VACUUM SINTERING OF HIGHLAND SIMULANT CSM-LHT-1G

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Controlled Vacuum sintering of Highland Simulant Project Overview

Sintering of lunar regolith is a process of interest to consolidate the ubiquitous granular material in durable structures on the lunar surface such as platforms, landing/launch pads, roads, and foundations for long-term robotic and human activities.

- Directed Energy input techniques (solar, laser, microwave) are promising but local heating effects are difficult to measure in real-time

- Many publications do not report quantitative assessment of the effects of processing conditions on the strength properties of the sintered products.

- The work presented is an investigation into such effects on a carefully selected simulant, CSM-LHT-1G prepared for high temperature processing to eliminate undesired components that would not be present from lunar materials.

- Controlled heat input experiments under vacuum were conducted to identify the relevant factors that dominate the properties and the quality of the sintered product.

Lunar Mare Simulants – Sintering

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- **Air sintering**
  - Hawaiian basalts sintered at PISCES produced strong, homogeneous materials when balance in mineral compositions exist as detailed below.
  - Air sintering leads to formation of iron and magnesium oxide layers on mineral grains resulting in higher processing temperature than in vacuum or reduced atmosphere
- **Vacuum sintering**
  - FJS-1: Observations show a decrease of 100 °C in processing temperature of FJS-1 basalt to obtain similar densification and compressive strength than in air (Hoshino, 2016). Large (210 mm x 100 mm x 10 mm) samples processed at 4 x 10^{-4} torr. Best result obtained at ~ 1000 °C (Zhang 2021 reports Tm of 1100-1250 °C)
  - JSC-1A: Vacuum sintering obtained at lower temperatures than in air with higher compressive strength (152 Mpa vs. 98 Mpa) by Meurisse, 2017. Sintering T was 1100 °C in vac. and 1125 °C in air. Samples were pressed at 255 Mpa before process.
- **Observed effects of mineralogy (K. Edison 2021, Sintering of Hawaiian basalts in air)**
  - Olivine in excess of 10 wt.% decreases cohesion of the sintered product (used in sand casting of metal because of high mp)
  - Glass content 10–15 wt.% yielded best results (20-60% in lunar regolith).
  - Plagioclase content 40-50 wt.% yielded best results.
- **Variations in chemical composition and implications for vacuum processing**
  - Vacuum processing avoids the formation of oxide layers at grain boundaries by Fe and Mg from Olivine that reduce grain bonding in air. This enables sintering at lower temperature in vacuum for similar strength (Meurisse, 2017 and Hoshino, 2016)
  - Significant grain growth of Olivine and Plagioclase in vacuum-sintered JSC-1A from 1µm (original simulant) to 50 µm (sintered) during sintering at 1100 °C (Meurisse, 2017).
  - Presence of Albite instead of Anorthite in DNA-1 basaltic simulant result in higher sintering temperatures than JSC-!A in vacuum for similar strength (Meurisse, 2017) showing the importance for simulants to avoid replacing lunar Pg endmember Anorthite with lower %An minerals.

Lunar Highland Simulants - Sintering

- **Air sintering**
  - NU-LHT-2M: Samples processed at 1200 °C and cooled at 1 °C/min were weakly sintered (88% open porosity). (Matyas, 2011)
  - NU-LHT-2M: Samples processed at 1250 °C and 1300 °C and cooled at 1 °C/min were more fully sintered (5.3-5.6% open porosity). (Matyas, 2011)

- **Vacuum sintering**
  - NU-LHT-2M: low open porosity sintered samples (5-6%) obtained at 1250 °C cooled in vacuum (Engelschiøn, 2020).
  - NU-LHT-2M: Sintered samples of low-medium open porosity (~10%) processed to 1300 °C. Heat rate 5 °C/min. (Matyas, 2011)

- **Observed effects of mineralogy**
  - NU-LHT-2M and -4M materials contain ~ 35 wt.% as glass made from Stillwater "mill sand" and should be selected for sintering experiments over NU-LHT-3M that does not contain added glass. The -3M material is fully crystallized and provide a reference melting point (see DSC data).

- **Variations in chemical composition and implications for vacuum processing**
  - Similar to findings for mare simulants

Implications for design of experiments

Sintered samples in vacuum

- Processing pressure (vacuum level)
  - Affects sublimation behavior of Na_2O, K_2O below sintering temperatures – these oxides are likely to evolve from the glass phase when it softens, melts and devitrifies.
    - JSC-1A (~3% Na_2O; ~0.8% K_2O)
    - NU-LHT (~1.8% Na_2O; ~ppm K_2O – similar to lunar materials)
  - Na_2O: m.p. 1132°°C, sublimates near 400 °C at 10^{-2} Torr: Potential contribution to voids, reduced deposits of Na, lowering effect on glass melting point
  - K_2O: m.p. 740°°C, sublimates near 300 °C at 10^{-2} Torr: Potential contribution to voids, reduced deposits of K
- Processing temperature
  - JSC-1A (Meurisse 2017) or -1AF (Matyas 2011)
    - Final sintering T and soak time: 1100 °C (Meurisse 2017) / 15-30 min (Farries 2021)
    - Heating rate: 5 °C/min (Matyas 2011); 6.6 °C/min (400 °C/h) (Meurisse 2017)
    - Cooling rate: 1 °C/min (Matyas 2011) ; 6.6 °C/min (400 °C/h) (Meurisse 2017)
  - NU-LHT-2M (Matyas 2011)
    - Final sintering T and soak time: 1250 °C (Matyas 2011) / 15-30 min (Farries 2021)
    - Heating rate: 5 °C/min (Matyas 2011)
    - Cooling rate: 1 °C/min (Matyas 2011)
- Mineralogy of simulants
  - JSC-1A
    - Higher Olivine content (12%) than lunar mare materials (Apollo 11 & 12 range is 2-10%) may result in more friable sintered product than what may be possible with mare basalts
  - NU-LHT-2M
    - Higher in Olivine (~ 5.8%) than lunar highland materials (A16-64001/2: 0.8-0.9% Olivine). Less than the 10% limit reported in sintering results of Hawaiian basalts
    - Iron content is high compared to polar materials
    - Glass content lower (~ 35%) compared to lunar highland materials (A16-64001/2: 44-46% glass) but -2M material constitutes the best representation of A16 samples for sintering tests.

