Biosynthesized Thermoplastic/Regolith Composites for Closed-Loop In-Space Manufacturing

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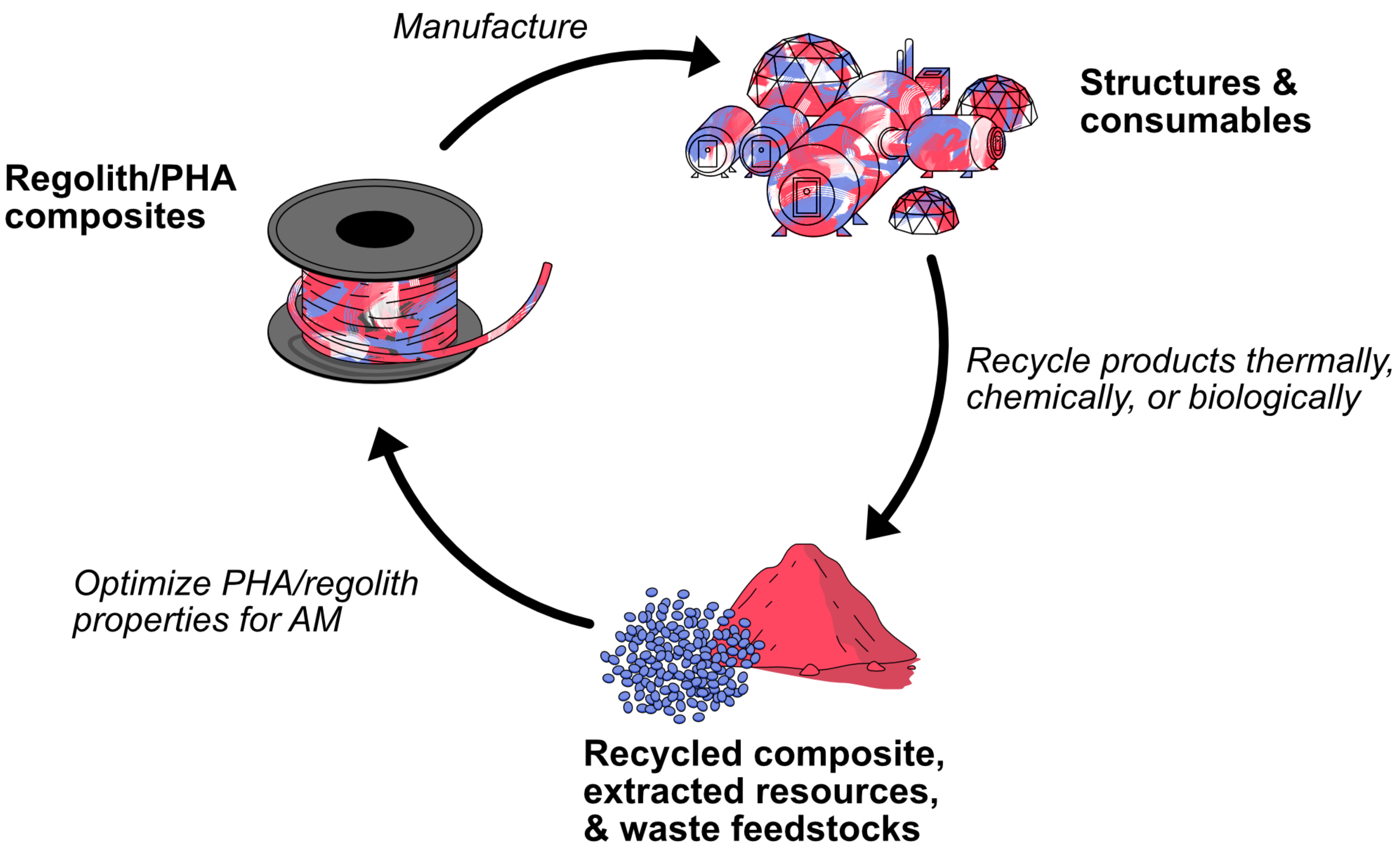
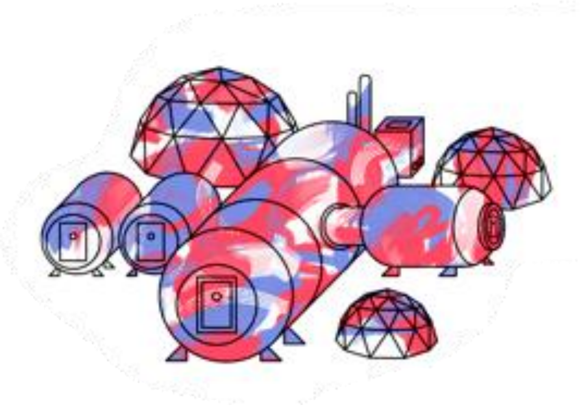
Advanced Polymeric Materials Branch

NASA Glenn Research Center, Cleveland, OH

Presentation overview

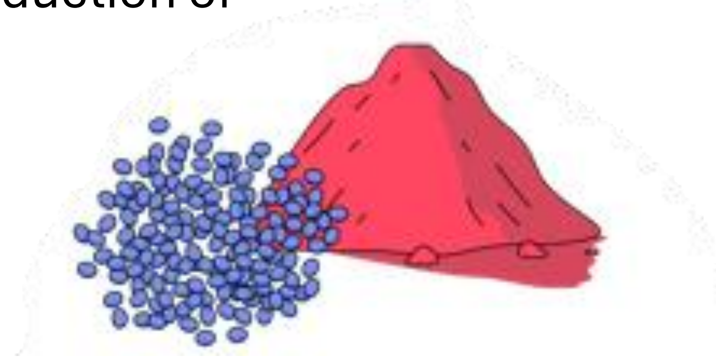
Overview of Closed-Loop ISM System

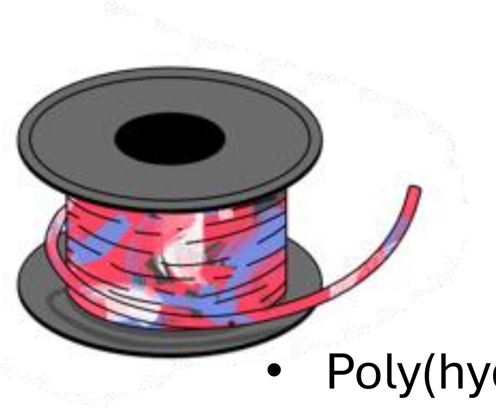




Manufacturing & Closing the Loop

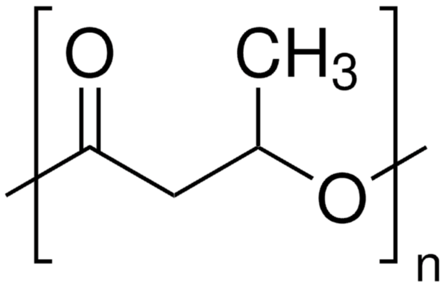
- Manufacturing targets include consumables and outfitting supplies
- Fundamental materials data will guide additional applications
- Multiple routes of manufacturing expand versatility of system
- End-of-life recycling methods include bacterial fermentation and repolymerization, chemical, and mechanical recycling routes
- Closed-loop is key for sustained long-duration missions and reduction of waste





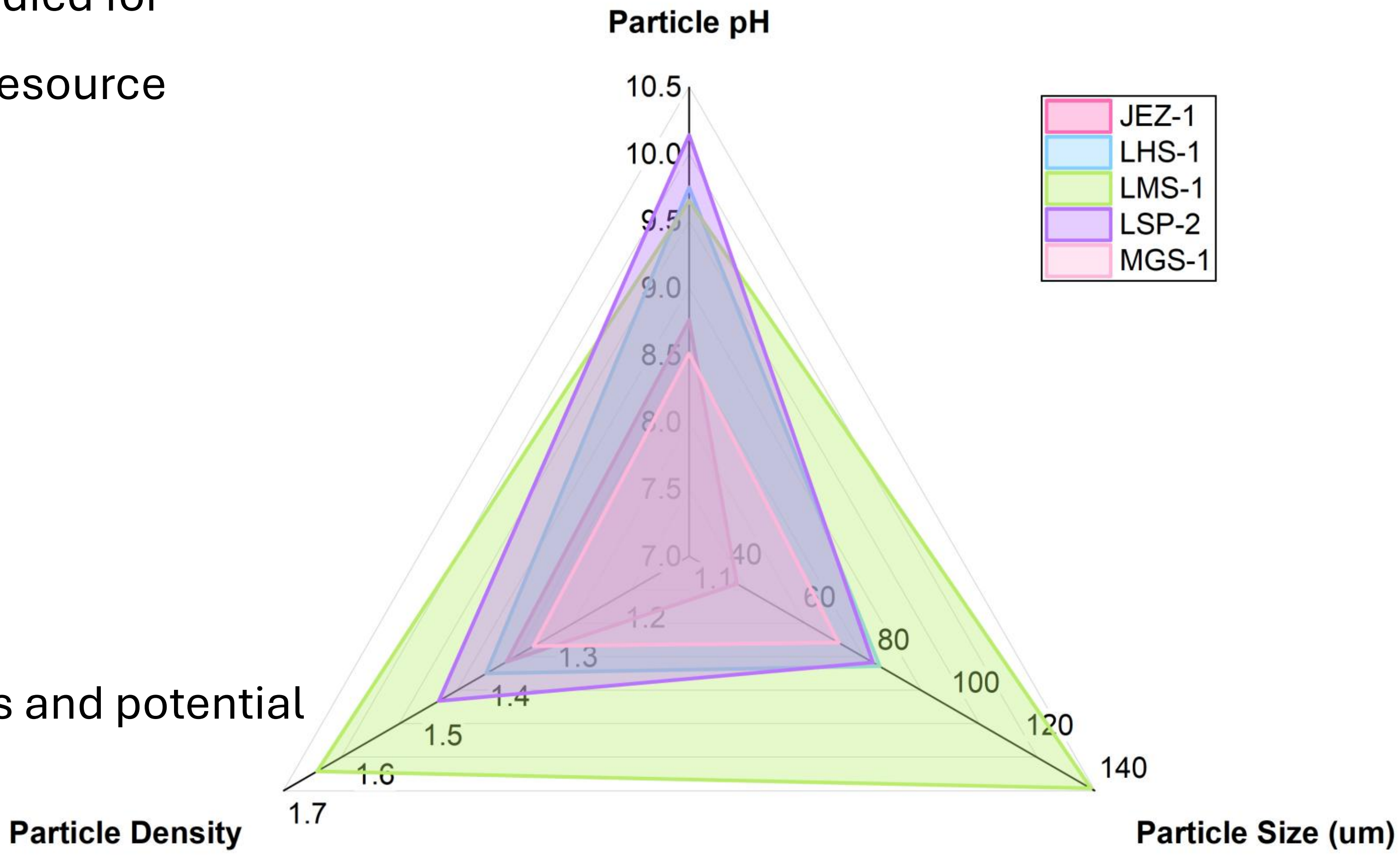
Regolith/Resin Composites

- Poly(hydroxyalkanoate)s (PHAs) are bacterially synthesized resins which offers route to in-situ manufacturing & reduced initial payload requirements for long-duration missions
- PHAs can use various sources of carbon (including waste products, CO_2, polymers etc.) to create more material
- Poly(3-hydroxybutyrate) (PHB) is the most common PHA, but generally requires additives to improve performance & manufacturability due to high crystallinity
- PHB will be used as proof of concept for initial study, although other biosynthesized resins are also viable candidates
- Plant based, additive free PHB used for all samples



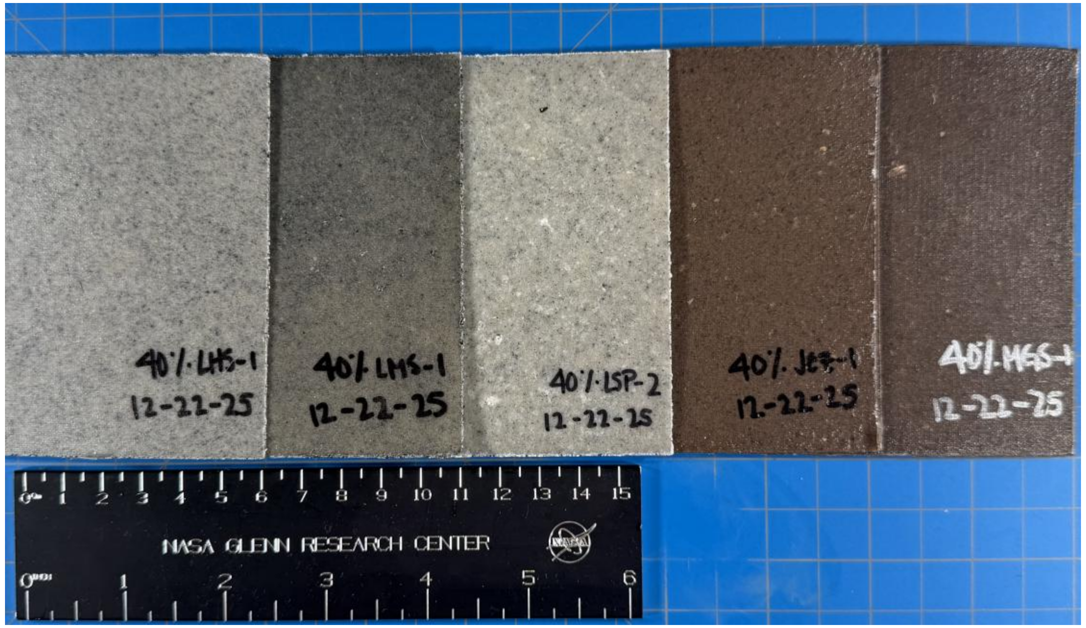
Regolith Simulant Candidates

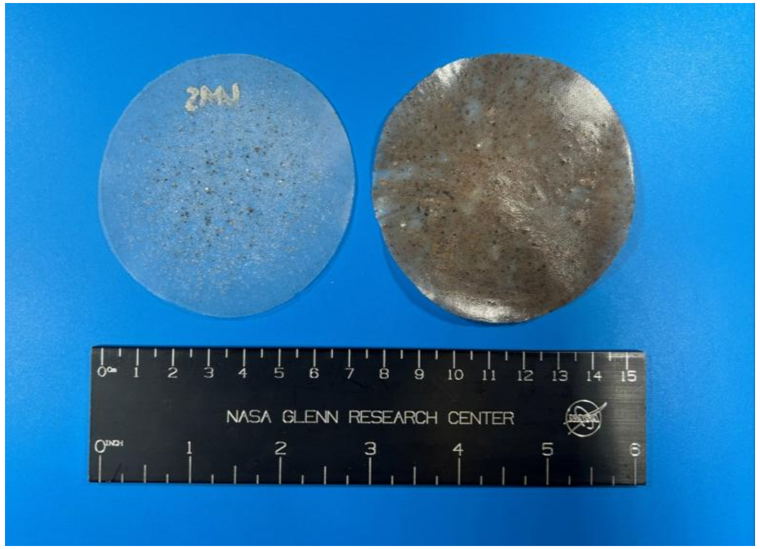
- 5 candidate regolith simulants studied for materials screening from Space Resource Technologies
  - Mars Jezero Delta (JEZ-1)
  - Lunar Highlands (LHS-1)
  - Lunar Mare (LMS-1)
  - Lunar South Pole (LSP-2)
  - Mars Global (MGS-1)
- Design system for as many regions and potential applications as possible



