscosity behavior. To evaluate the model, a pressure of 1 bar was used. In cases where the iron oxidation state is not given, Duan used the method of Moretti (2005) to calculate the redox state as a function of oxygen fugacity, pressure, temperature, melt composition, and H<sub>2</sub>O content. In this work, the redox state was calculated using the model by Kress and Carmichael (1991).

**Sehlke and Whittington (Sehlke and Whittington 2016):** This model was developed to predict the viscosity of planetary melts and is fitted on 496 experimental data points from 20 anhydrous tholeiitic planetary basaltic melt compositions at 1 atm. Fifteen of these compositions represent known or estimated surface compositions of Mars, Mercury, the Moon, Io, and Vesta. The model performed well for previous lunar viscosity data and outperformed the GRD model for the tested planetary compositions. As this model was designed for a wide range of oxygen fugacities, the calculated redox state using the model Kress and Carmichael (1991) was used.

**ViscCalc (Langhammer et al. 2022):** The ViscCalc model was trained on 1603 data points of 50 anhydrous volcanic melts. It is based on the physically motivated MYEGA model (Mauro et al. 2009):

$$\log_{10} \eta(T) = \log_{10} \eta_{\infty} + \frac{T_g}{T} (12 - \log_{10} \eta_{\infty}) \cdot \exp \left[ \left( \frac{T_g}{T} - 1 \right) \left( \frac{m}{12 - \log_{10} \eta_{\infty}} - 1 \right) \right]$$

where  $\log_{10} \eta_{\infty}$  is the asymptotic viscosity at infinitely high temperature,  $m$  the fragility index, and  $T_g$  the glass transition temperature, which is defined at  $10^{12}$  Pa s for the MYEGA equation. Langhammer used the notation  $A = \log_{10} \eta_{\infty}$  with a fixed

value of -2.9. As the model differentiates between the redox state of iron, the values are calculated by using the Kress and Carmichael (1991) model.

**GlassNet (Cassar 2023):** This model is trained on more than 795 000 data points over 218 000 different glass compositions, implemented using the SciGlass database. Predictions are based on the MYEGA equation, considering the fragility index  $m$ , the glass transition temperature  $T_g$ , and the asymptotic viscosity  $A$ . To properly apply this model, the Kress and Carmichael (1991) redox state of iron was used.

The characteristic parameters needed to calculate the viscosity using the six tested viscosity models are shown in Table 5. To assess which model is most appropriate for modeling the viscosity of the lunar regolith simulants, the root mean square error (RMSE) was employed:

$$\text{RMSE} = \sqrt{\frac{\sum_{n=1}^N (\eta_{c,n} - \eta_{m,n})^2}{N}}$$

To obtain comparable values, only the seven highest-temperature viscosity points in the temperature range between 1344 and 1490 °C were considered in the RMSE calculation. The accuracy of the models at the glass transition temperature was compared by calculating the difference between the predicted viscosity at the measured glass transition temperature and the measured glass transition viscosity. The comparison of the viscosity measurements with the predictions based on the six models is shown in Figure 5 and Table 6. The comparison of only the high-temperature viscosity is displayed in Figure 6.

The Shaw model provides good high-temperature viscosity predictions, with an RMSE (superliquidus) ranging from 0.126 to 0.261 log units (Mean RMSE: 0.186 log units). However, it exhibits a significant deviation at the glass transition, with a difference of 5.26 to 6.26 log units [Mean  $\Delta\eta(T_g)$ : 5.86 log units], attributed to its underlying Arrhenian basis.

The GRD model yields moderate accuracy with an RMSE (superliquidus) spanning 0.071 to 0.893 log units [Mean RMSE: 0.451 log units] and a glass transition difference between 0.13 and 2.63 log units [Mean  $\Delta\eta(T_g)$ : 1.07 log units].

Duan's model proves to be the least efficient for the given compositions, showing an RMSE between 0.637 and 1.784 log units (Mean RMSE: 1.316 log units). The glass transition temperature is predicted with a difference of 0.79 to 2.02 log units [Mean  $\Delta\eta(T_g)$ : 1.34 log units].