Simulant selection – CSM-LHT-1G B1

CSM-LHT-1 lunar highland simulant produced by Colorado School of Mines (K. Cannon).
Original mineralogical composition of CSM-LHT-1 (70 wt.% Greenspar, 30 wt.% Merriam Crater basalt)

CSM-LHT-1G

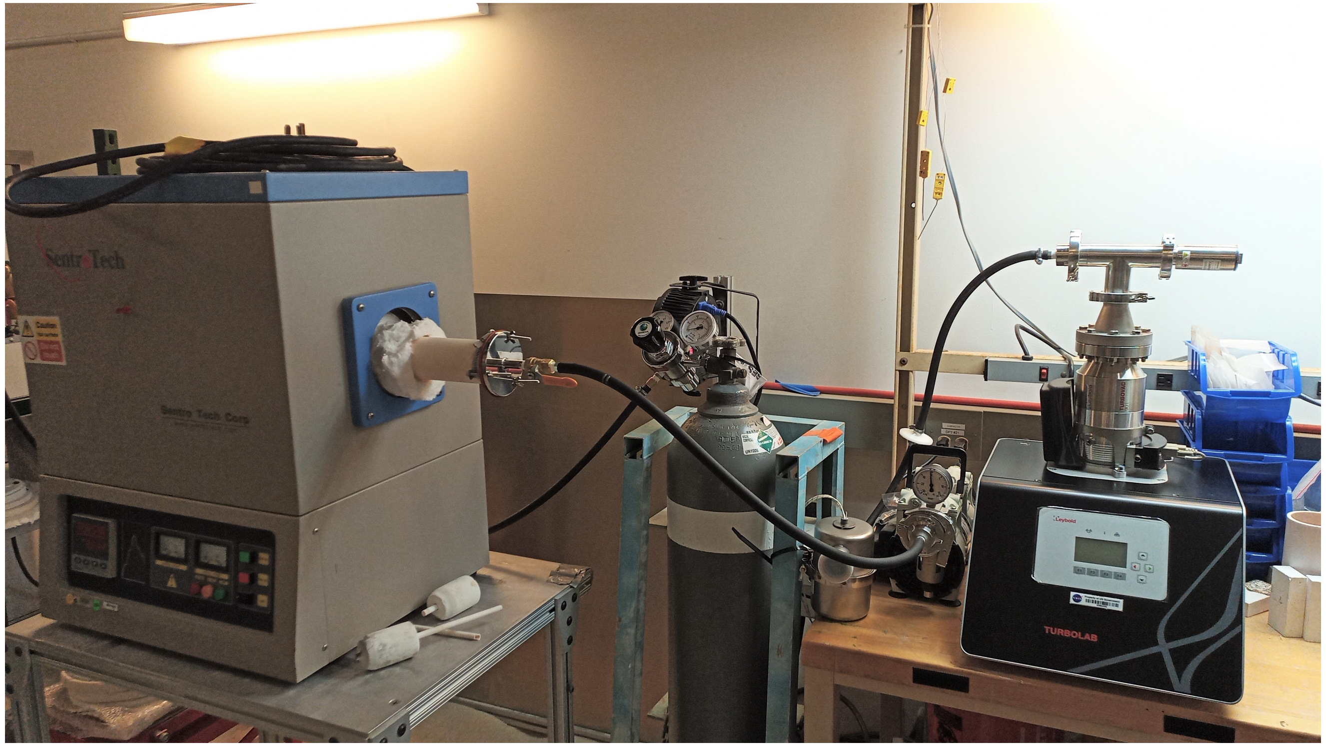
modified with the addition of NU-LHT-5M glass balanced with pyroxene (augite) to obtain a total 30 wt.% glass content.

Heat reduction treatment (B1)

- 5% H_2 - 95% Ar at 750 °C
- Remove as much as possible S, P

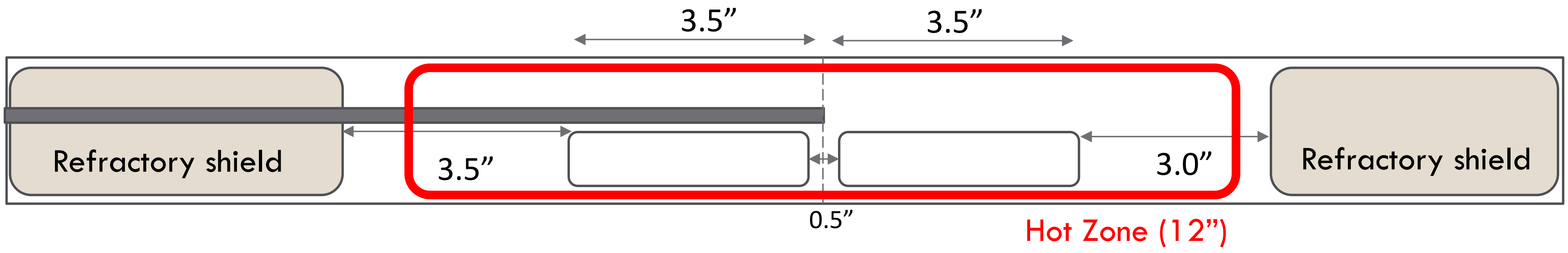
Equipment Used

- **Horizontal vacuum furnace ST-1700C**
  - Vacuum: 0.003 Torr with redesigned end caps
  - 2.5" ID Alumina tube
  - 12" long isothermal hot zone
  - No volatile measurement at this time
- **Crucibles**
  - Alumina boat (AdValueTech)
    - 10 cm x 4.5 cm x 1.9 cm
    - Sample mass: ~ 100 g