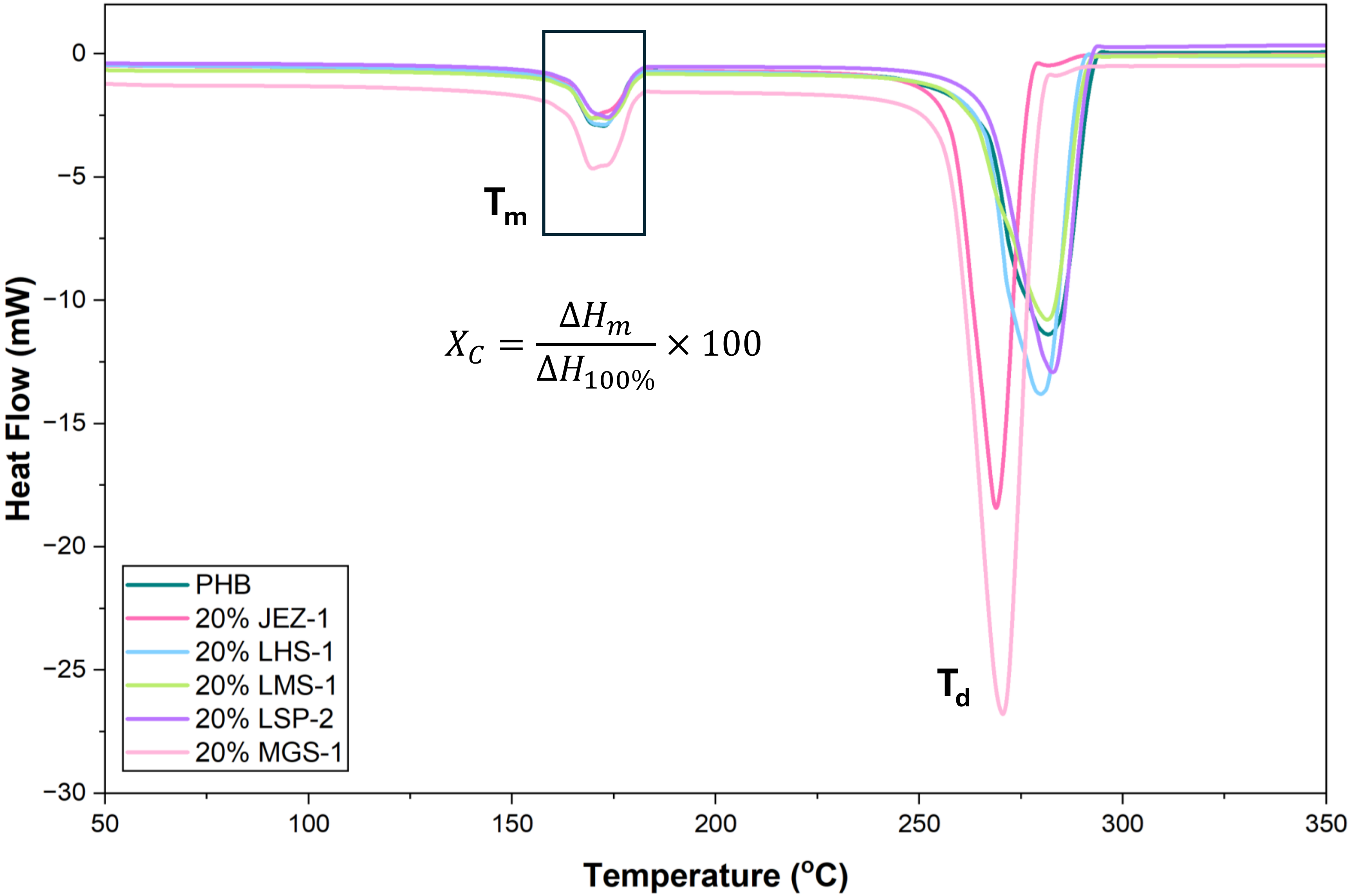
Regolith as In-Situ Nucleating Agent

- PHB is highly crystalline with slow crystallization kinetics, narrow processing temperature
  - This makes material very brittle and processing challenging
  - Mineral additives have been used terrestrial to improve properties
- Could regolith particles be used as in-situ nucleating agents to reduce or control crystallinity?
  - Used compression molding to achieve up to 80 w/w% loading with sufficiently thin films for study



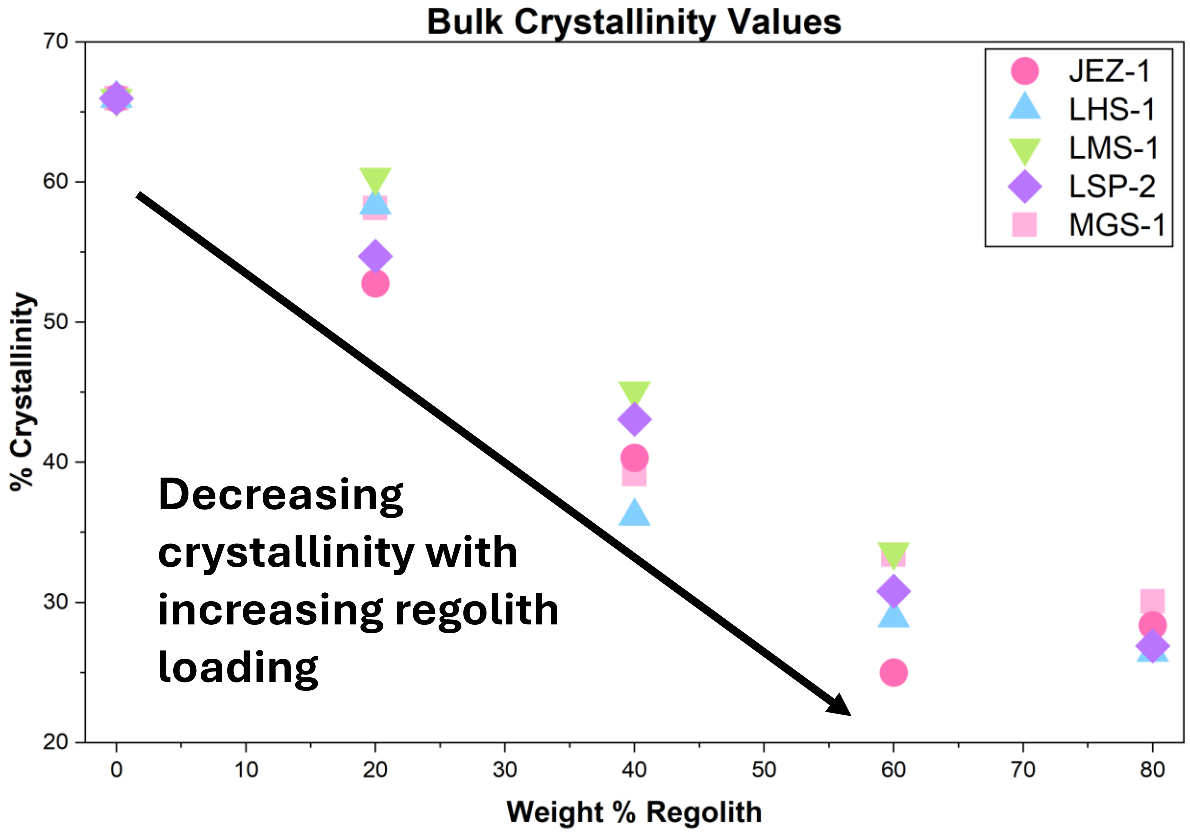


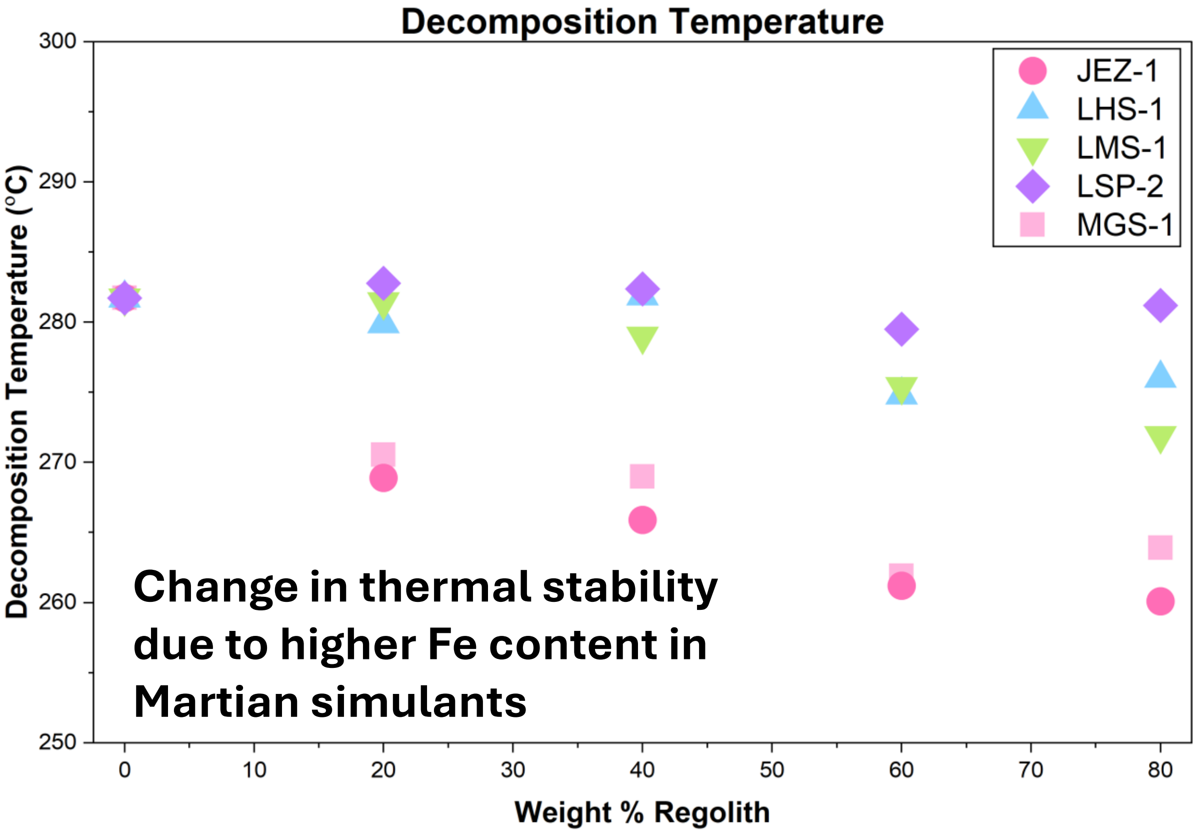
Regolith/PHB Composite Crystallinity – *Differential Scanning Calorimetry*



$$X_C = \frac{\Delta H_m}{\Delta H_{100\%}} \times 100$$

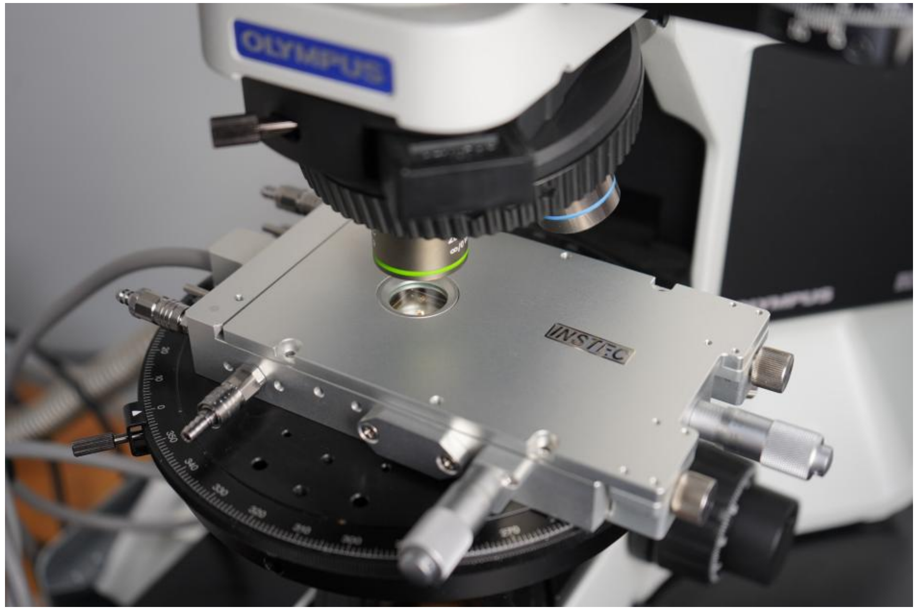
Regolith/PHB Composite Crystallinity – *Differential Scanning Calorimetry*

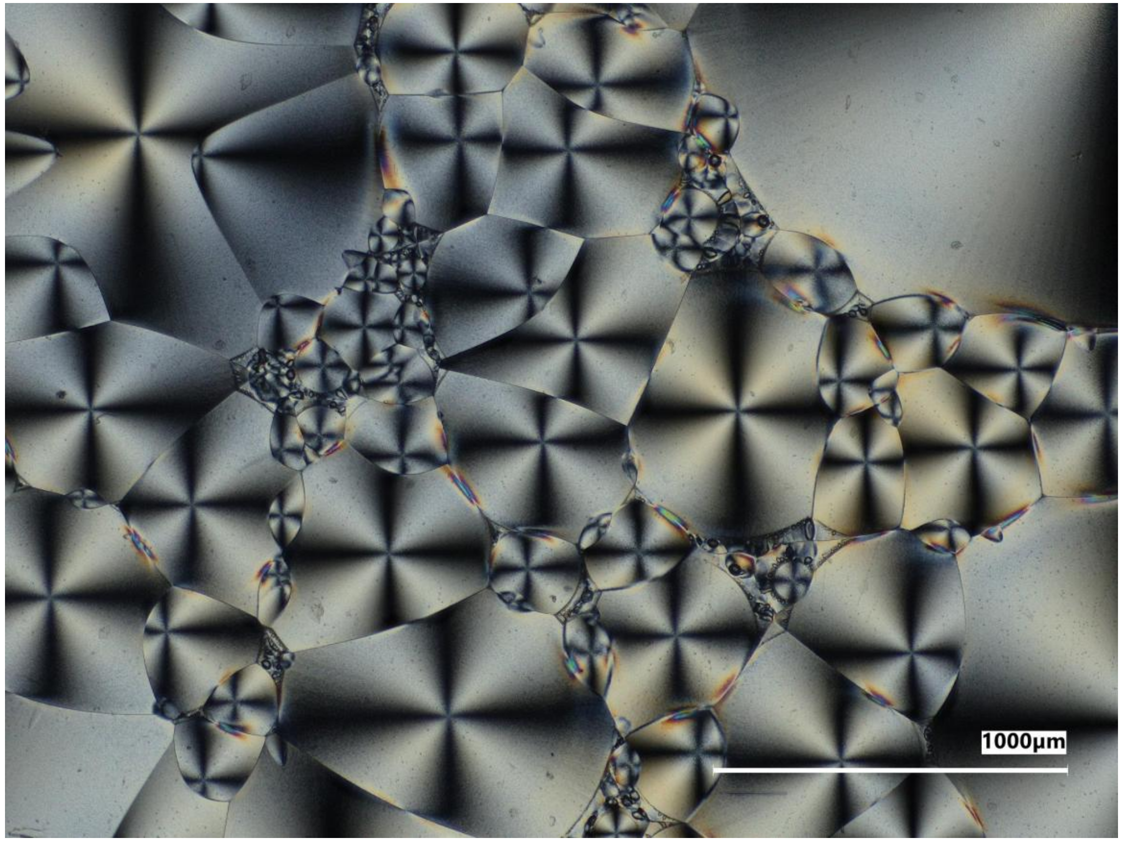




Regolith/PHB Composite Crystallinity – *Cross Polarized Optical Microscopy*

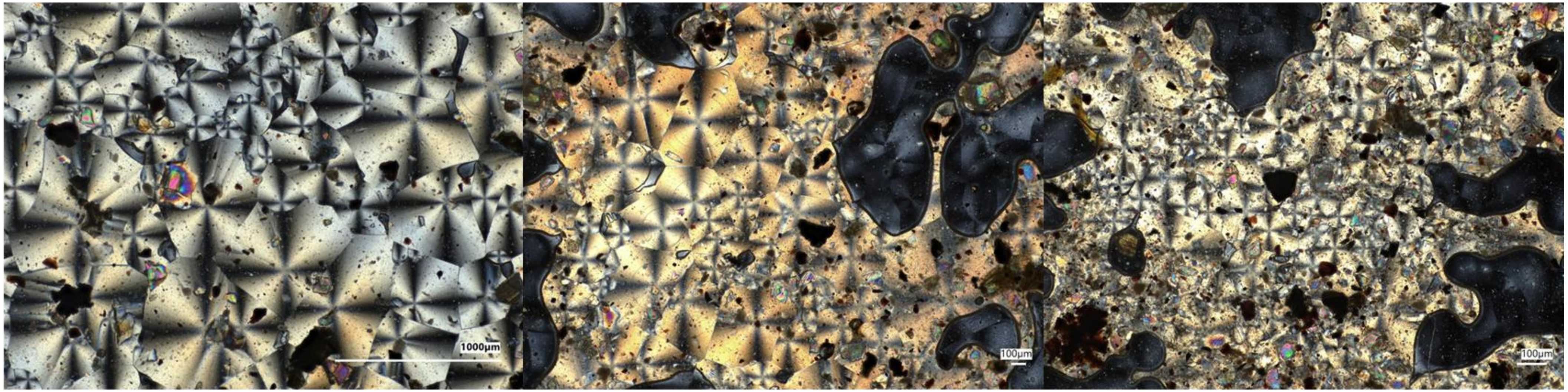
- Captures variations in crystal structure
- Hot stage attachment allows for real-time monitoring of crystallization
  - 20% regolith samples chosen for study