Sehlke and Whittington's model performed the best, with a quite consistent RMSE in the high-temperature regime, ranging from 0.020 to 0.078 log units (Mean RMSE: 0.052 log units). However, it falls off in predicting the glass transition temperature, with a difference of 0.41 to 1.98 log units [Mean  $\Delta\eta(T_g)$ : 0.97 log units].

Langhammer's ViscCalc, an artificial neural network model, presents a moderate RMSE (superliquidus) between 0.057 and 0.397 log units (Mean RMSE: 0.243 log units). The difference in the glass transition temperature varies from 0.03 to 0.97 log units [Mean  $\Delta\eta(T_g)$ : 0.37 log units], making it the best model for predicting the glass transition temperature.

The GlassNet model, the most recent and based on artificial neural networks, exhibits an RMSE in the high-temperature

**TABLE 5.** Resulting parameters from the different viscosity models

	TUBS-M	TUBS-T	TUBS-I	Station-5 CAS	LRV-6 CAS	LRV-7/8 CAS
$s^a$	2.19	2.68	2.45	1.98	2.26	2.18
$A_{VFT}$ (Pa s) <sup>b</sup>	-4.55	-4.55	-4.55	-4.55	-4.55	-4.55
$B_{VFT}$ (K) <sup>b</sup>	5719	5361	5568	5781	5513	5722
$C_{VFT}$ (K) <sup>b</sup>	587	672	630	677	670	633
$T_g$ (K) <sup>c</sup>	945	1034	950	977	966	960
$m^c$	48	43	38	49	43	51
$A$ (log Pa s) <sup>d</sup>	-3.00	-3.20	-2.97	-2.51	-2.90	-2.72
$T_g$ (K) <sup>d</sup>	928	1048	994	935	983	949
$m^d$	41	43	43	43	42	42
$A_e$ (log Pa s) <sup>e</sup>	-3.34	-3.34	-3.34	-3.34	-3.34	-3.34
$B_e$ (J mol <sup>-1</sup> ) <sup>e</sup>	161 184	189 124	176 190	159 231	171 182	163 739
$S_{conf}^{conf}$ (mol <sup>-1</sup> K <sup>-1</sup> ) <sup>e</sup>	10.87	13.47	12.39	11.02	12.19	11.32
$C_{conf}^P$ (J mol <sup>-1</sup> K <sup>-1</sup> ) <sup>e</sup>	22.8	19.7	21.3	24.2	22.5	22.9
$T_g^{3R+}$ (K) <sup>e</sup>	927	1,053	987	900	958	932
$A_{VFT}$ (Pa s) <sup>f</sup>	-4.75	-4.75	-4.75	-4.75	-4.75	-4.75
$B_{VFT}$ (K) <sup>f</sup>	8162	8264	8209	8163	8194	8175
$C_{VFT}$ (K) <sup>f</sup>	491	504	497	491	495	492

<sup>a</sup> According to Shaw (1972).

<sup>b</sup> According to Giordano et al. (2008).

<sup>c</sup> According to Langhammer et al. (2022).

<sup>d</sup> According to Cassar (2023).

<sup>e</sup> According to Sehlke and Whittington (2016).

<sup>f</sup> According to Duan (2014).

regime ranging from 0.066 to 0.541 log units (Mean RMSE: 0.292 log units). It excels at the glass transition, displaying a viscosity difference between 0.06 and 1.66 log units [Mean  $\Delta\eta(T_g)$ : 0.63 log units].

Consequently, the model by Sehlke and Whittington (2016), which was calibrated specifically for planetary melts, also emerges as the most suitable choice for the accurate prediction of superliquidus viscosities within the tested melt compositions. It excels in providing precise estimates of high-temperature viscosities. The model by Langhammer et al. (2022) performed best at predicting the DSC-derived viscosity values at the glass transition temperature. However, it is imperative to underscore that none of the models demonstrate the requisite accuracy in anticipating viscosities at the glass transition temperature for all the tested samples. This limitation highlights the ongoing challenge of adequately modeling glass transition phenomena, signaling the need for further investigation and model refinement in this domain.