KSC Vacuum Furnace

Sintering Furnace Configuration



Summary

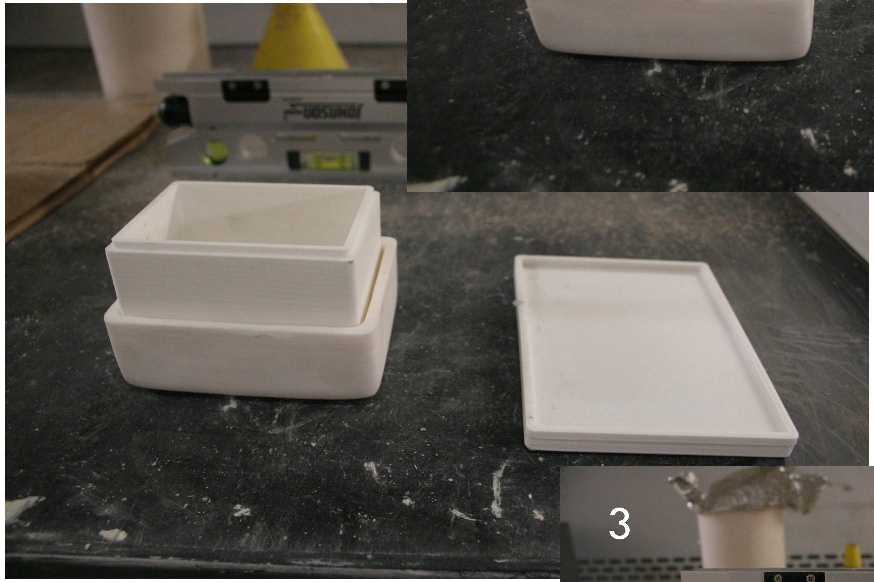
Sintering of CSM-LHT-1G B1

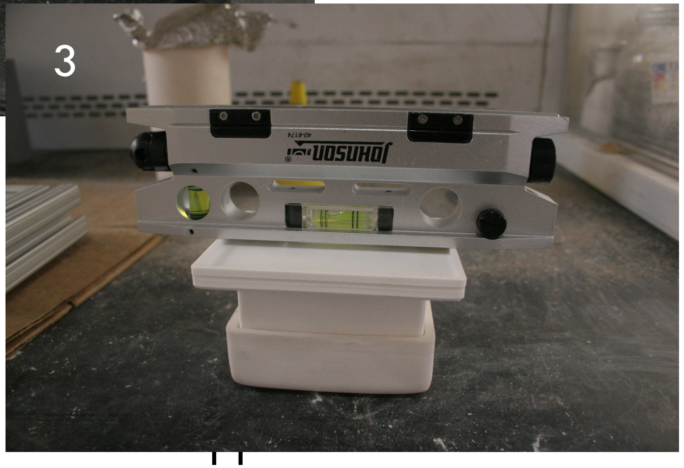
- Investigated temperature range: 1125 – 1230 ℃
- Samples processed in range 1125 – 1175 ℃ are sintered without exhibiting melting phase (outside observation)
- Samples processed in range 1190 – 1210 ℃ exhibit sintering and melting to various extent. Evidence of gas evolution breaking the surface.
- Samples processed beyond 1210 ℃ show extensive melting and gas evolution, voids formation.
- A temperature gradient of a few degrees in the hot zone is manifested by samples sintered at one end and melted at the other (near center of hot zone). Samples at 1190, 1200, 1210 ℃ exhibit this.