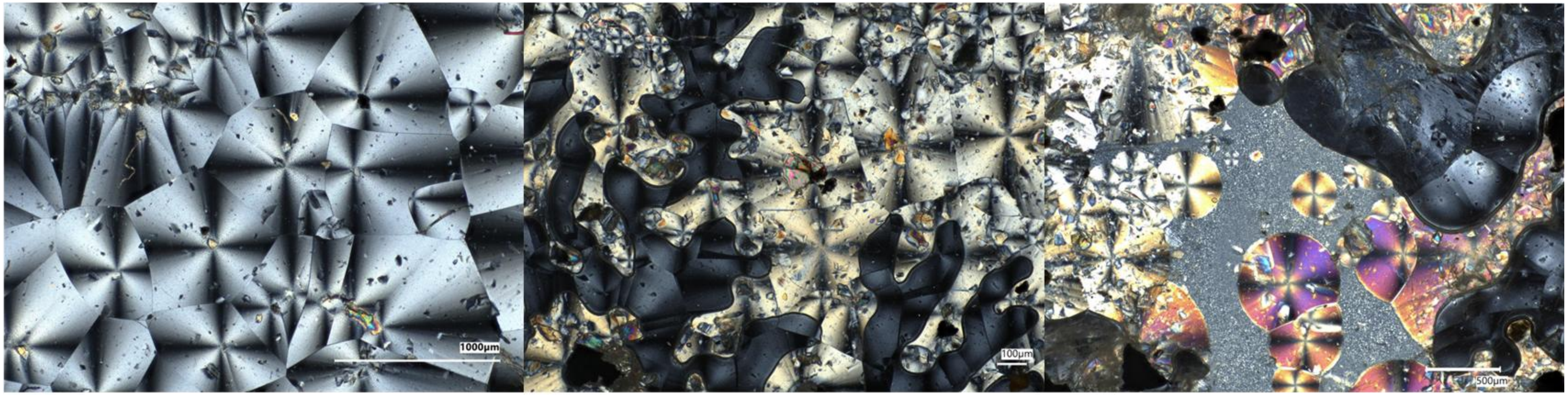


Regolith/PHB Composite Crystallinity –
Effect of Regolith Loading

JEZ-1

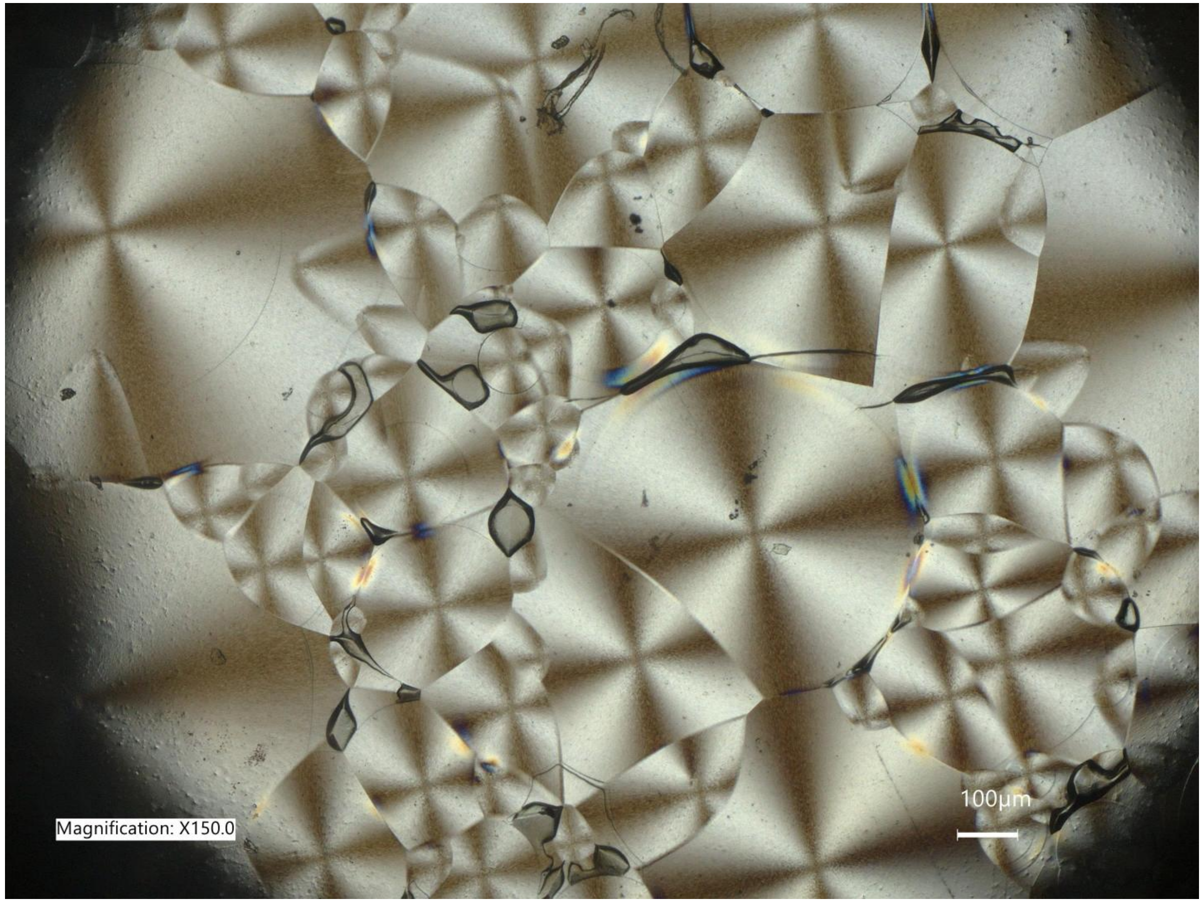


LSP-2

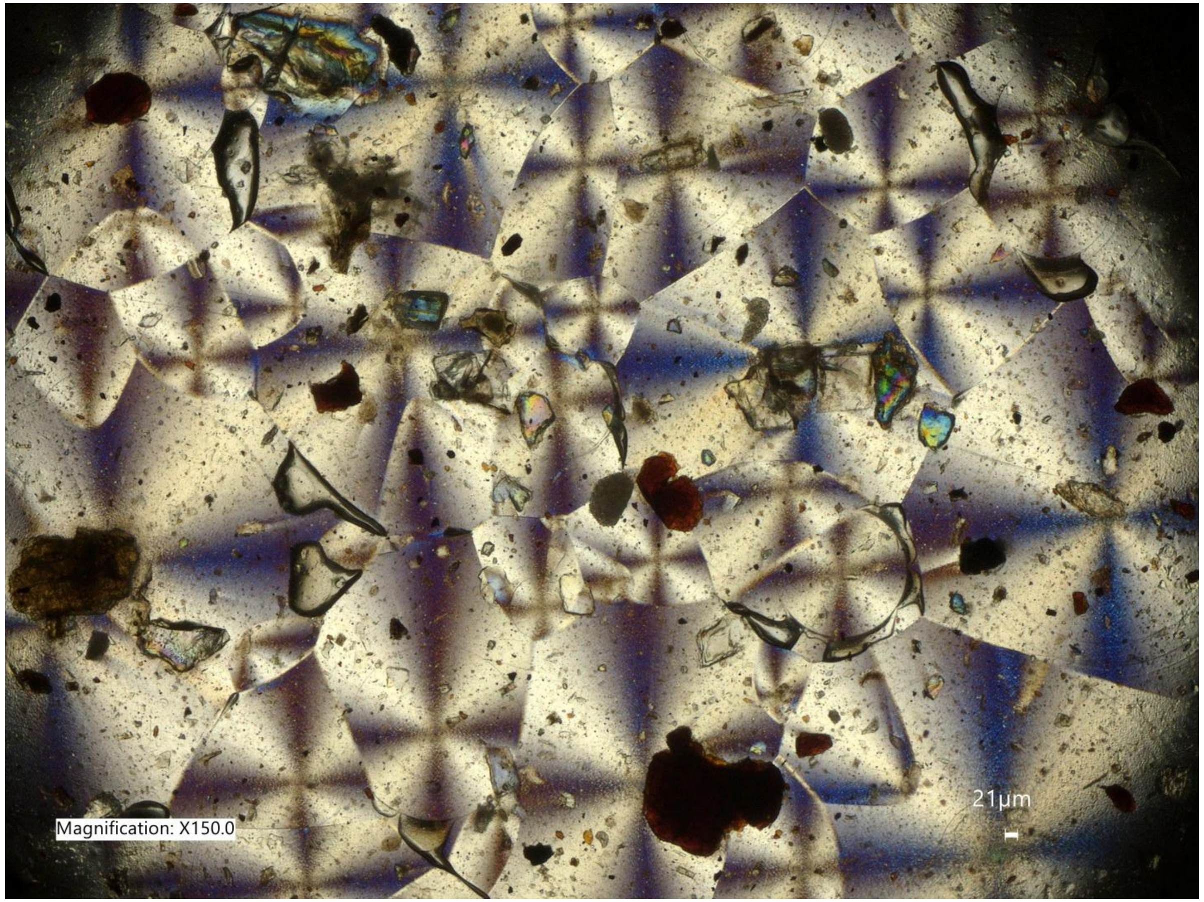


20% **40%** **80%**

Regolith/PHB Composite Crystallinity – *Hot Stage Microscopy*



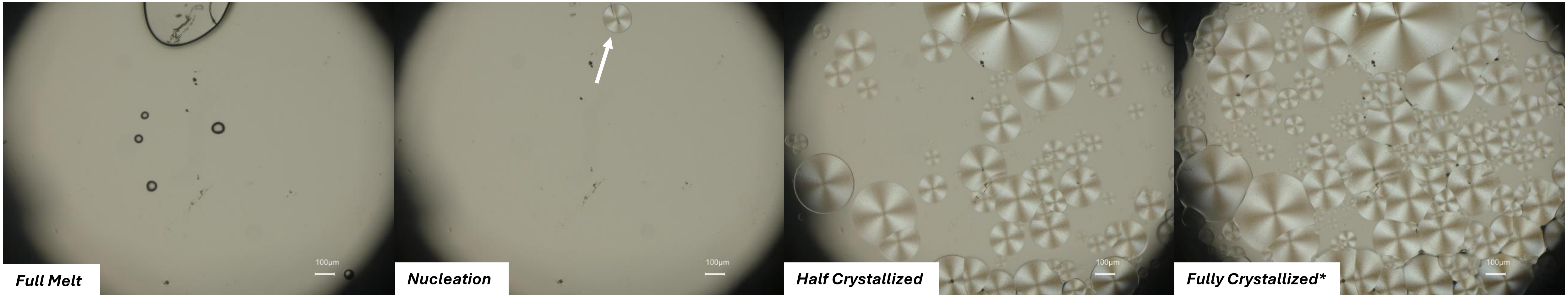
Neat PHB



20 % MGS-1

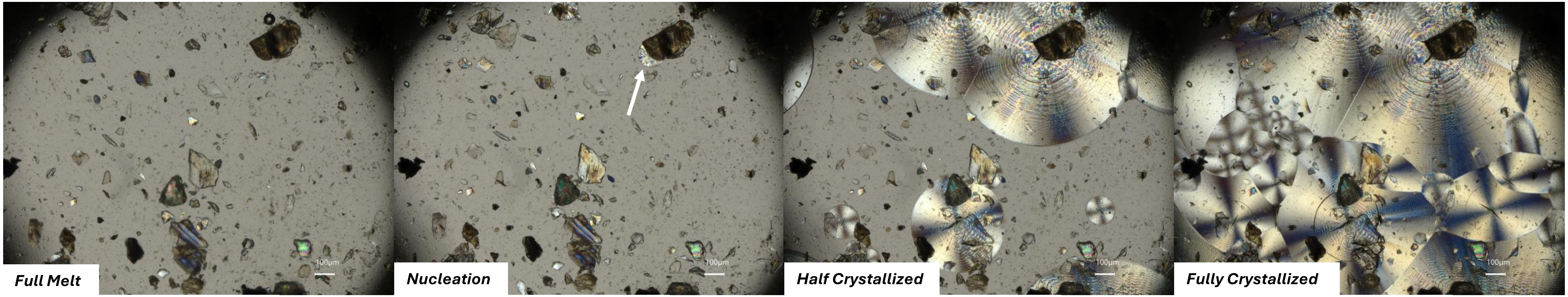
Regolith/PHB Composite Crystallinity – *Hot Stage Microscopy*

(a) Neat PHB



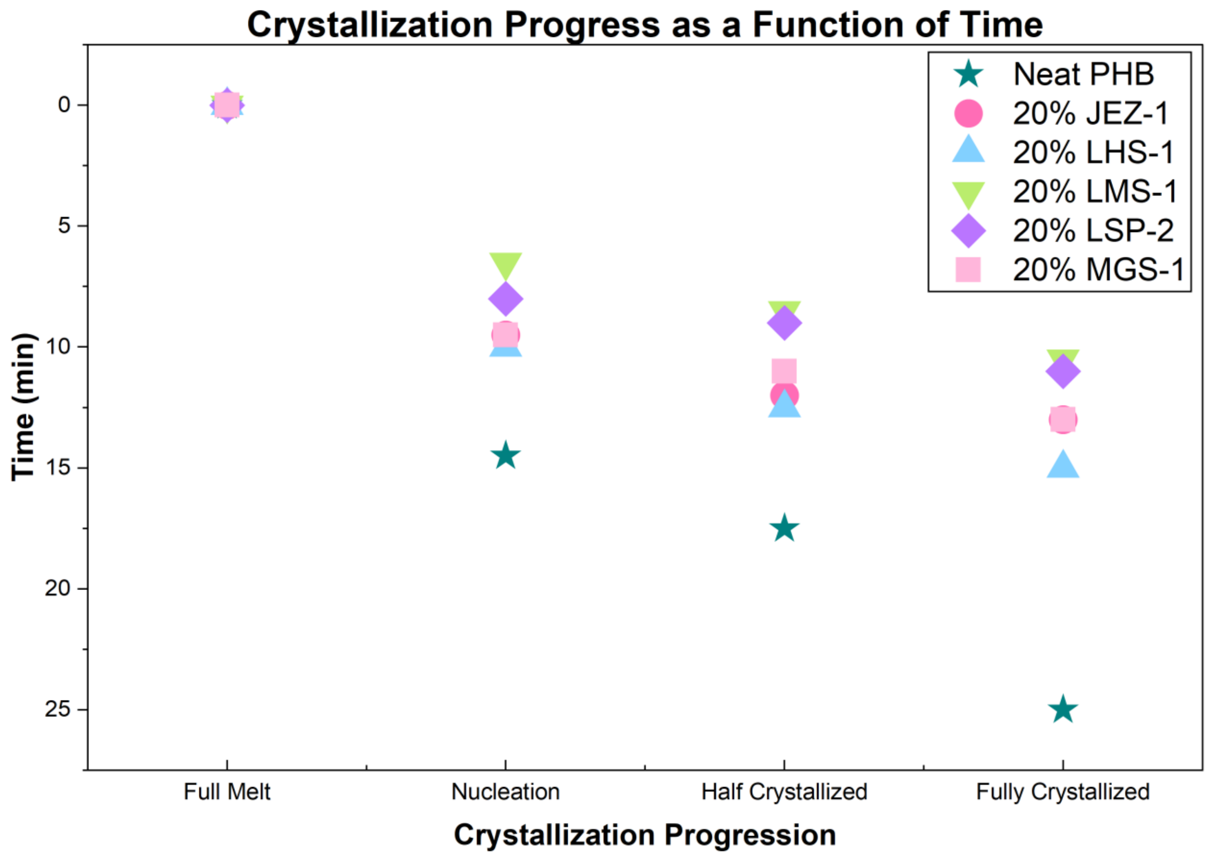
T = 0 min, 190ºC | T = 14.5 min, 45ºC | T = 17.5 min, RT | T = 25 min, RT