### Expected results on the Moon

Previously it has been shown that the oxidation state and structure of iron play a decisive role in the structure and properties of silicate melts (e.g., Mysen et al. 1984; Chevrel et al. 2013, 2014; Cukierman and Uhlmann 1974; Dingwell 1988, 1991; Dingwell and Virgo 1987, 1988; Giuli et al. 2011; Knipping et al. 2015; Lange and Carmichael 1987; Toplis and Carroll 1995; Toplis et al. 1994).

The Fe<sub>2</sub>O<sub>3</sub>-FeO ratio of silicate melts depends on temperature, chemical composition, and redox state, which is expected to be very low on the Moon (e.g., Richet and Bottinga 1986; Kress and Carmichael 1991; Jackson et al. 1993; Wilke et al. 2001, 2006; Giuli et al. 2003; Magnien et al. 2008). As shown by Cukierman and Uhlmann (1974), heavily reduced melts, like

those expected on the Moon, have a lower glass transition temperature than melts created using terrestrial materials but have a similar form of the variation of viscosity with temperature near the glass transition.

The influence of the oxidation state of highly reduced lunar regolith simulants should be investigated in more detail in further measurement campaigns to validate the applicability of the Earth-based viscosity models to actual lunar regolith. Real lunar regolith, in contrast to all existing lunar regolith simulants, is characterized by a significant reduction in oxygen content, critically impacting melt viscosity. Iron in this regolith is primarily found as ferrous iron (Fe<sup>2+</sup>), with a smaller fraction existing as metallic iron (Fe<sup>0</sup>) (e.g., Wiczorek 2006; Lucey et al. 2018). Iron oxides, specifically FeO and Fe<sub>2</sub>O<sub>3</sub>, serve as key influencers of melt viscosity, acting as network modifiers and network formers within the silicate melt structure (Liebske et al. 2003; Bouhifd et al. 2004; Stabile et al. 2016; Di Genova et al. 2017). FeO, functioning as a network modifier, disrupts the silicate network by breaking Si-O bonds, leading to a reduction in viscosity. Conversely, Fe<sub>2</sub>O<sub>3</sub>, acting as a network former, enhances the structured silicate network by contributing bridging oxygen atoms, consequently elevating melt viscosity. Experiments on synthetic silicate liquids at a fixed temperature at superliquidus and different oxygen fugacities performed by Chevrel et al. (2014) showed that the viscosity of more-reduced samples slightly decreases until Fe<sup>2+</sup>/Fe<sub>tot</sub> = 0.6 and increases slightly after a ratio of Fe<sup>2+</sup>/Fe<sub>tot</sub> > 0.6 is reached.

It was further shown that the glass transition temperature increases with a decreasing Fe<sup>2+</sup>/Fe<sub>tot</sub> ratio (Liebske et al. 2003; Bouhifd et al. 2004; Di Genova et al. 2017). Research on lunar iron-rich basalts by Cukierman and Uhlmann (1974) found that changing the Fe<sup>2+</sup>/Fe<sub>tot</sub> ratio from 0.94 to 0.76 had no impact on the viscosity near the glass transition. However, adjusting the