Regolith Compaction in Crucibles

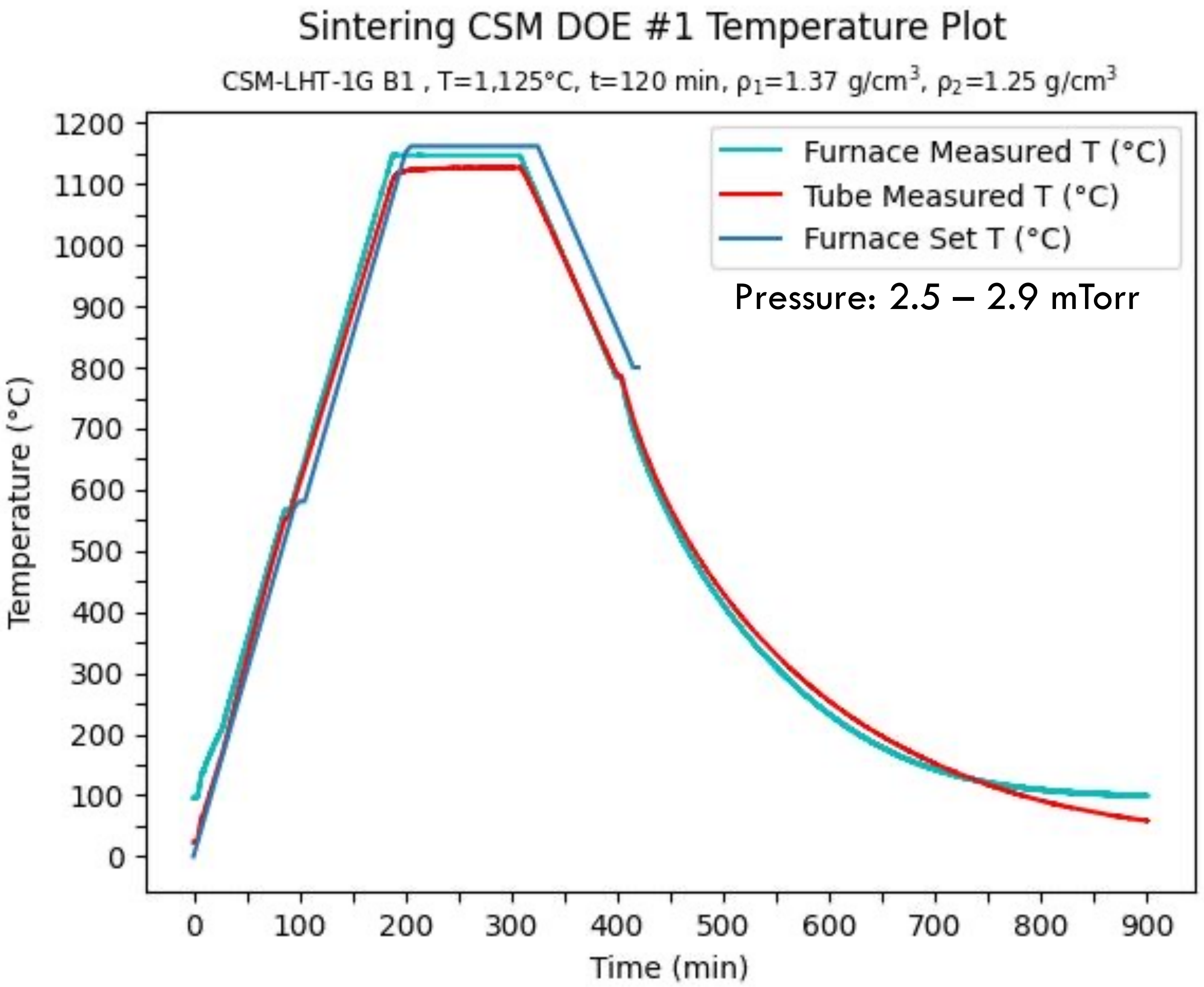
Objective: Create bed of regolith with a target density inside crucible reproducibly

- **3D printed boat with weight platform**
  - Boat
  - sides are within 1mm of crucible walls.
  - Boat can be filled with known masses
  - Has platform for large weights and for level verification
- Procedure
  - Fill crucible with regolith to within a few mm of top edge. Weigh.
  - Smooth regolith and use boat with level to create horizontal regolith surface, Read gradations on side of the boat to determine initial regolith volume.
  - Place known masses in or on top of boat
  - Place crucible and boat on stand with tapping plate
  - Tap a set number of times the underside of tapping plate with rubber mallet
  - Read gradations on side of the boat to determine final volume of regolith
  - Calculate final density