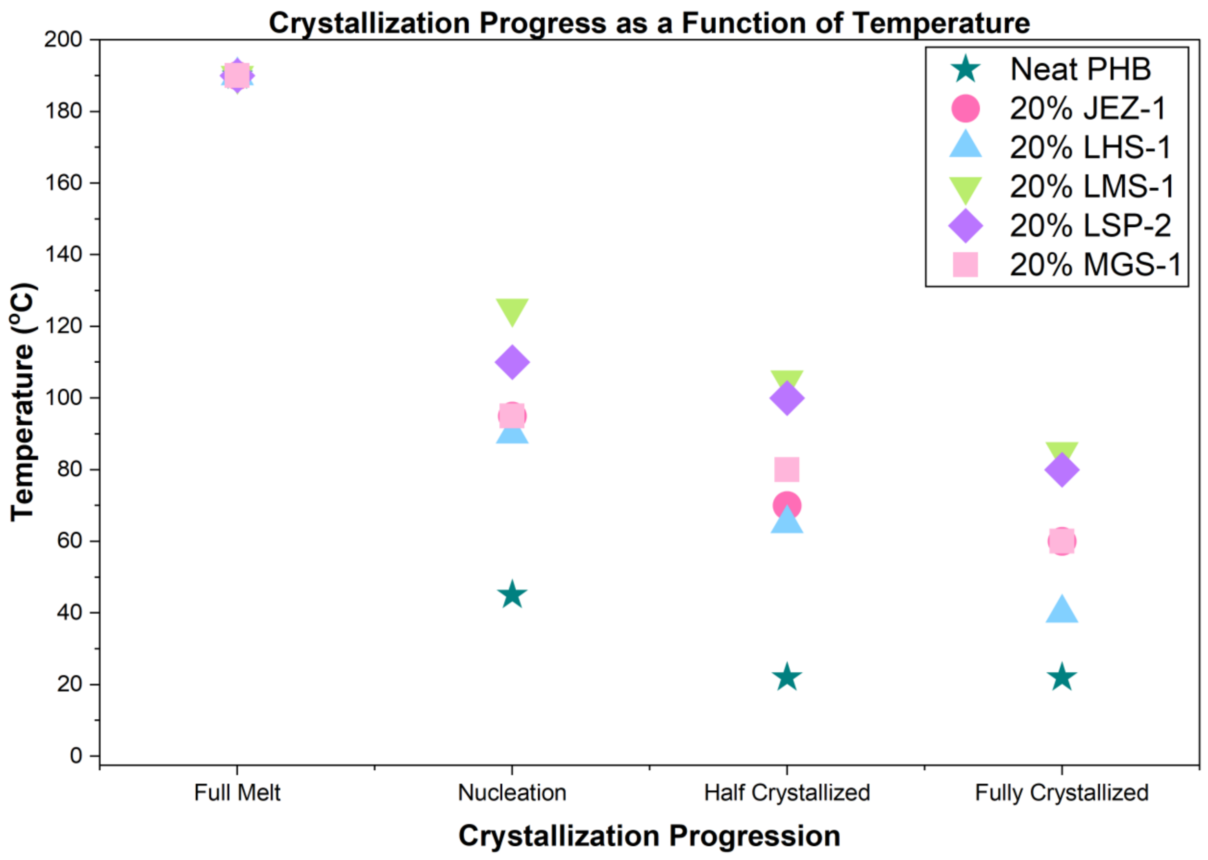
(b) 20% LMS-1



T = 0 min, 190ºC | T = 6.5 min, 125ºC | T = 8.5 min, 105ºC | T = 10.5 min, 85ºC

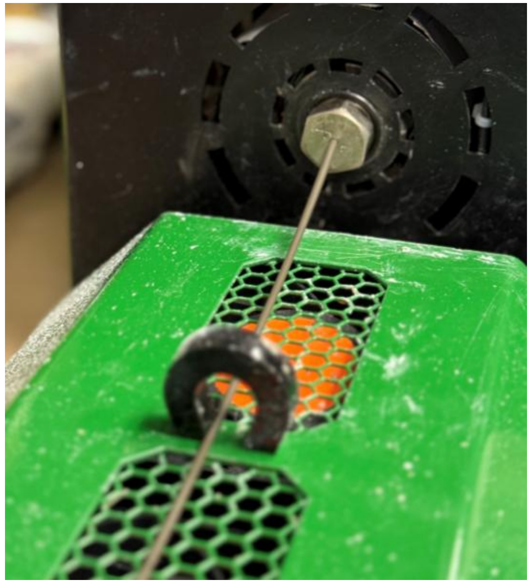
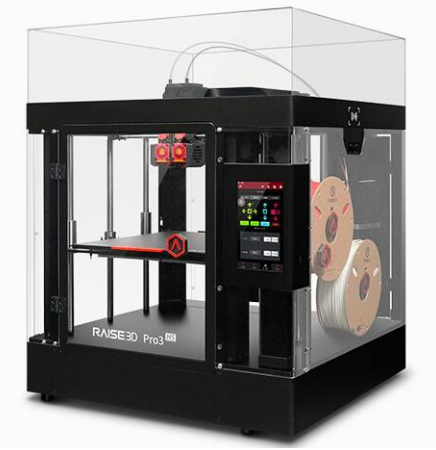
Regolith/PHB Composite Crystallinity – *Hot Stage Microscopy*





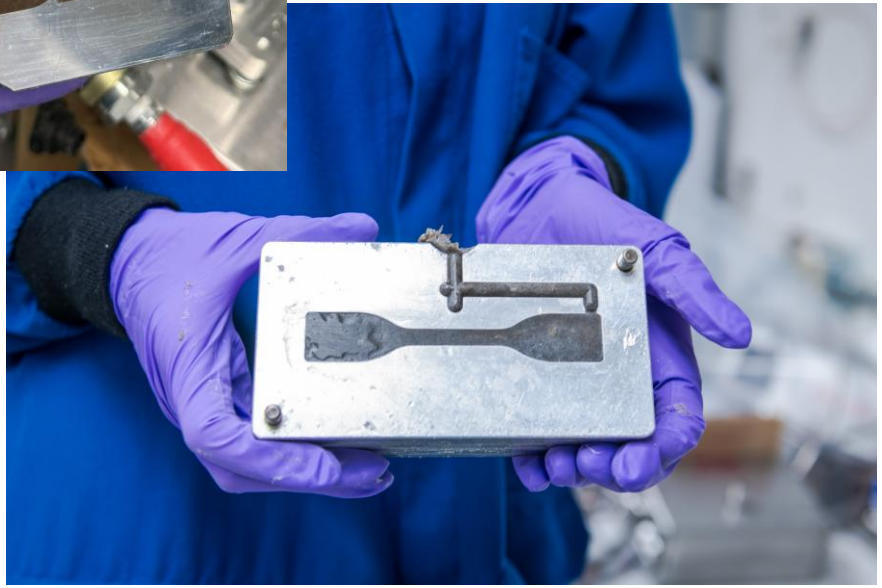
Regolith/PHB Composite Development

- Compression molding
  - High loading, high pressures
  - Inconsistent particle distribution
- Injection molding
  - High particle loading, consistent particle distribution
  - More complex shapes
- Extruding
  - Most consistent particle distribution
  - Lower particle loading
- Additive manufacturing
  - More complex shapes
  - Lower particle loading, thermal exposure

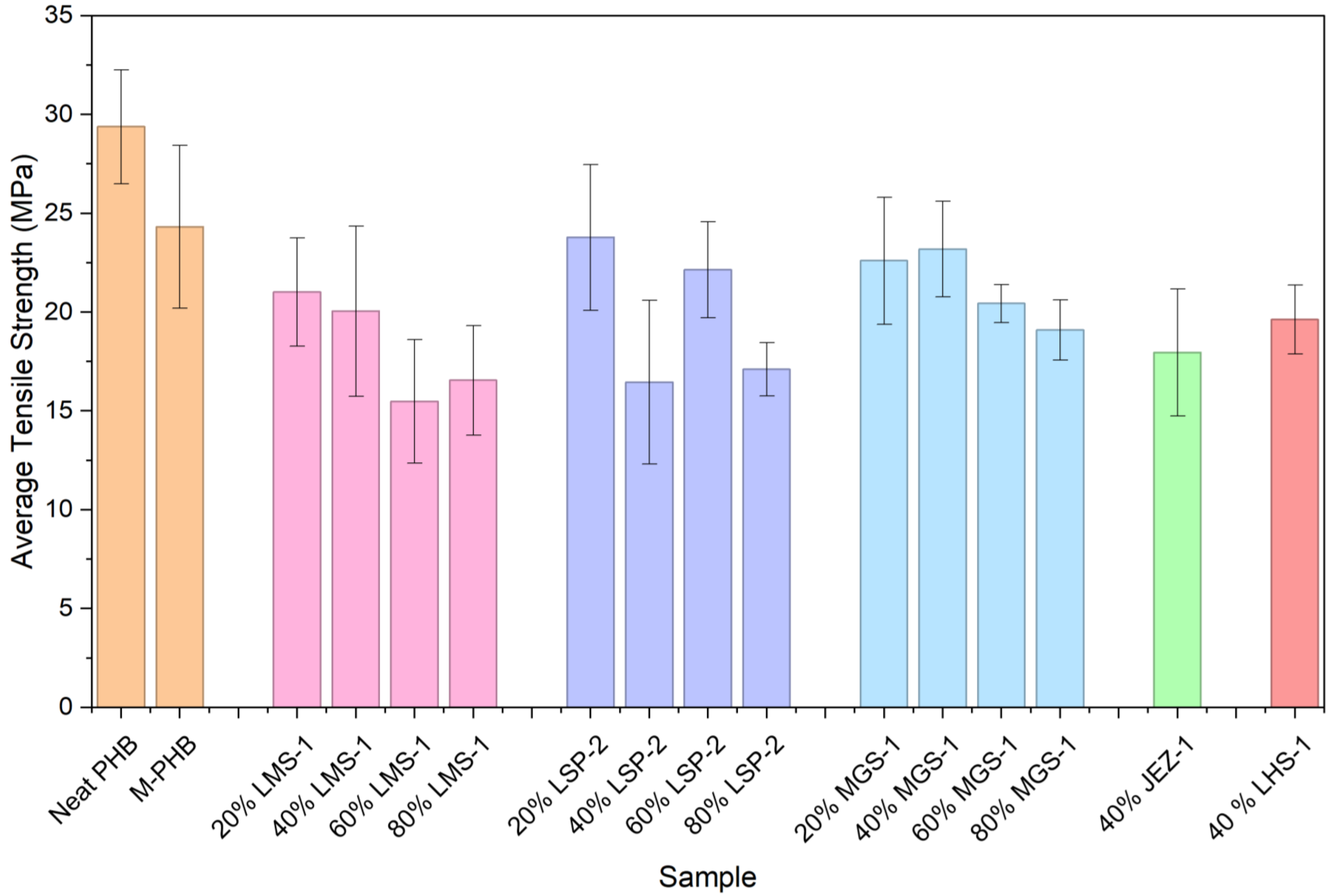



Injection Molded Composites

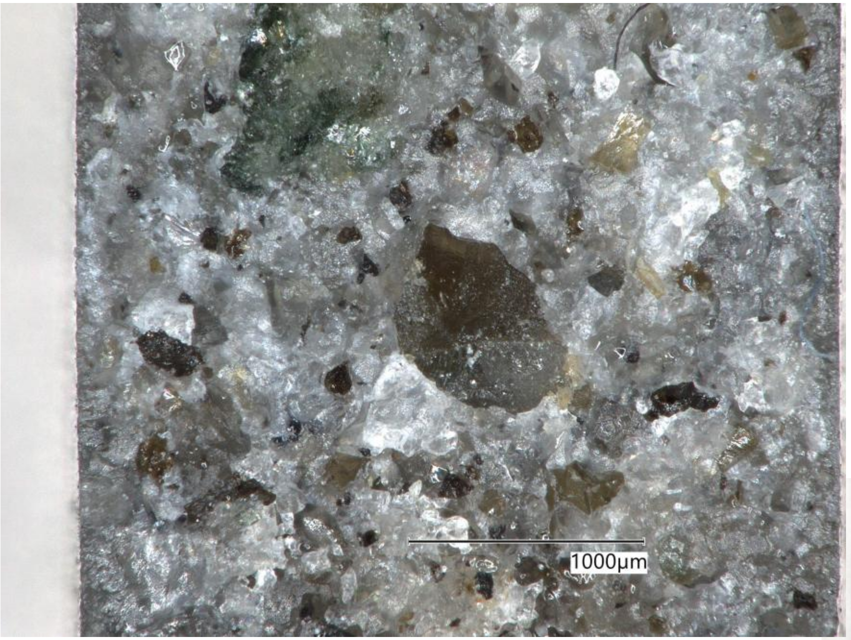
- Optimized processing profile for each material
- Increased viscosity at higher loadings



Injection Molded Composites – *Tensile Strength*

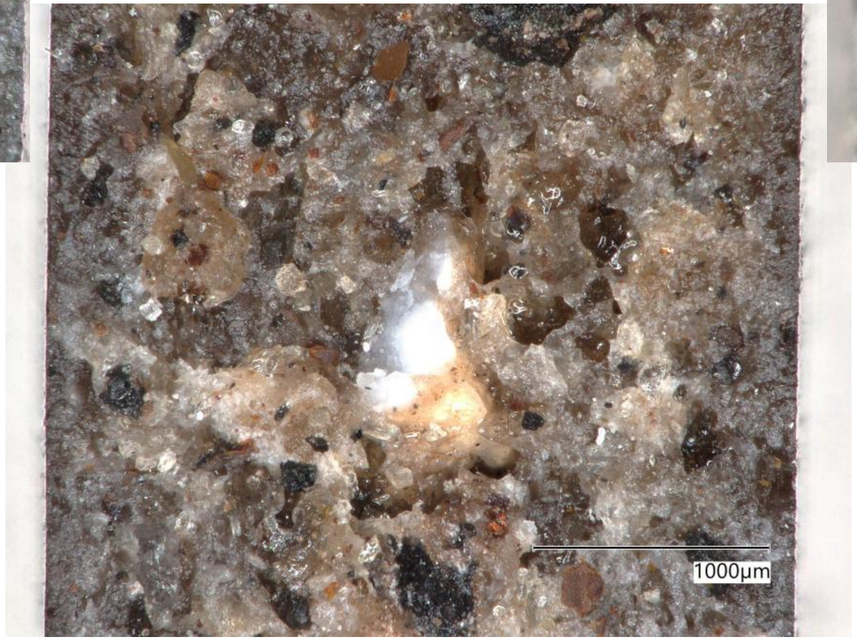


Injection Molded Composites – *Fracture Surface*



60% LHS-1

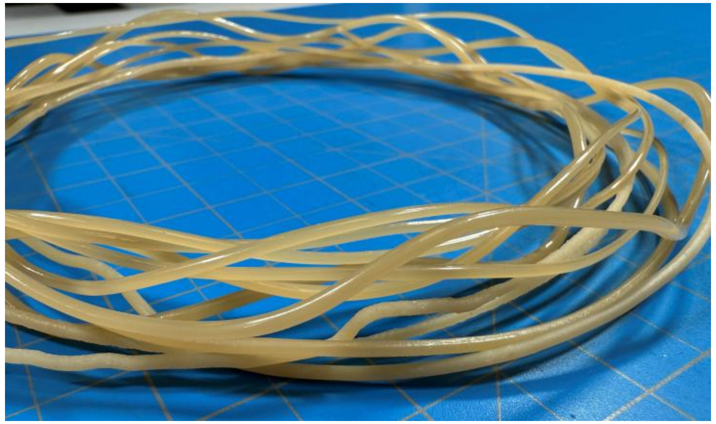
60% MGS-1

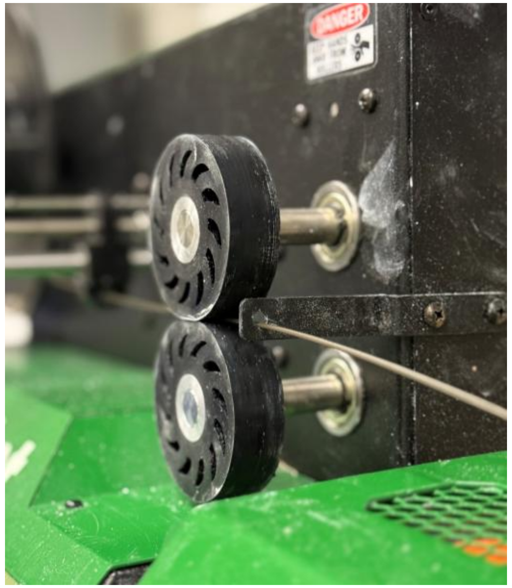


60% LMS-1

Extruding Composites

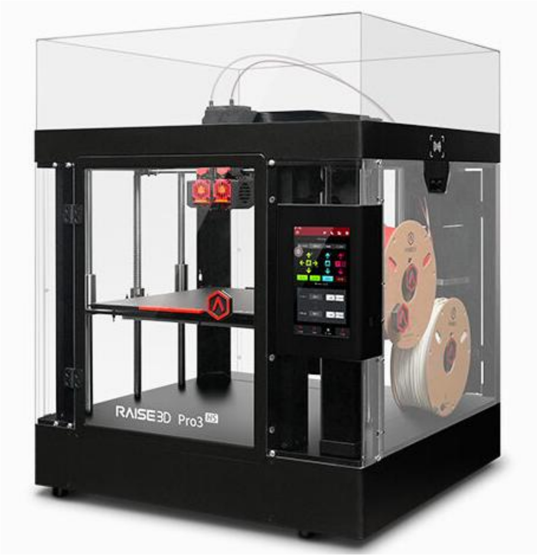
- Cooling sensitivity, particle size distribution, and narrow melt window presented challenges to manufacturing
- Successfully extruded filament using neat PHB, 10% and 20% LMS-1D, and began 40% MGS-1