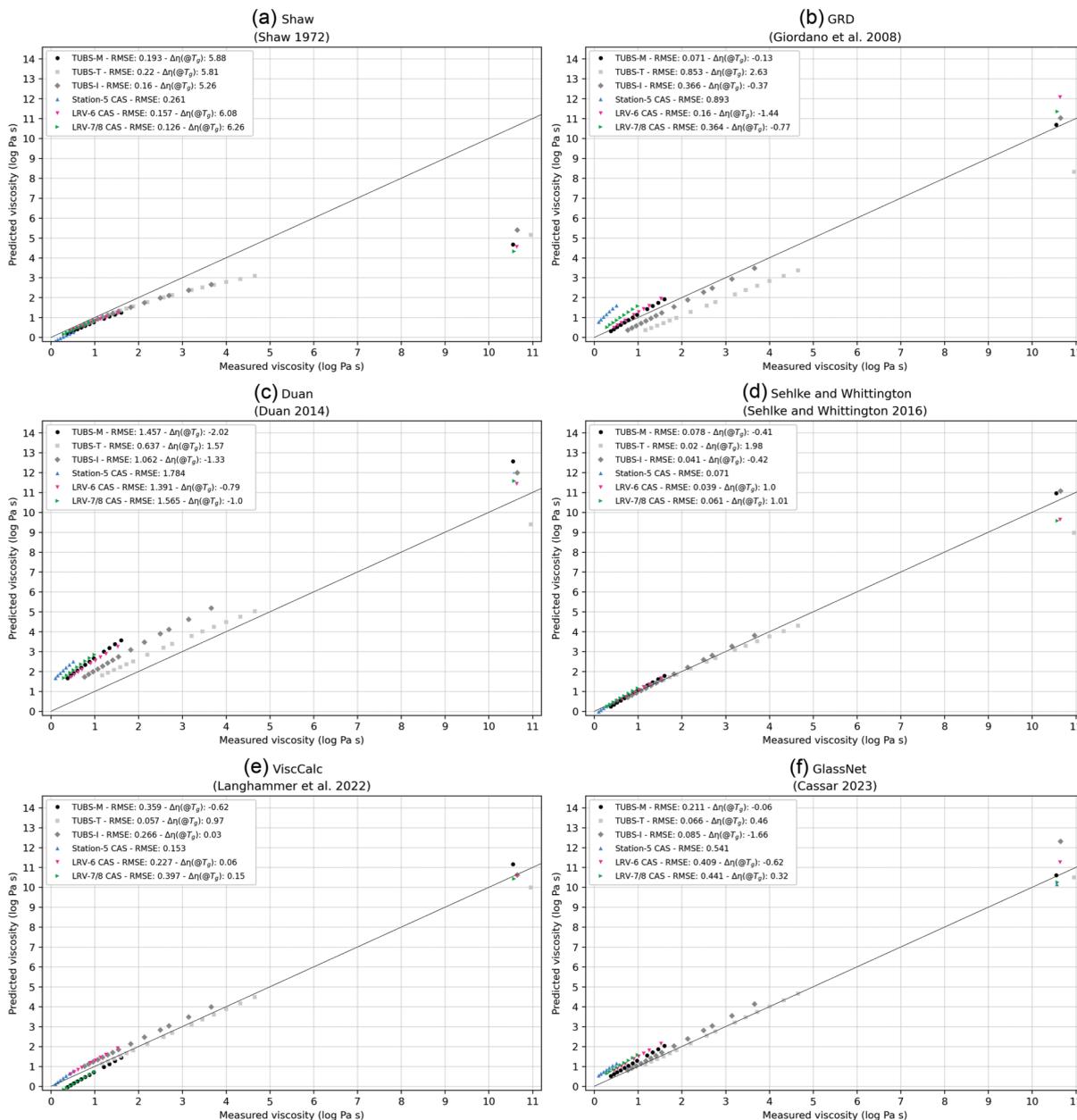


FIGURE 5. Comparison of measured viscosity and DSC-derived viscosity data against predicted viscosities. All values given in log units.

TABLE 6. Performance of the viscosity models

	Shaw		GRD		Duan		Sehlke and Whittington		Langhammer (ViscCalc)		Cassar (GlassNet)	
	RMSE	$\Delta\eta(T_g)$	RMSE	$\Delta\eta(T_g)$	RMSE	$\Delta\eta(T_g)$	RMSE	$\Delta\eta(T_g)$	RMSE	$\Delta\eta(T_g)$	RMSE	$\Delta\eta(T_g)$
TUBS-M	0.193	5.88	0.071	-0.13	1.457	-2.02	0.078	-0.41	0.359	-0.62	0.211	-0.06
TUBS-T	0.220	5.81	0.853	2.63	0.637	1.57	0.020	1.98	0.057	0.97	0.066	0.46
TUBS-I	0.160	5.26	0.366	-0.37	1.062	-1.33	0.041	-0.42	0.266	0.03	0.085	-1.66
Station-5 CAS	0.261		0.893		1.784		0.071		0.153		0.541	
LRV-6 CAS	0.157	6.08	0.160	-1.44	1.391	-0.79	0.039	1.00	0.227	0.06	0.409	-0.62
LRV-7/8 CAS	0.126	6.26	0.364	-0.77	1.565	-1.00	0.061	1.01	0.397	0.15	0.441	0.32
Mean	0.186	5.86	0.451	1.07	1.316	1.34	0.052	0.97	0.243	0.36	0.292	0.63

Notes: Only the seven highest temperature viscosity data points in the temperature range between 1344 and 1490 °C were considered in the RMSE calculation. All values given in log units.