CSM-LHT-1G B1 Simulant Heating Profile

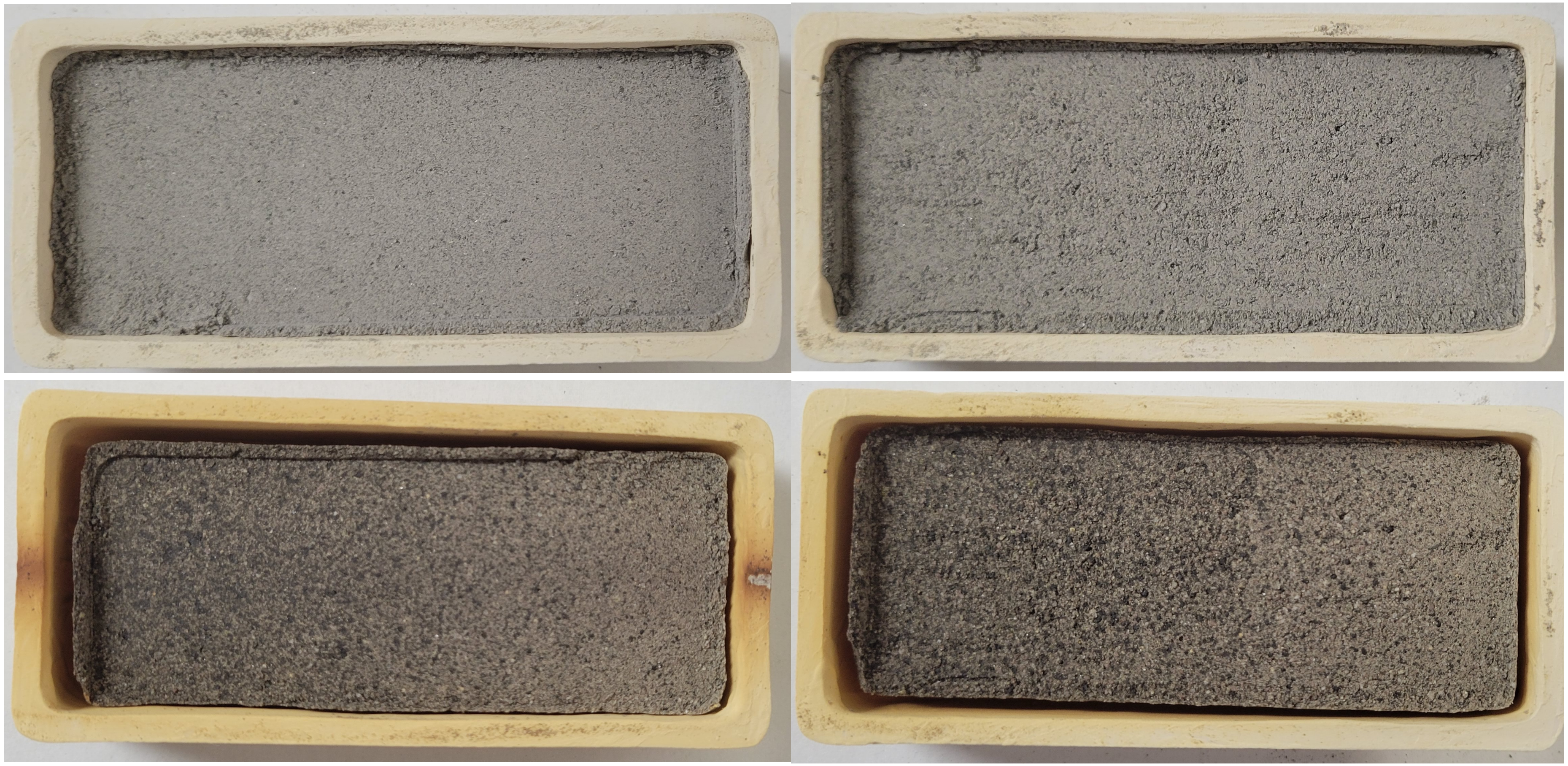


Aspect of Samples

Sample #1 **Sample #2**

Before

After



Effect of Temperature Gradient (Mare basalt JSC-1A)

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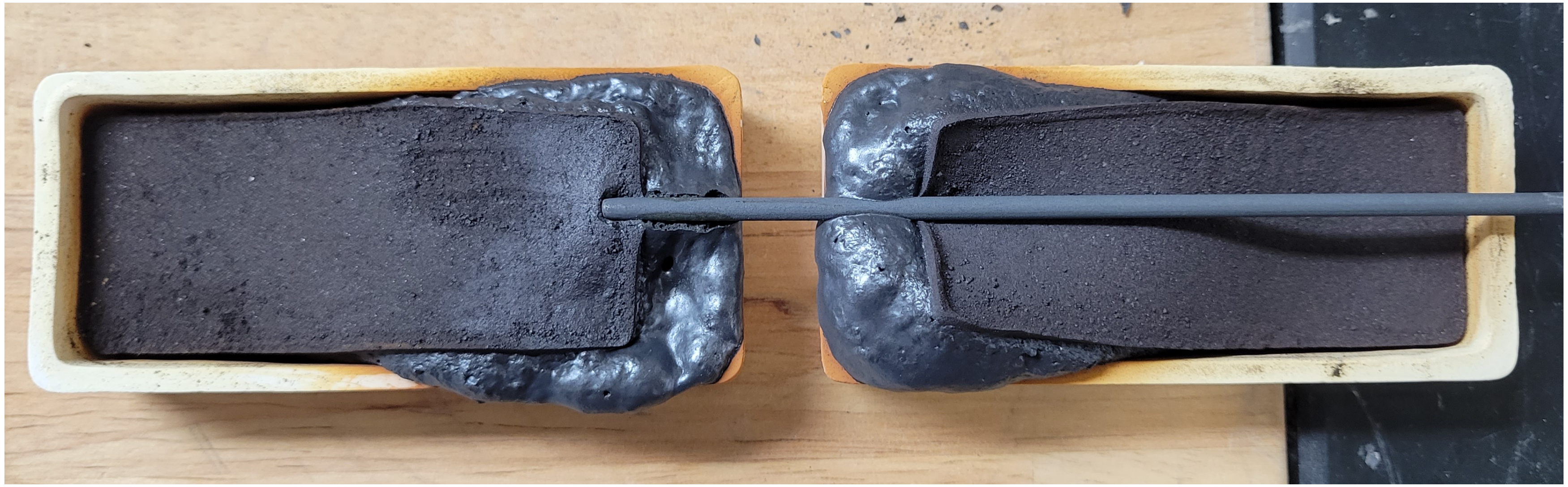
Sample #1