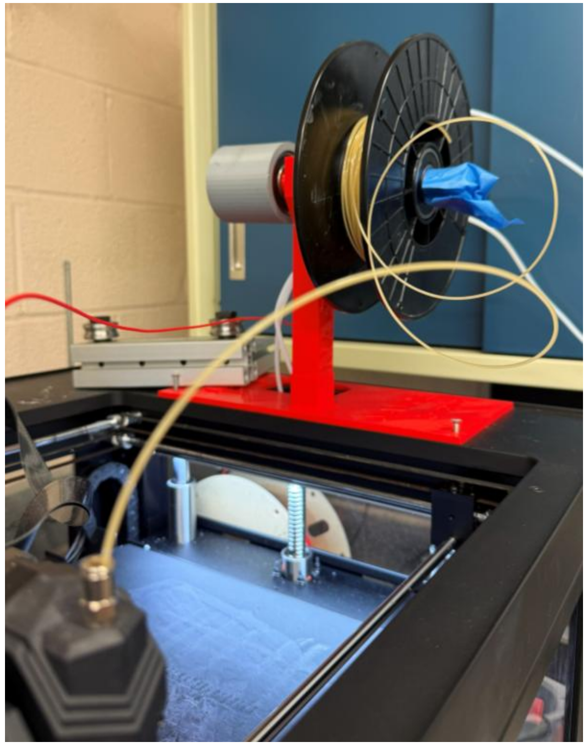


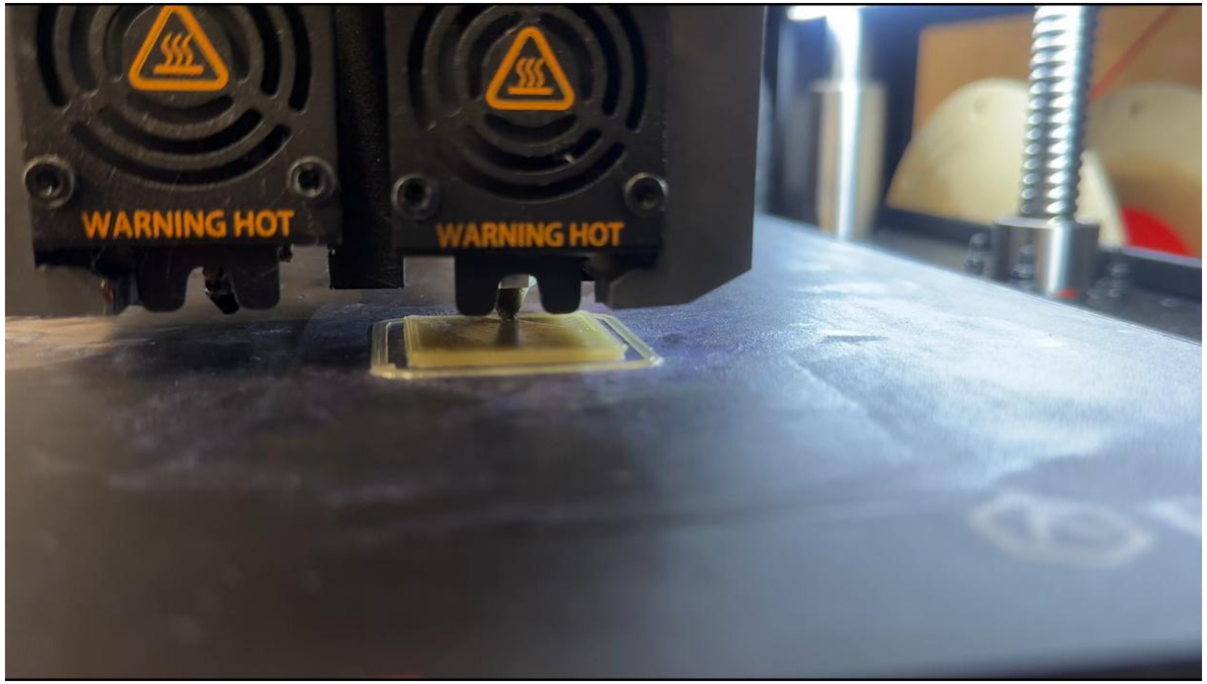


Printing Filament

- Raise 3D Pro, direct drive extruder
- Custom print profile for neat PHB and composite filament
  - Best results with unheated print bed

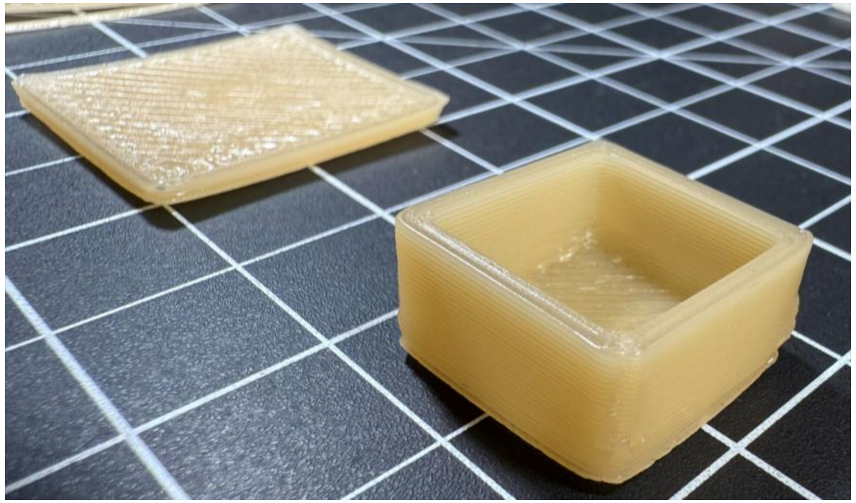


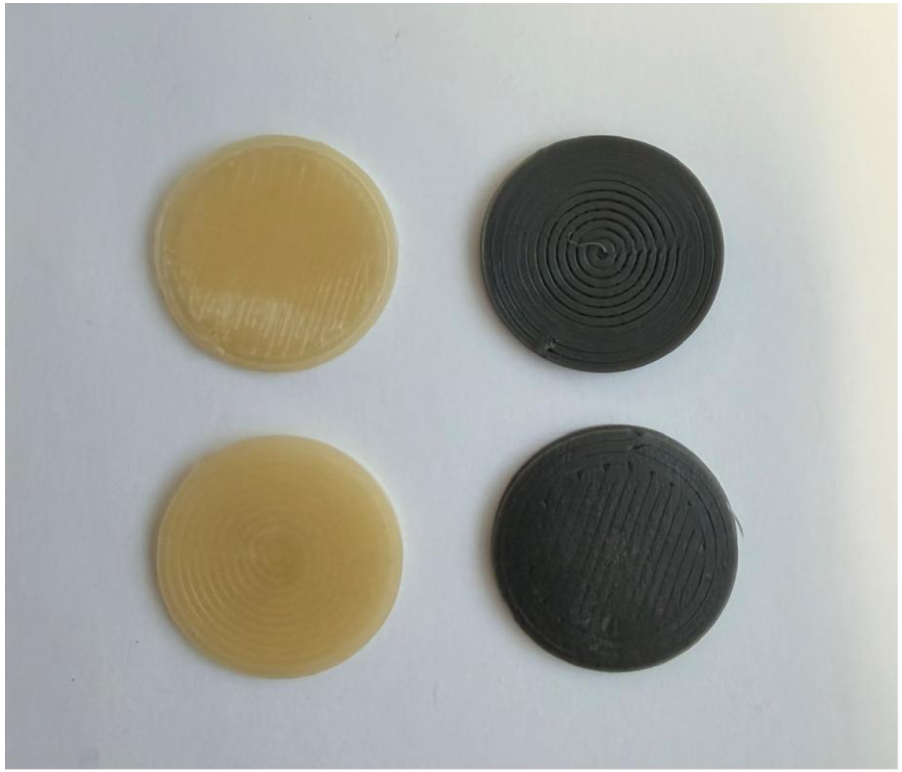


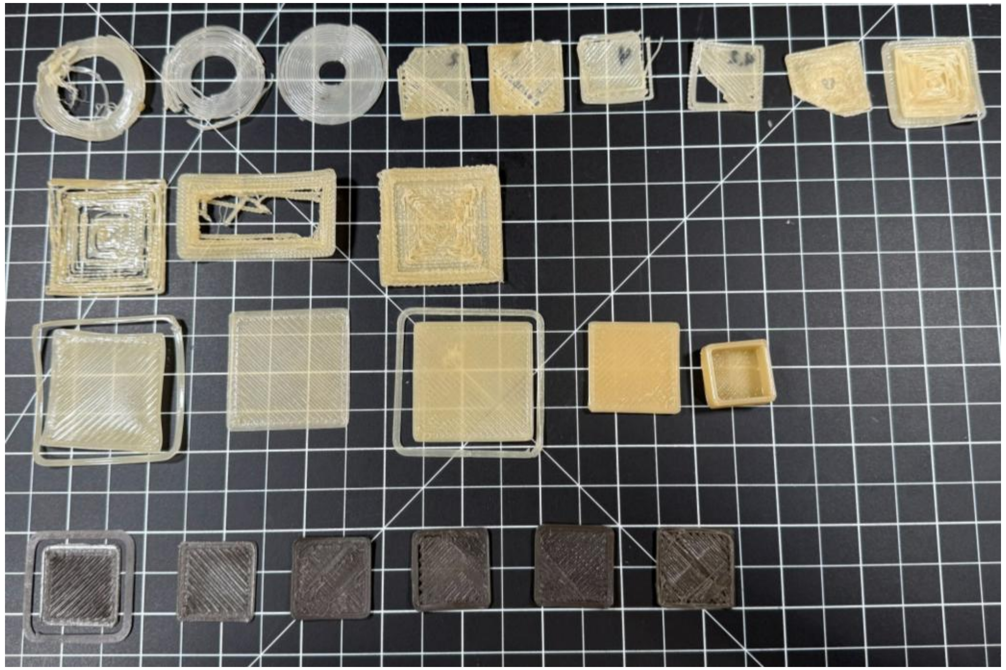


Printing Filament – *Key Takeaways*

- Successfully printed neat PHB and composite filament
- Consistent filament diameter crucial
- Composite filaments prone to clogging or under extrusion
  - Nozzle size is limitation for larger particles

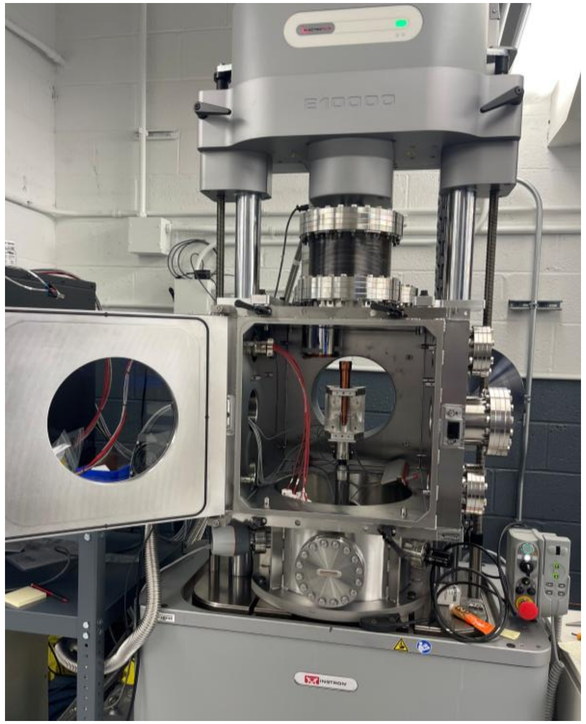


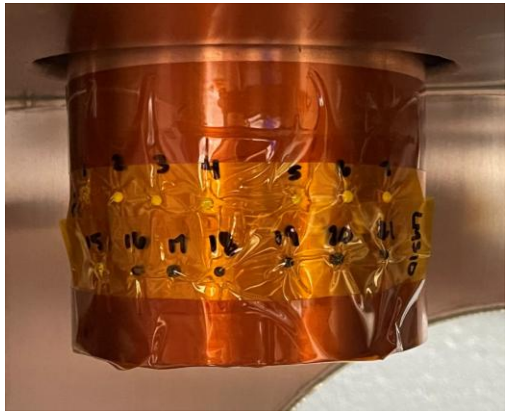




Lunar Environment Structural Test Rig (LESTR) Exposure

- LESTR capable of testing under simulated Lunar conditions, can cycle ~40K to 125K
- Composite samples included as ride-alongs to assess effects of vacuum and cold temperatures on material
- Looking for signs of thermal stress:
  - Cracking
  - Embrittlement
  - Surface changes





Lunar Environment Structural Test Rig (LESTR) Exposure

- Injection molded and 3D printed samples tested
- Samples held at 12.0945 K and 7.37E-6 Torr for 24 h
- No signs of catastrophic failure or significant thermal stress

Before LESTR

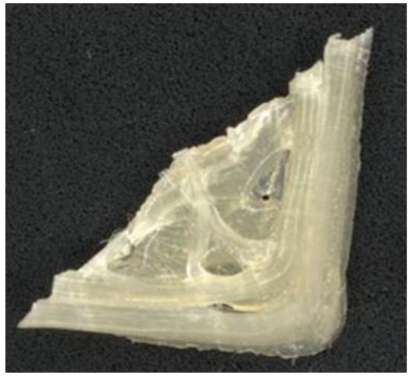
After LESTR

Neat PHB

LSP-2 40%

Before LESTR

After LESTR

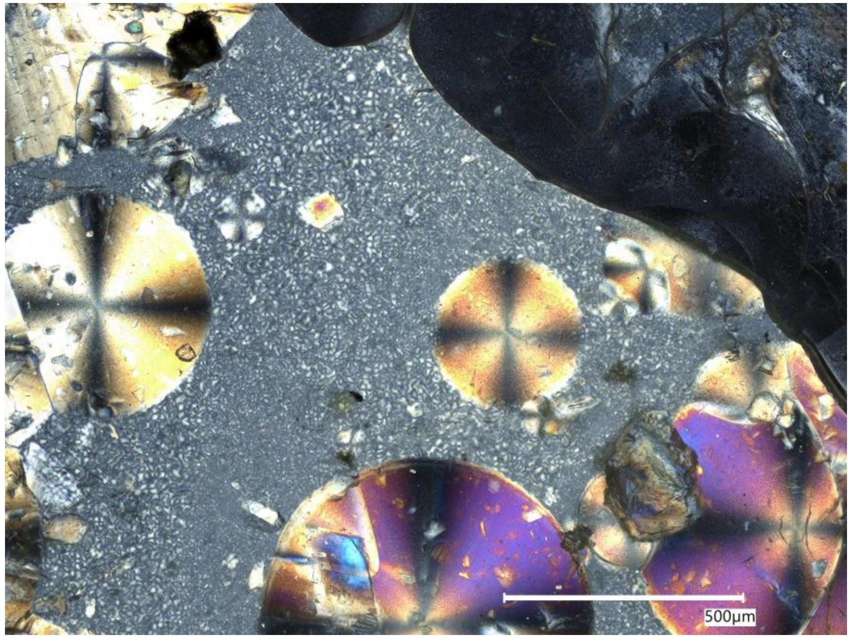


Sample

Sample #5

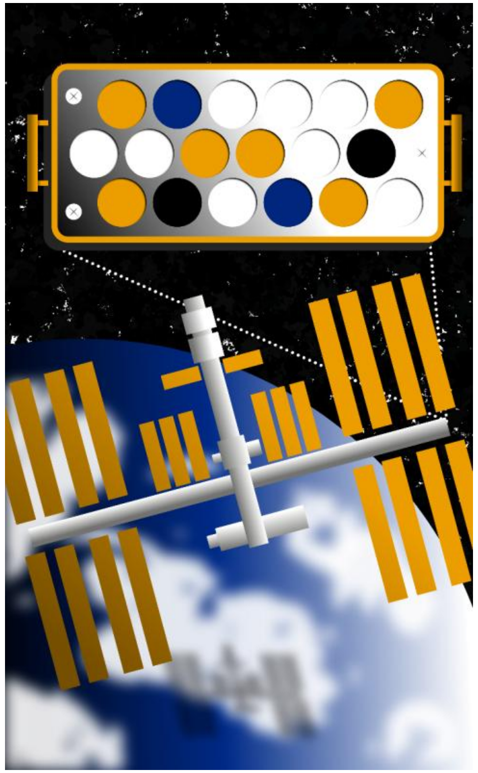
Key Takeaways & Ongoing Work

- **Successful proof of concept!**
  - **Regolith as in-situ additive to improve PHB crystallinity**
  - **Multiple routes of manufacturing**
- **Type and amount of regolith affects the mechanical and thermal properties of composite**