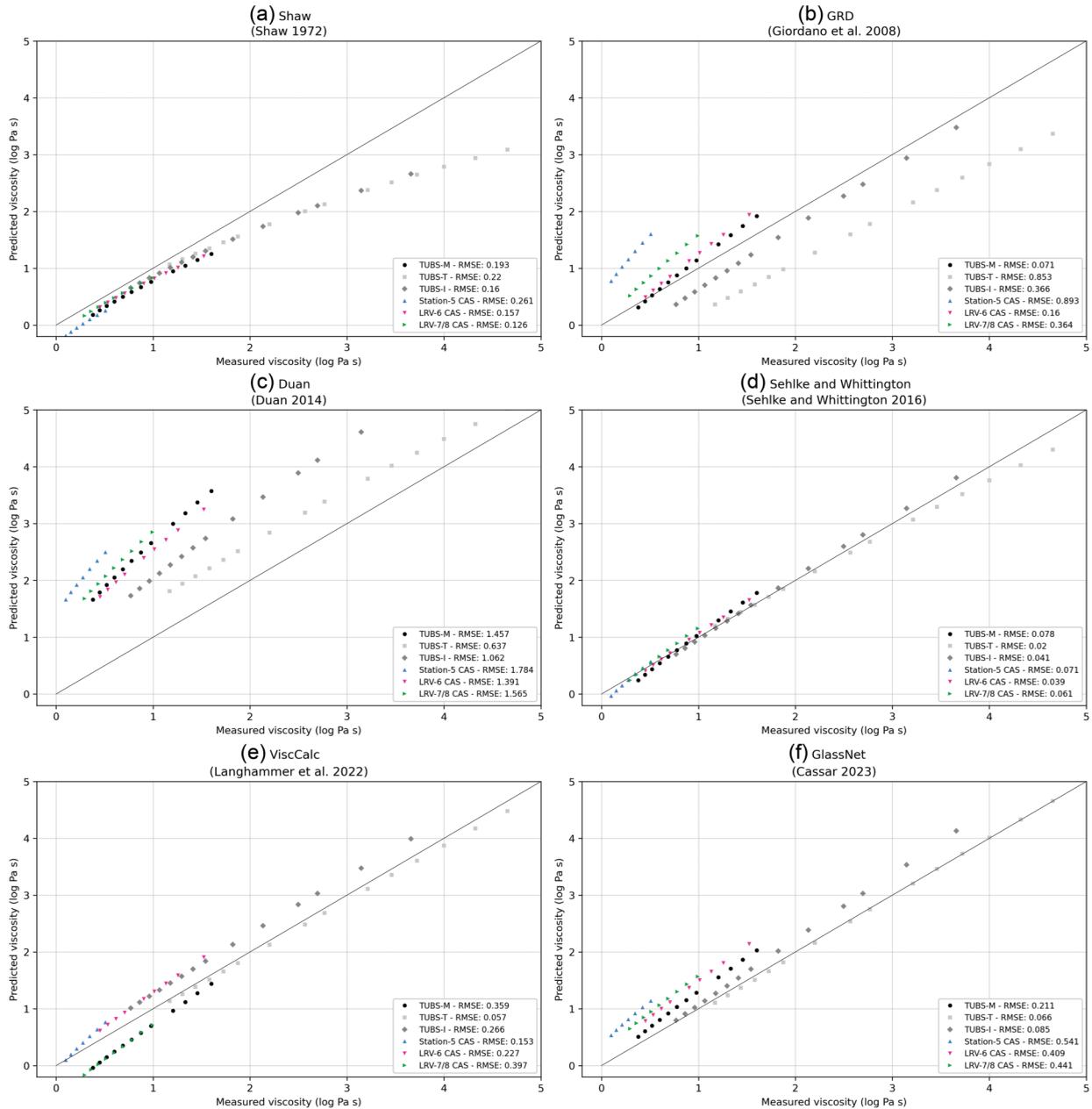


FIGURE 6. Comparison of measured viscosity data against predicted viscosities at high temperatures. All values given in log units.