Sample #2

Before

After

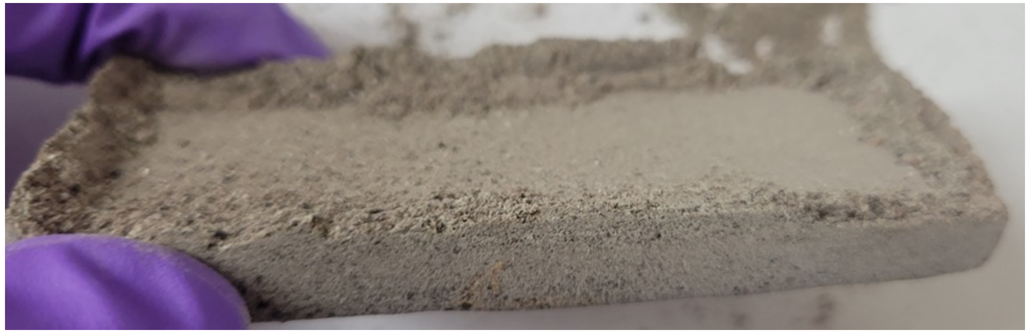


Sintered products

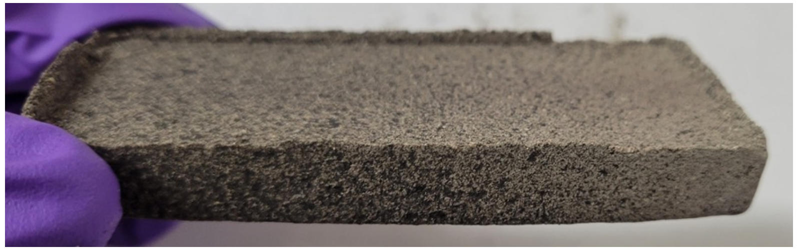
Highland simulant CSM-LHT-1G B1

1125 °C
Poured

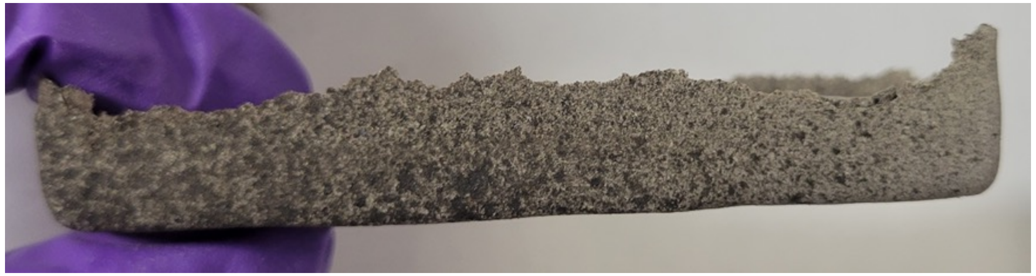
1125 °C
Compacted



1150 °C
Poured

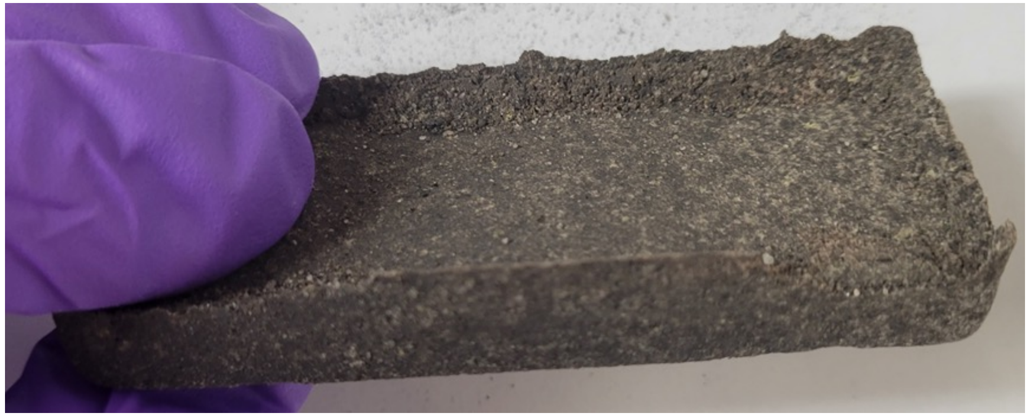


1150 °C
Compacted



1175 °C
Poured

1175 °C
Compacted



Sintered products
Highland simulant CSM-LHT-1G B1

1220 °C
Poured

Materials Testing

Materials Testing

- Specimen cutting by waterjet
- Materials testing by KSC Materials Analysis Branch
- Specimen dimensions dictated by ASTM C1161 and C1424

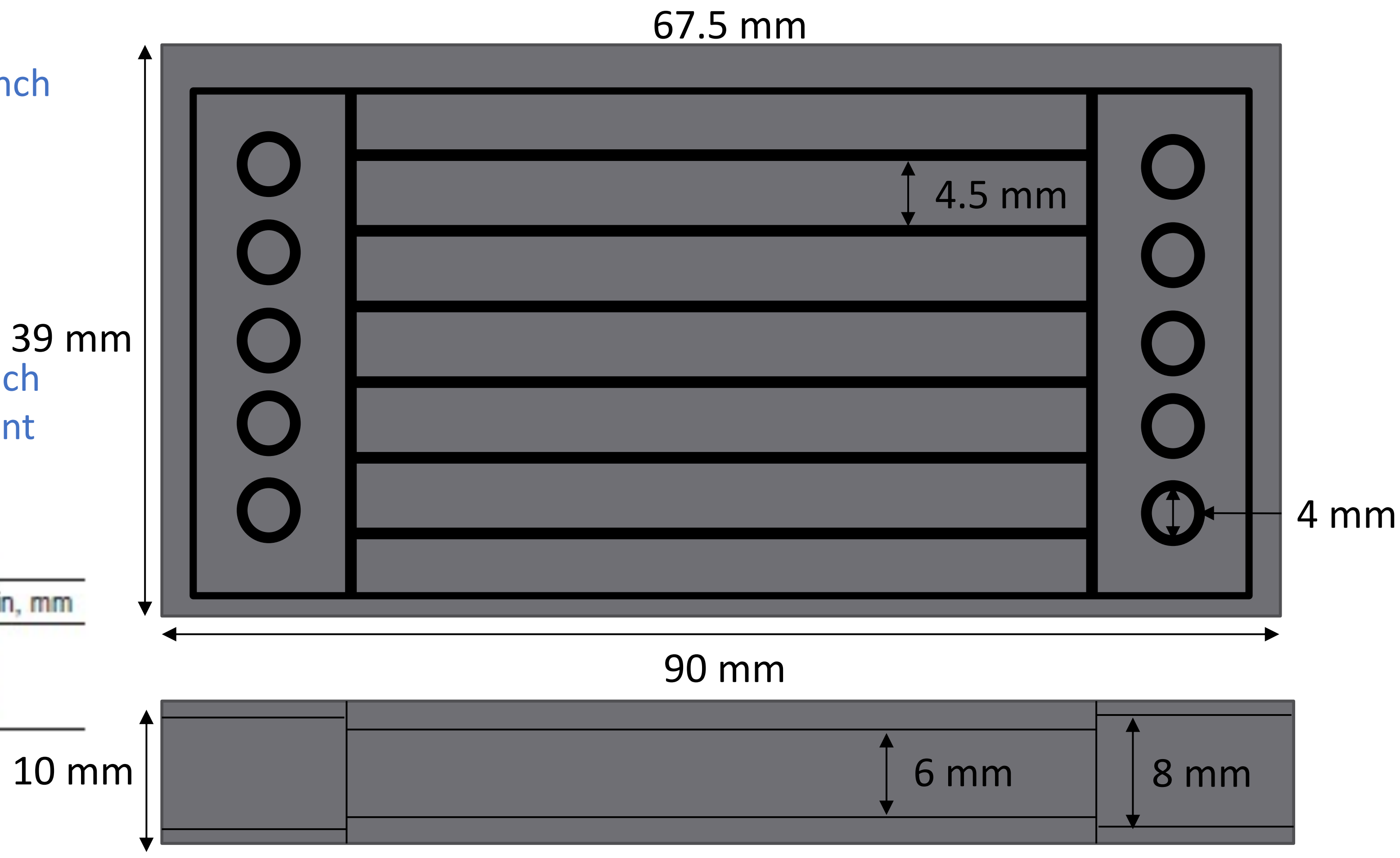
- Each crucible could yield:
  - 7 specimen for flexural testing
  - 10 specimen for compressive testing (5 at each end of the sample to identify possible gradient in processing heat)