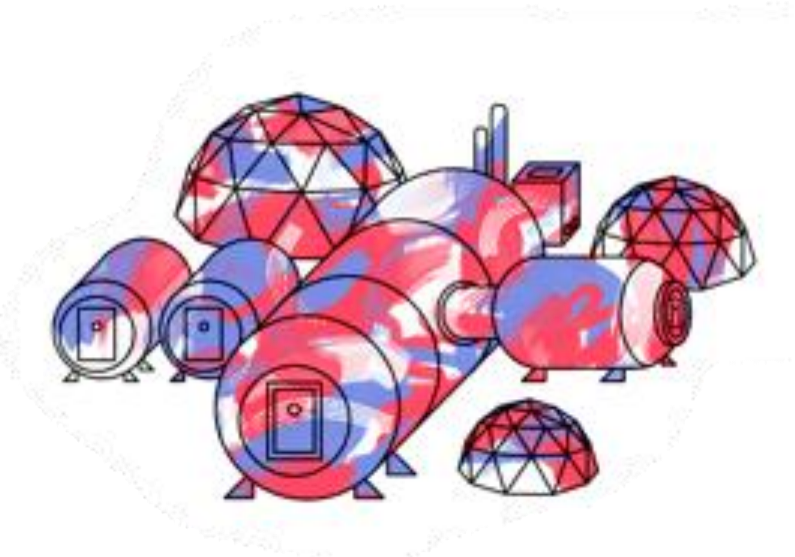
Key Takeaways & Ongoing Work

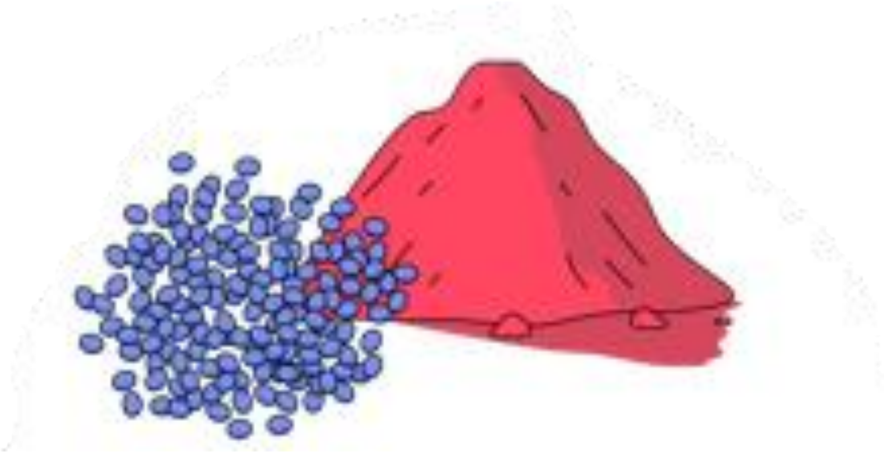
- Successful proof of concept!
    - Regolith as in-situ additive to improve PHB crystallinity
    - Multiple routes of manufacturing
- Type and amount of regolith affects the mechanical and thermal properties of composite
- **Further process optimization, additive manufacturing improvement**
- **Compression, flexural, impact injection molding samples and testing**
- **Additional LESTR testing, environmental exposure evaluation**
- **Samples on MISSE-23 and MISSE-24 experiments for space exposure evaluation**



Next Steps

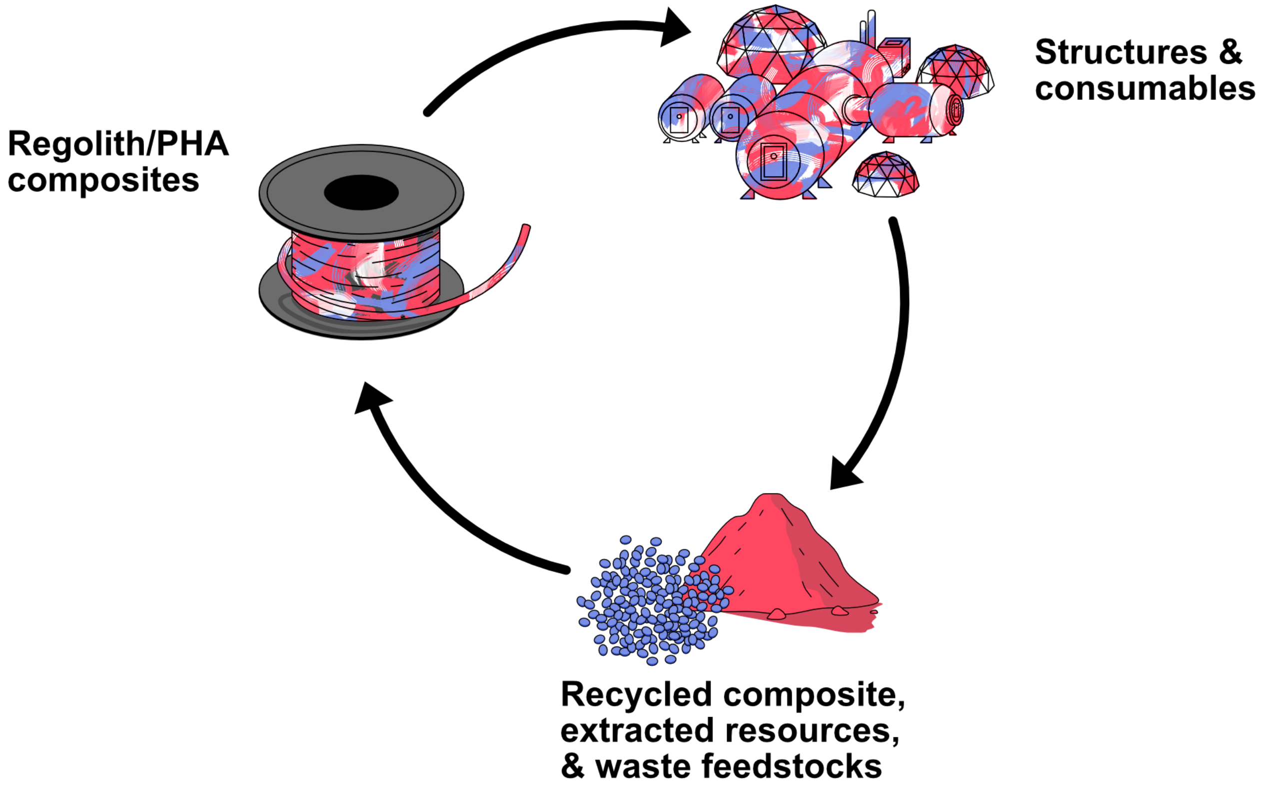
- Manufacturing complex parts and larger structures
- Development of bio-recycling and biosynthesis of resin from waste products
- Explore mechanical recycling methods
- Computational modeling to predict materials performance
- Create materials with bio-engineered PHAs
- Systems analysis





Acknowledgements

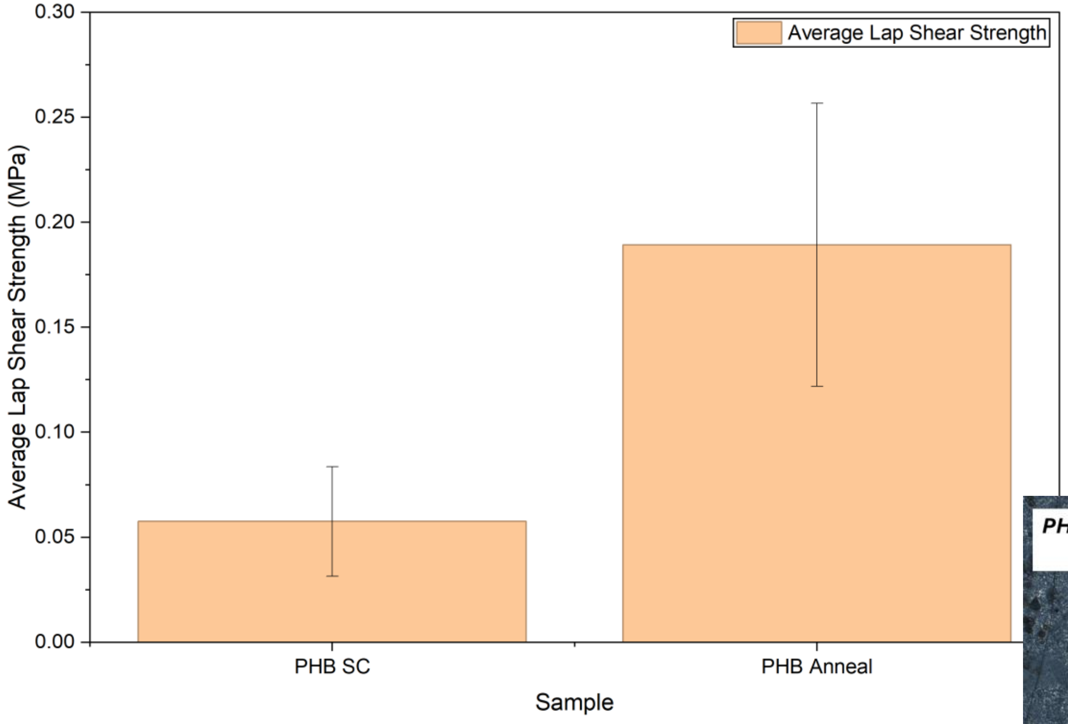
- Dan Scheiman, GRC, Thermal Data
- Laura Wilson & Rich Martin, GRC, Microscopy
- NASA STMD CIF program
- Mango Materials
- Frances Donovan & the NASA CUBES Team

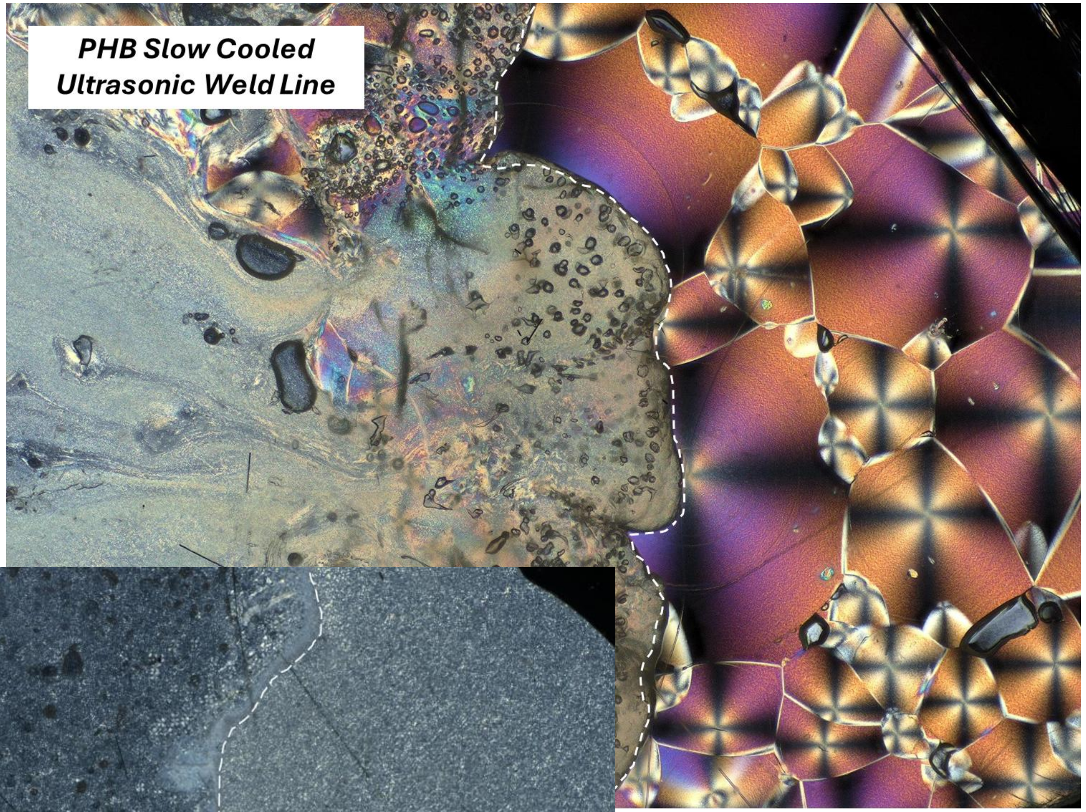


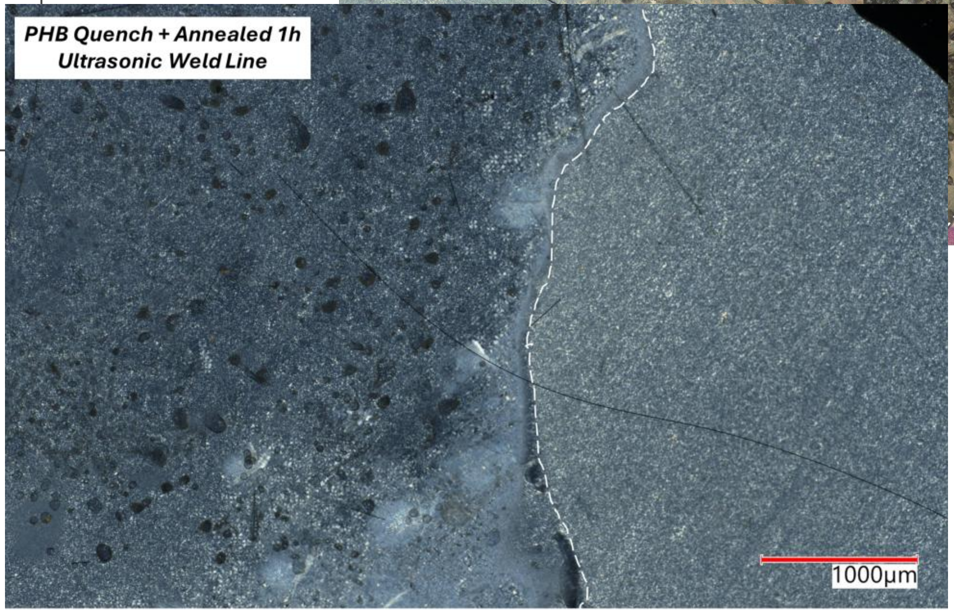
Questions?