ratio from 0.76 to 0.20 resulted in a remarkable increase in viscosity by approximately three orders of magnitude at a given temperature without altering the pattern of viscosity variation with temperature (Cukierman and Uhlmann 1974). These findings correlate well with research done by Chevrel et al. (2013), where viscosity and glass transition temperatures decrease for more-reduced melts until a threshold of  $Fe^{2+}/Fe_{tot} = 0.5$  is reached. The calculated iron oxidation state of roughly 0.6 for all samples brings the studied melts close to the threshold ( $Fe^{2+}/Fe_{tot} = 0.5$ ) at which the glass transition temperature no longer decreases with the redox state but remains constant (Chevrel et al. 2013).

### Cooling behavior

To investigate the influence of fragility and viscosity on the 3D printing process with molten lunar regolith, the cooling behavior of the different melts in vacuum needs to be understood. The cooling process in vacuum is mainly governed by radiative heat transfer, described by the differential equation:

$$\frac{dT}{dt} = -\frac{\epsilon\sigma A}{mc}(T^4 - T_{env}^4)$$

where  $\epsilon$  is the emissivity,  $\sigma$  is the Stefan-Boltzmann constant,  $A$  is the surface area,  $m$  is the mass,  $c$  is the specific heat capacity,  $T$  is the temperature of the object, and  $T_{env}$  is the ambient

temperature (here: 293.15 K). The melts to be used in the 3D printing process are expected to be at the working temperature (here: at  $10^3$  Pa s) and are defined to solidify at the temperature at the softening point ( $10^{6.6}$  Pa s), which is where liquids stop to deform under their own weight. To get the cooling time of the melts by radiation, the differential equation is solved for time:

$$t_{\text{cooling}} = -\frac{mc}{\epsilon\sigma A} \int_{T(\eta=10^3 \text{ Pa s})}^{T(\eta=10^{6.6} \text{ Pa s})} \frac{dT}{T^4 - T_{\text{env}}^4}$$

which can further be simplified by assuming that the variables are equal for each melt:

$$C = \int_{T(\eta=10^3 \text{ Pa s})}^{T(\eta=10^{6.6} \text{ Pa s})} \frac{dT}{T^4 - T_{\text{env}}^4}$$

where  $C$  is a normalized “Cooling Value” that indicates the time that a melt needs to reach the softening point and, therefore, does not flow under its own weight. While the “Cooling Value” does not depend on the emissivity and heat capacity, it is important to note that the emissivity and heat capacity heavily influence the cooling behavior of the melt and, therefore, need to be considered if these properties are known. Due to the faster radiative cooling at higher temperatures, it is evident that melts with a high temperature at the working point have lower “Cooling Values,” as they reach the softening point faster and are therefore more suitable for particular 3D printing applications where quick solidification is required. Based on the calculations, the highland TUBS-T simulants solidifies most quickly in a vacuum environment. The calculated “Cooling Values” for the tested simulants are displayed in Table 7.

## IMPLICATIONS

When determining the optimal lunar regolith for 3D printing, several factors related to the melting and solidification behavior of the melts must be considered. For technological purposes, melts with a low melting point are preferred, as they generally require less energy to melt and get fluid enough to be worked. Operating at lower temperatures enables the usage of a wider diversity of materials for the hot zone of the furnace and reduces wear and tear, minimizing the need to repair after longer use. This argument favors the use of mare regolith over highland regolith from a technical point of view.

In terms of fragility, more fragile melts may be preferred as the viscosity changes faster in the high-temperature and working range regime, meaning that fragile melts reach the softening point

faster and, therefore, freeze their shape faster than strong glasses, which is beneficial for extrusion-based 3D printing applications, rendering fragile melts more suitable in this regard. Stronger melts need a higher change of temperature to get from the melting point to the softening point and less temperature change to get from the softening point to the glass transition than fragile melts. Since most of the tested melts have fragility values within a similar range, the cooling rates of the melts are expected to have a greater influence on the solidification than the fragility. Melts that require higher temperatures to reach their working point can provide benefits in terms of cooling behavior. Since radiative heat transfer is proportional to the fourth power of the temperature, higher-temperature melts cool more rapidly, leading to a faster increase in viscosity. These properties are advantageous in processes that demand rapid solidification of the melt and where energy consumption is not a primary concern.