TABLE 3 Specimen Size

Configuration	Width (b), mm	Depth (d), mm	Length (L_T), min, mm
A	2.0	1.5	25
B	4.0	3.0	45
C	8.0	6.0	90

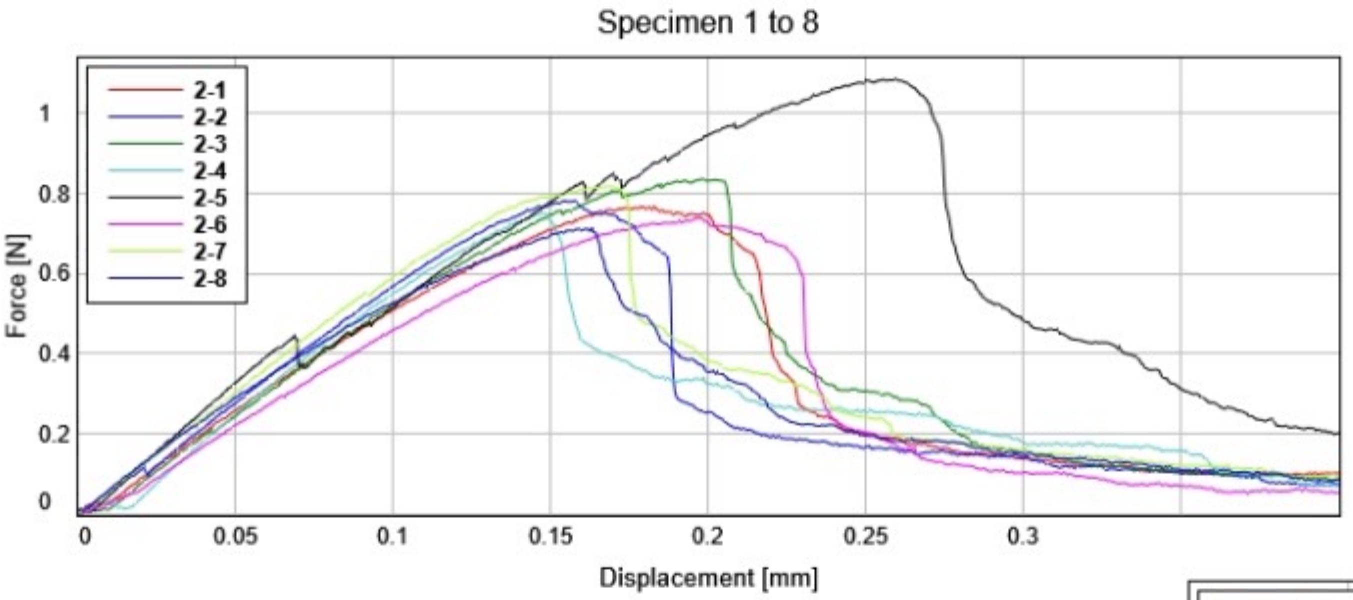
ASTM C1161-18

Test specimen dimensions: 6 mm (W) x 4.5 mm (D) x 67.5 mm (L)



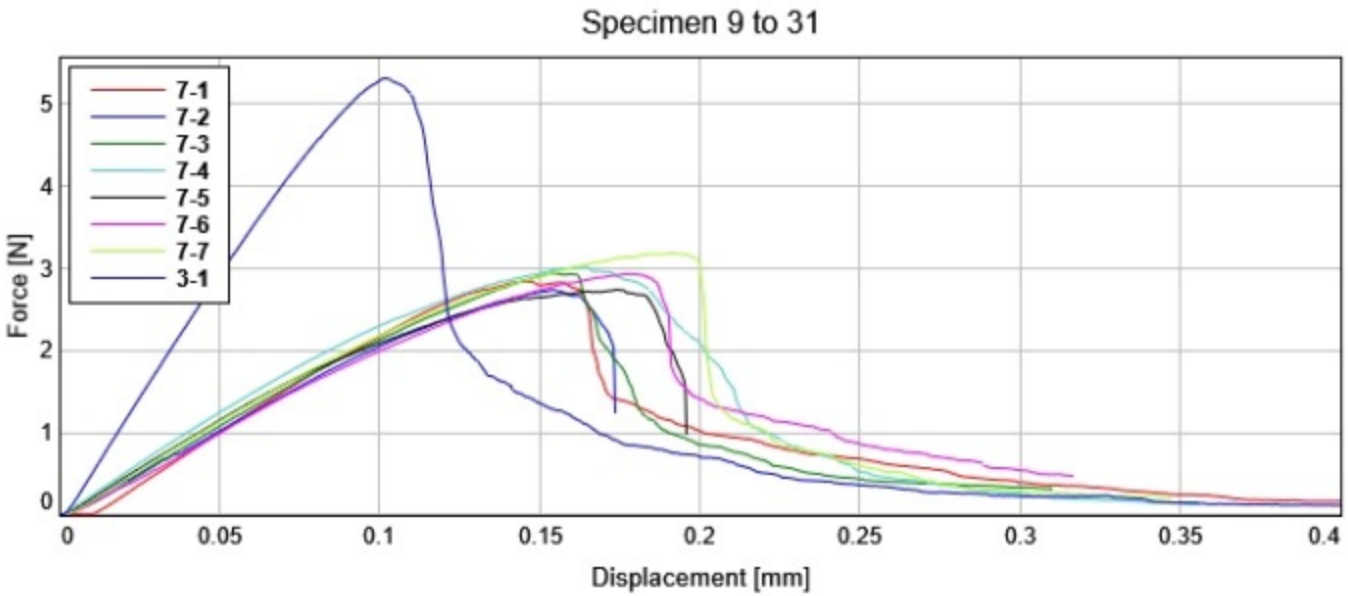
ASTM C1161-18 Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
ASTM C1424-15 Standard Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature

Flexural strength

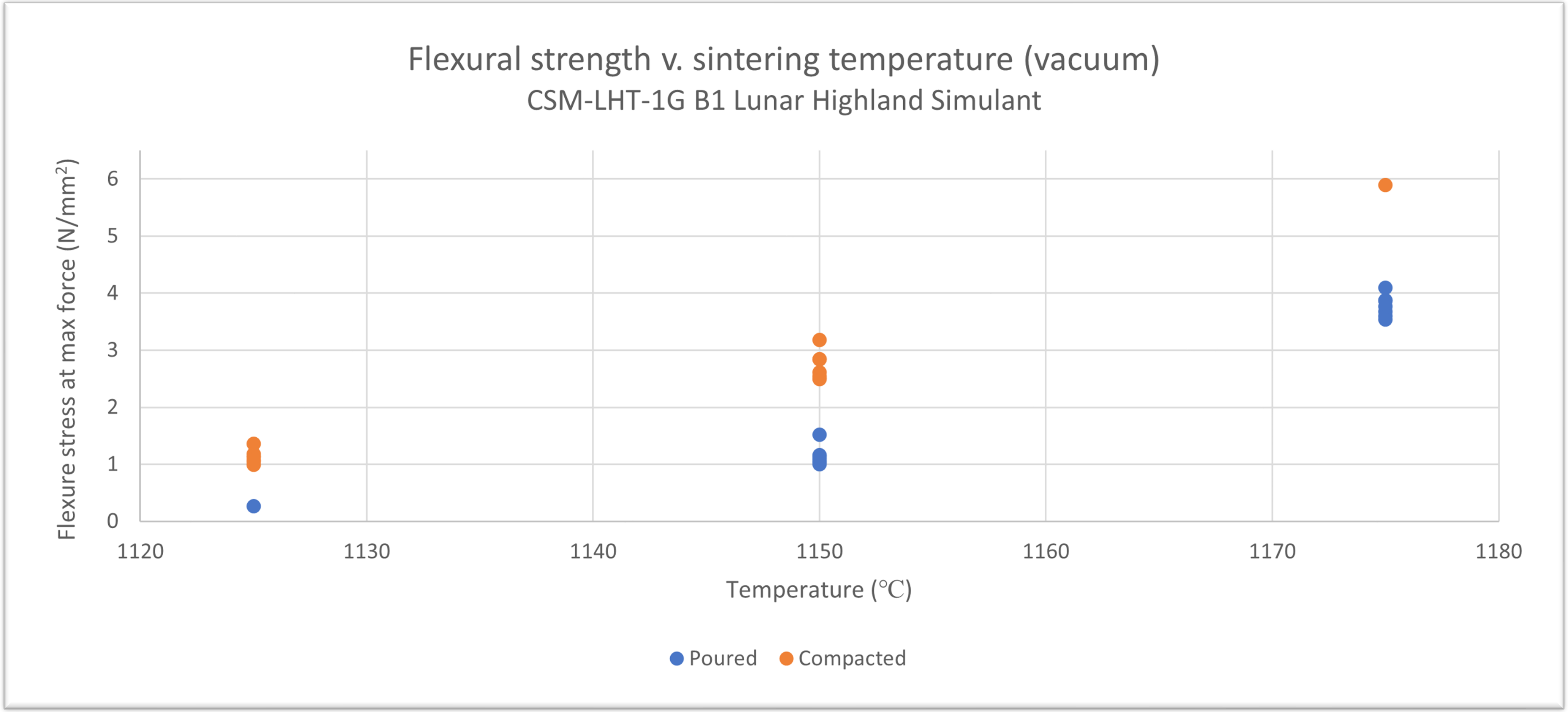


Lower density and lower heat input results

Higher density and higher heat input results



Flexural strength



Preliminary Conclusions

Vacuum experiments are critical for lunar material sintering to validate heating techniques

- They yield different products than those sintered in atmospheres
- They expose the effects of volatile compounds
- They reveal the effects of inhomogeneous heat input

Control of the heat input locally appears critical near the melting point of the glass component

- Temperature gradients near m.p. on the order of 10 ℃ can result in large volatile evolution and large voids or local melting flows

Careful control of heat input and relative density yield consistent results for sintered highland material under vacuum

- Vacuum furnace experiments provide benchmark material behavior and properties for known heat input
- These benchmark data provide the knowledge to understand more complex behaviors with directed heat input techniques (laser, microwave, solar beam)

Know your simulant !!!

- Research the potential effects of the mineral and chemical variations between terrestrial simulants and lunar materials

Publish your results !

Acknowledgments

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NASA Simulant Advisory Committee

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