Backup Slides

Ultrasonic Welding for In-Situ Assembly and Repair



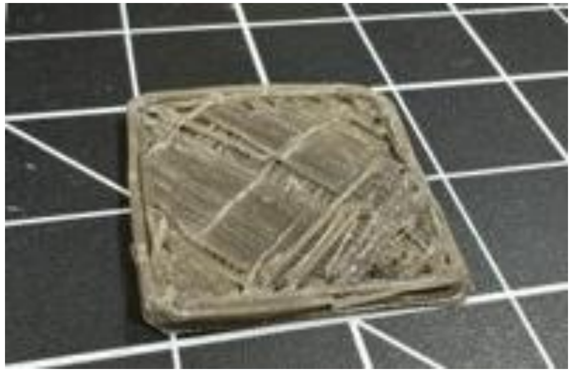


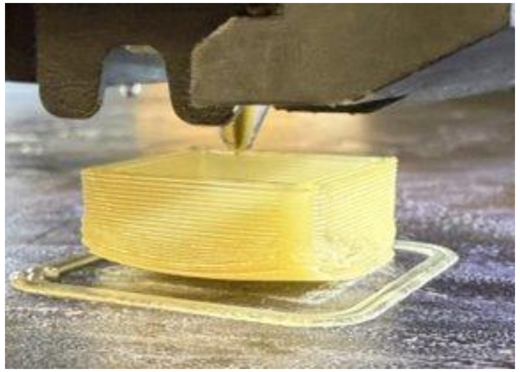


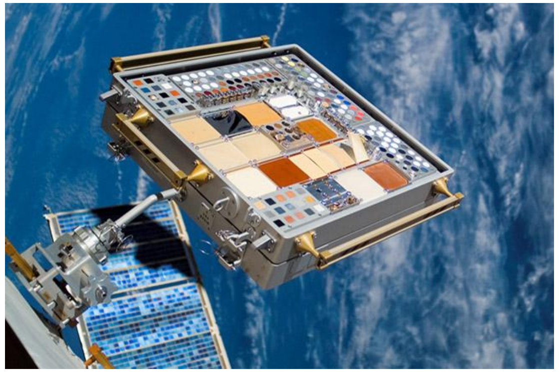
Future Work

Future Work:

- Optimize composite parameters for improved extrusion stability
- Explore lower regolith loadings & alternative simulants
- Investigate additives to tune PHB melt behavior
- Expand PHB printing trials into more complex geometries
- Continue environmental testing on printed parts

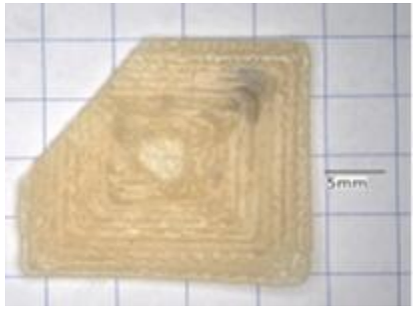


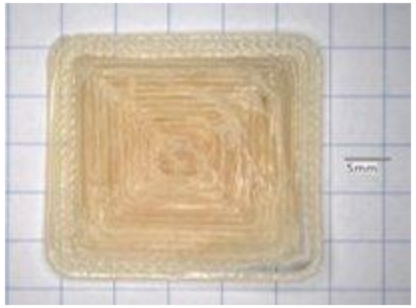


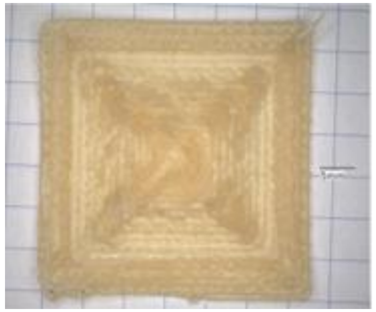


Conclusion

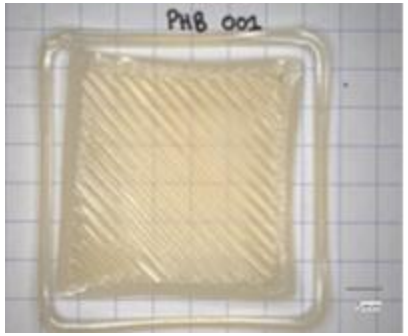




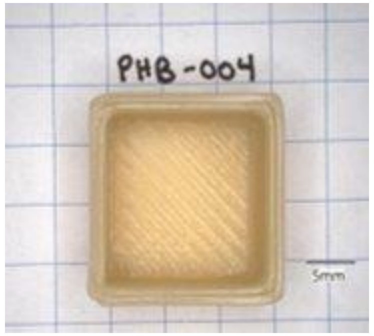
Conclusion

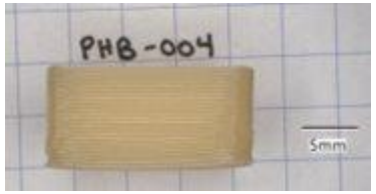

PHB 001

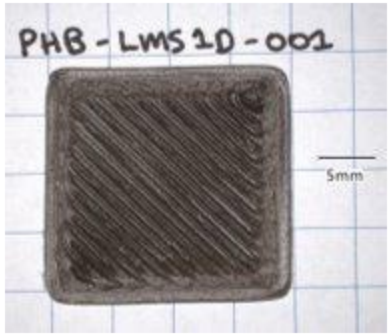

PHB 001 (w GLUE)


PHB 002


PHB -003


PHB-004
5mm


PHB-004
5mm

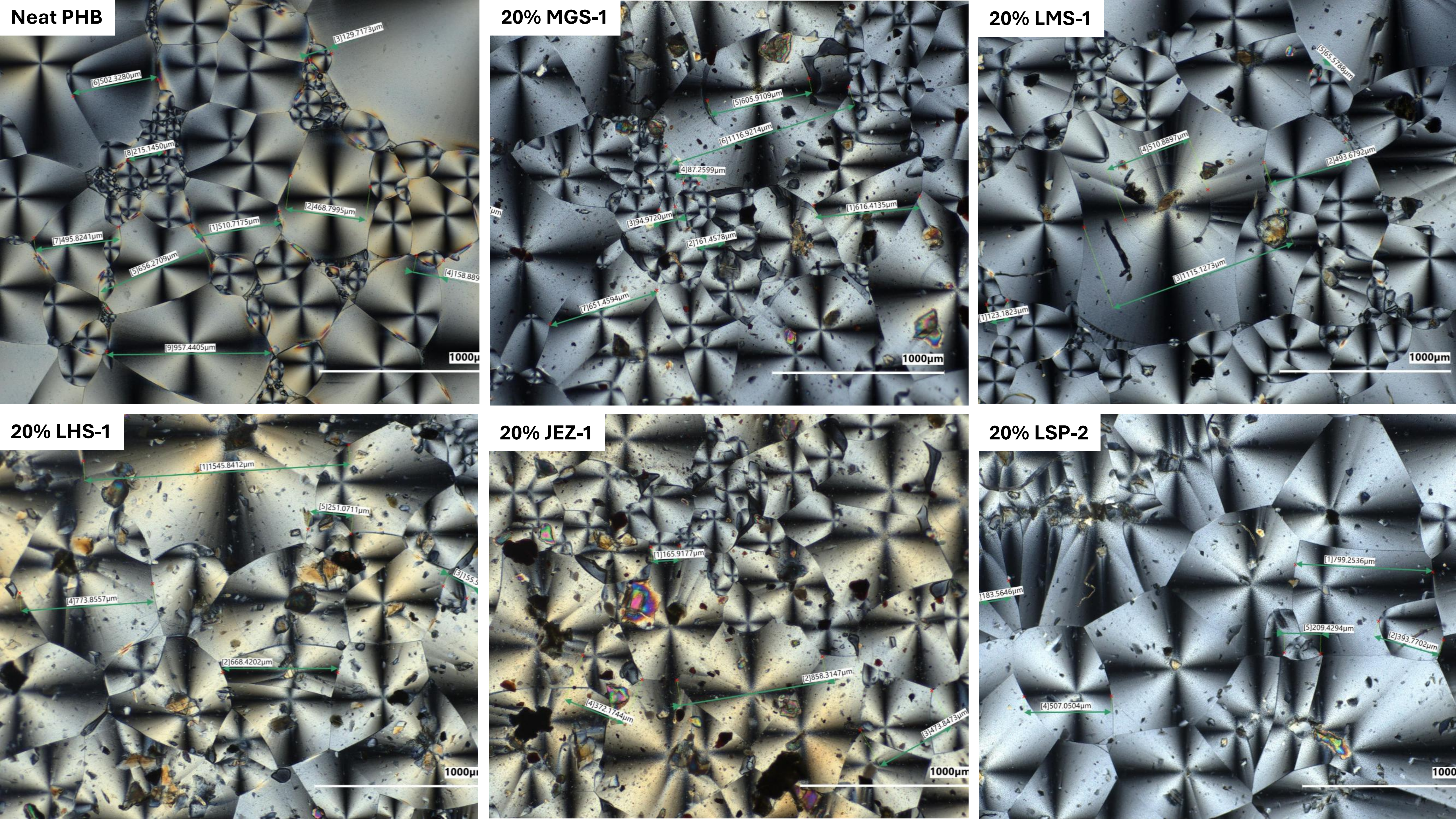

PHB - LMS1D - 001
5mm

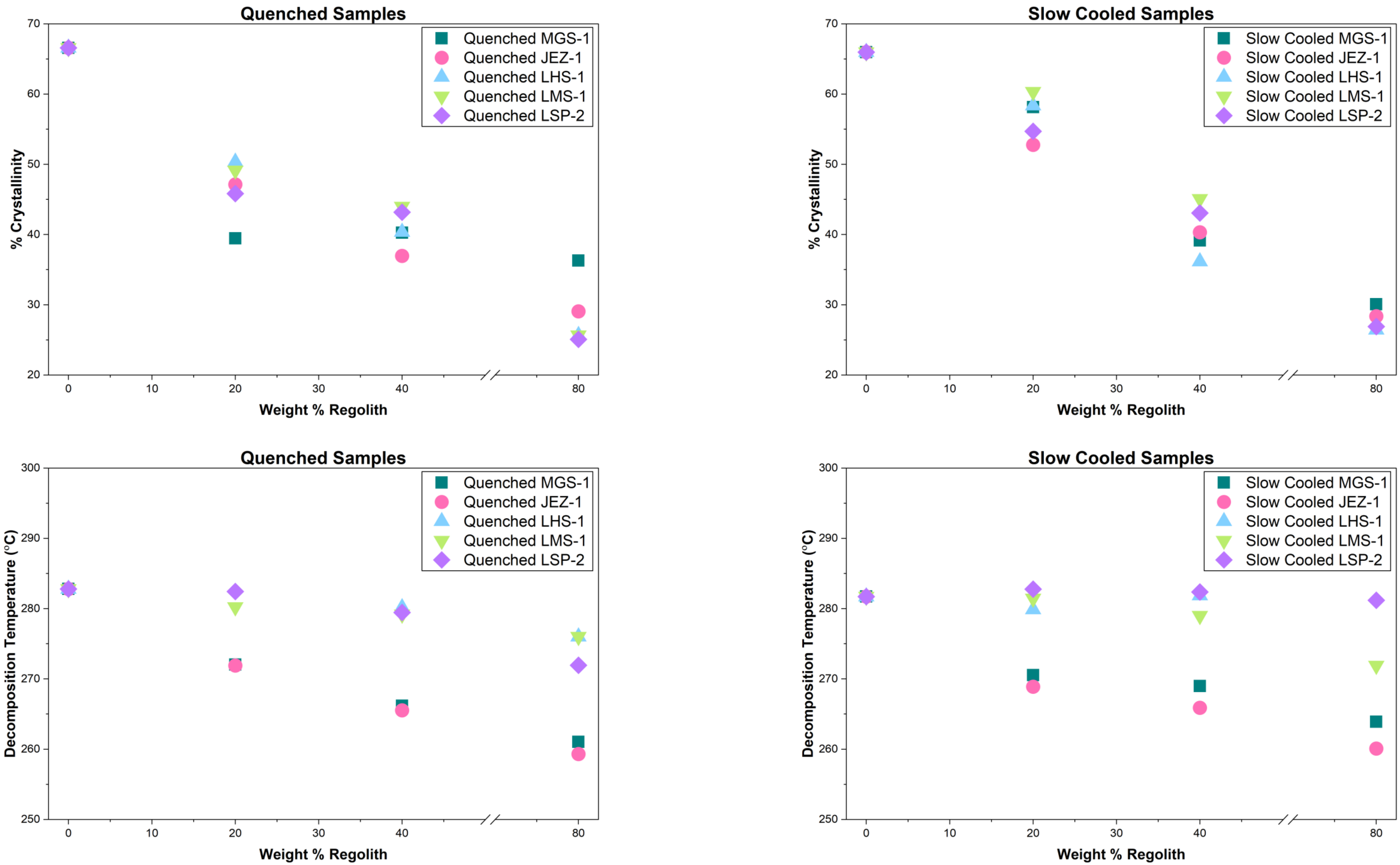

PHB-LMS1D-002
5mm


PHB -LMS1D-003
5mm

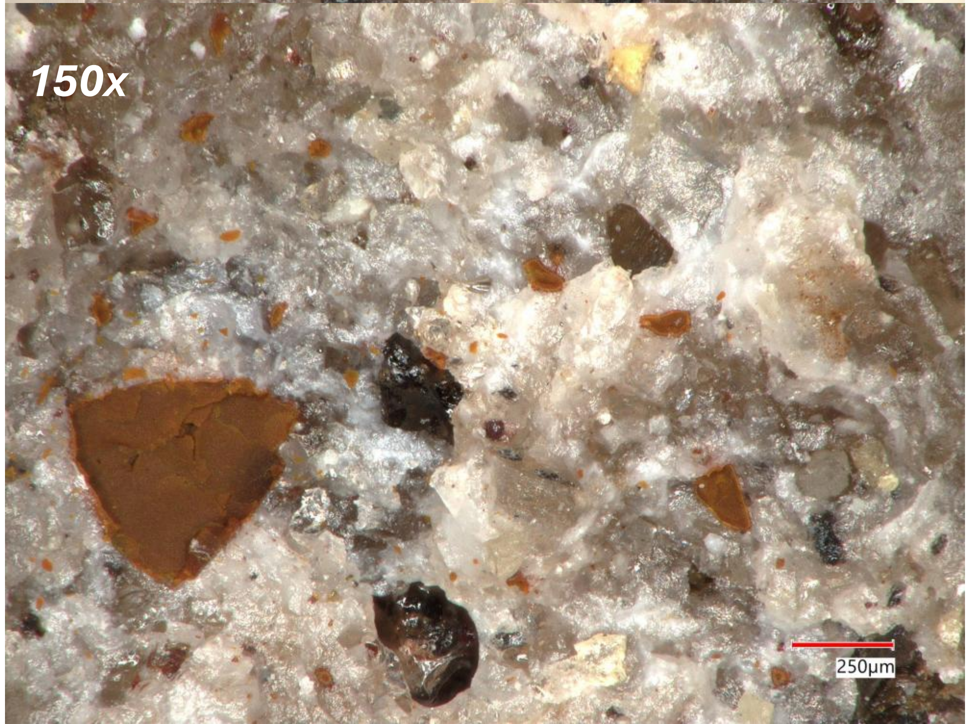

PHB-LMS1D-004 (FAIL)
5mm


PHB-LMS1D-005
5mm


Neat PHB
[6]502.3280μm
[3]129.7173μm
[8]215.1450μm
[2]468.7995μm
[1]510.7175μm
[7]495.8241μm
[5]656.2709μm
[9]957.4405μm
1000μm
20% MGS-1
[5]605.9109μm
[6]1116.9214μm
[4]87.2599μm
[1]616.4135μm
[3]94.9720μm
[2]161.4578μm
[7]651.4594μm
1000μm
20% LMS-1
[5]65.5788μm
[4]510.8897μm
[2]493.6792μm
[3]1115.1273μm
[1]123.1823μm
1000μm
20% LHS-1
[1]1545.8412μm
[5]251.0711μm
[4]773.8557μm
[2]668.4202μm
20% JEZ-1
[1]165.9177μm
[2]858.3147μm
[4]372.1744μm
[3]473.8473μm
1000μm
20% LSP-2
[1]799.2536μm
[5]209.4294μm
[2]393.7702μm
[4]507.0504μm

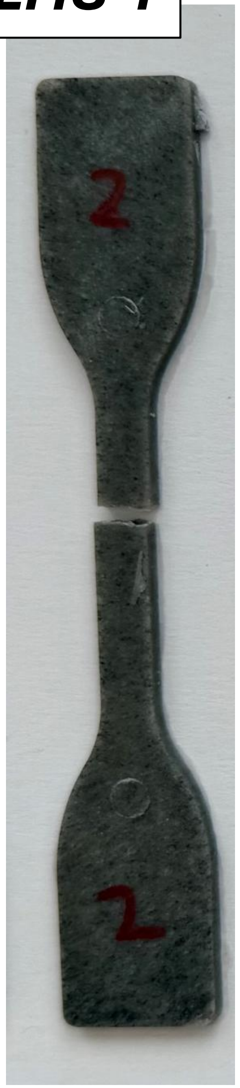


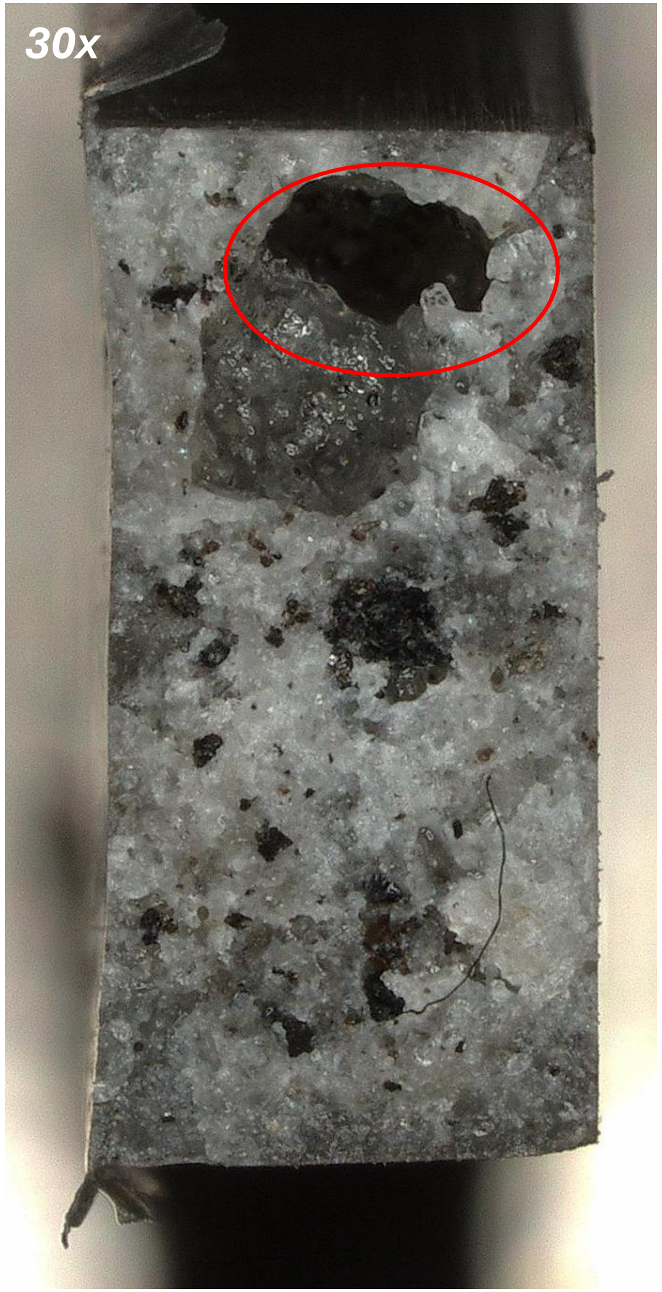
Quenched Samples
% Crystallinity
Weight % Regolith
Quenched MGS-1
Quenched JEZ-1
Quenched LHS-1
Quenched LMS-1
Quenched LSP-2
70
60
50
40
30
20
0
10
20
30
40
80
Slow Cooled Samples
% Crystallinity
Weight % Regolith
Slow Cooled MGS-1
Slow Cooled JEZ-1
Slow Cooled LHS-1
Slow Cooled LMS-1
Slow Cooled LSP-2
Quenched Samples
Decomposition Temperature (°C)
Weight % Regolith
300
290
280
270
260
250
Slow Cooled Samples
Decomposition Temperature (°C)
Weight % Regolith