As shown by Cukierman and Uhlmann (1974), reduced melts have a lower glass transition temperature while maintaining the viscosity-temperature variation pattern. On the other hand, reduced melts have lower viscosities than oxidized melts (Cukierman and Uhlmann 1974; Dingwell and Virgo 1987; Liebske et al. 2003; Bouhifd et al. 2004; Chevrel et al. 2013, 2014; Di Genova et al. 2017), reducing the required energy to reach the working temperature. The lower glass transition temperature in reduced melts could also facilitate the annealing of 3D-printed structures, as cooling through radiation at decreased temperatures is reduced, keeping the glass in the annealing range for a longer period. This indicates that reduced melts, which would be created on the Moon, may even be more suitable for extrusion-based, 3D printing processes with molten lunar regolith than the highly oxidized Earth-found materials.

The significant variability in the viscosities of lunar regolith simulants is a key factor in site selection, especially when using processes that utilize molten lunar regolith. On one hand, the lower viscosities expected of regolith in the lunar mare areas will produce melts that require lower temperatures to achieve the desired viscosity compared to the regolith of the lunar highlands. On the other hand, highland regolith requires higher temperatures to reach the desired viscosity, which results in faster cooling to the softening point, allowing it to retain its shape more quickly. The choice of feedstock material will depend on the application, as different conditions and requirements may favor different material properties. For extrusion-based processes, the authors recommend using a mare regolith simulant due to its lower temperature requirements and higher fragility. Highland regolith simulants are not generally worse, as their advantage of faster cooling to the softening point, as indicated by a low “Cooling Value” value, may offer a major advantage over mare simulants. Since the cooling behavior and crystallization of the melt are decisive for the material properties of the solidified bodies, these aspects should be investigated in more detail in future work. Given the planned Artemis Base Camp at the lunar south pole (Creech et al. 2022), the highland regolith present in that region appears to be a well-suited option for additive manufacturing using molten lunar regolith, assuming that the higher energy requirements can be managed.

The influence of heavily reduced regolith melts on the Moon requires further investigation. The reduction in viscosity could

**TABLE 7.** Comparison of the “Cooling Value” of the different simulants between the liquidus temperature and the softening temperature, calculated by using the VFT parameters

Simulant name	Working temperature <sup>a</sup> in K (at $\eta = 10^3$ Pa s)	Softening temperature <sup>a</sup> in K (at $\eta = 10^{6.6}$ Pa s)	“Cooling Value” <sup>b</sup> in $1/T^4$
TUBS-M	1286	1067	$118.72 \times 10^{-12}$
TUBS-T	1493	1234	$77.44 \times 10^{-12}$
TUBS-I	1388	1125	$109.14 \times 10^{-12}$
LRV-6 CAS	1362	1108	$91.21 \times 10^{-12}$
LRV-7/8 CAS	1282	1085	$103.31 \times 10^{-12}$

<sup>a</sup> Calculated using the VFT parameters.

<sup>b</sup> Calculated by  $C = \int_{T(\eta=10^3 \text{ Pa s})}^{T(\eta=10^{6.6} \text{ Pa s})} \frac{dT}{T^4 - T_{\text{env}}^4}$ .

be advantageous not only for energy efficiency but also for allowing the use of lower-cost materials in the furnace, such as heaters, crucibles, and insulation, due to the reduced temperature requirements. Additionally, the resulting lower glass transition temperature of reduced melts may aid in the annealing of the molten regolith. However, it also increases the cooling time to the softening point, where the melt no longer deforms under its own weight.

The model by Sehlke and Whittington (2016) is recommended for predicting the high-temperature viscosity of lunar regolith in additive manufacturing applications. The study revealed that current models fail to accurately predict viscosities at the glass transition temperature, underscoring the necessity for additional research in this domain.

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