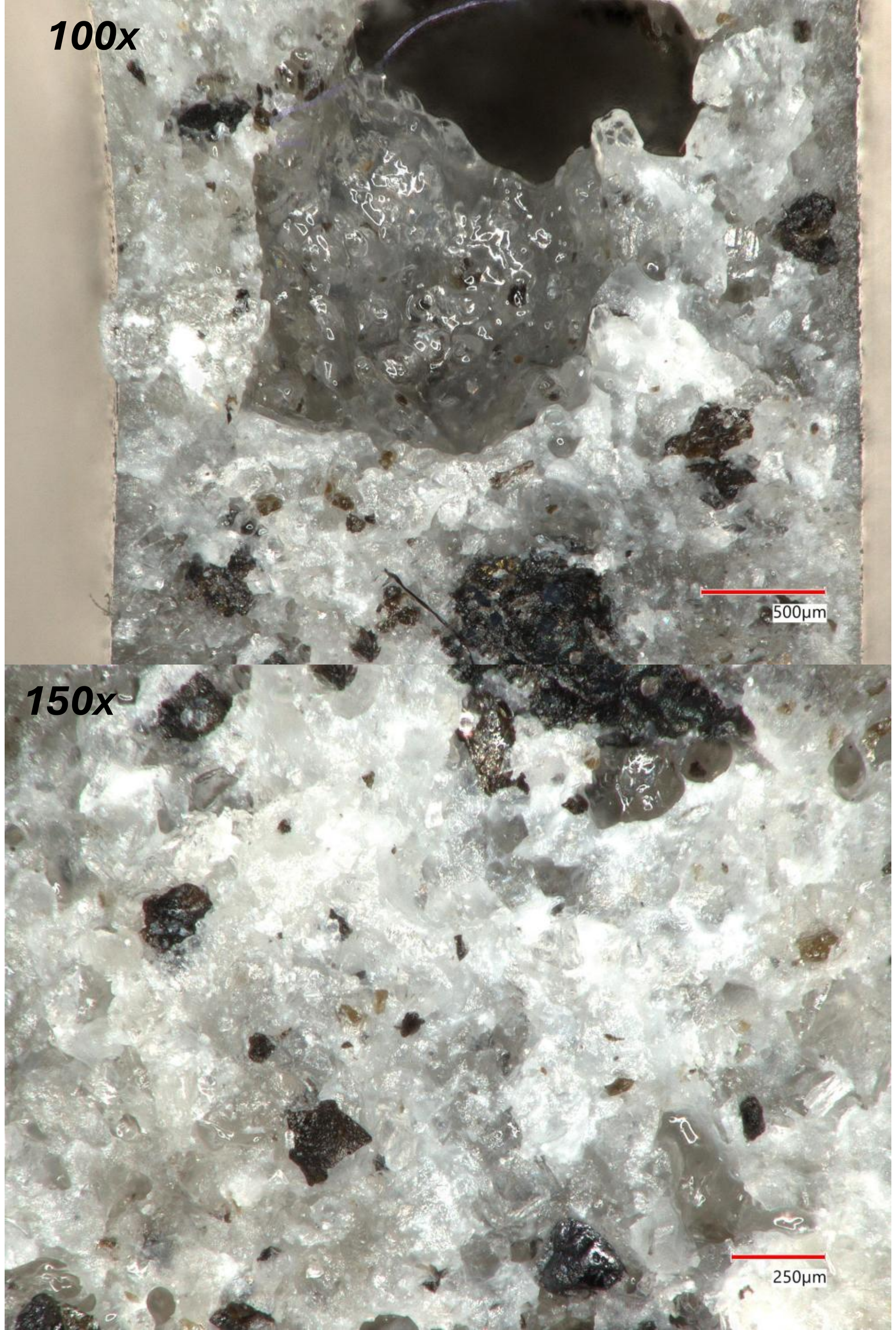


- Smallest average particle size
- Highest Mg, smallest Si content

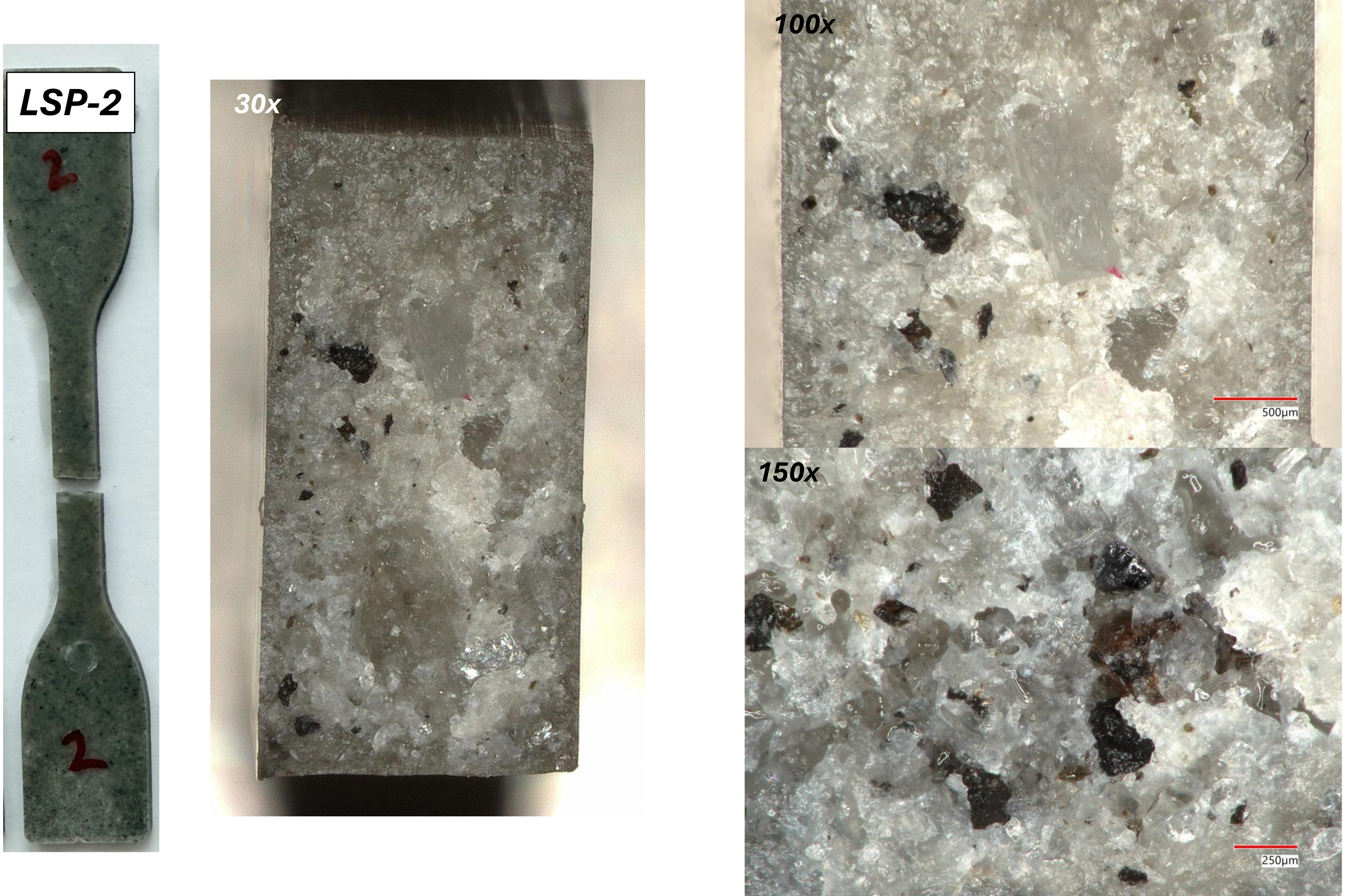
LHS-1



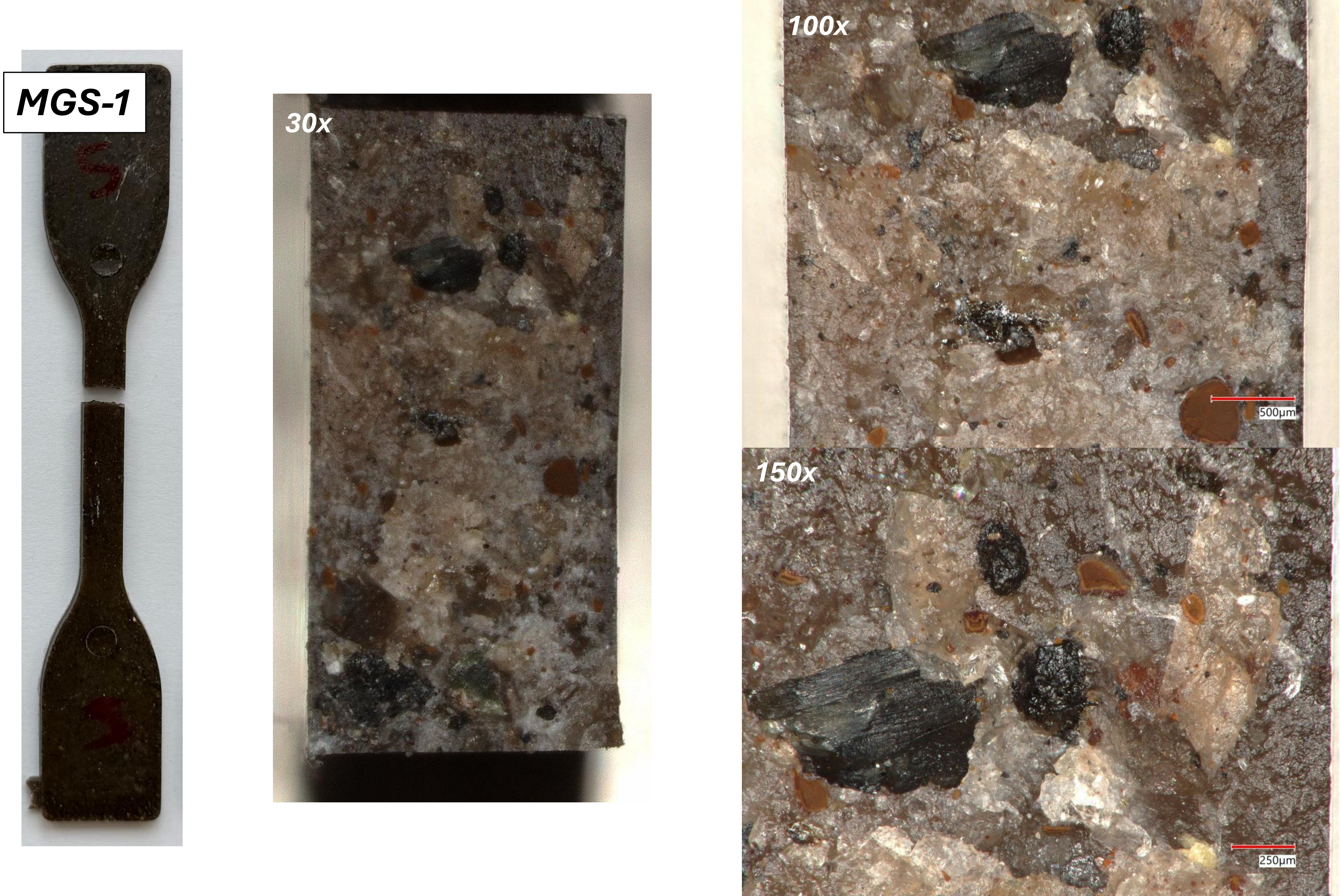





LMS-1
30x
100x
500µm
150x
250µm

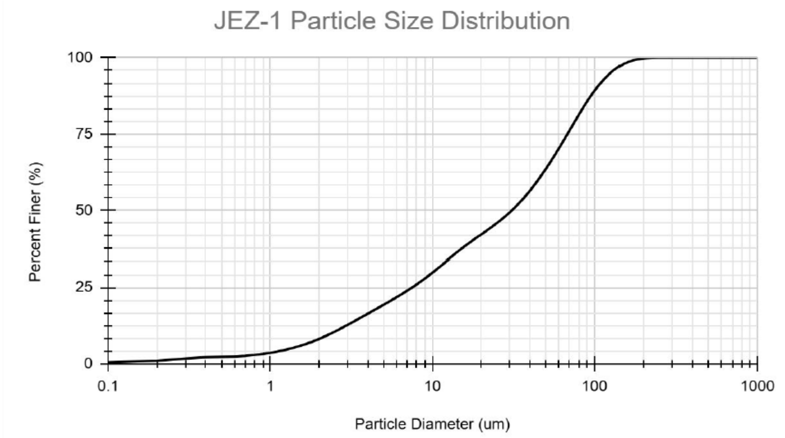


LSP-2
2
2
30x
100x
500µm
150x
250µm


MGS-1
30x
100x
500μm
150x
250μm

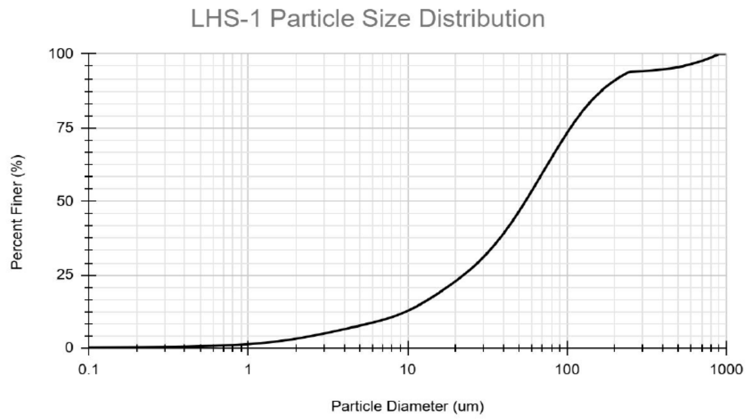
JEZ-1 Jezero Delta Simulant | **Fact Sheet**
003-08-001-1225

Particle Size Distribution
Using a combination of laser and sieve analysis



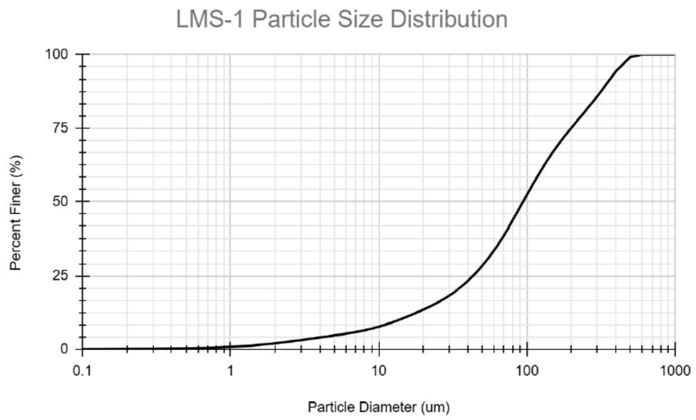
LHS-1 Lunar Highlands Simulant | **Fact Sheet**
003-01-001-1225

Particle Size Distribution
Using a combination of laser and sieve analysis



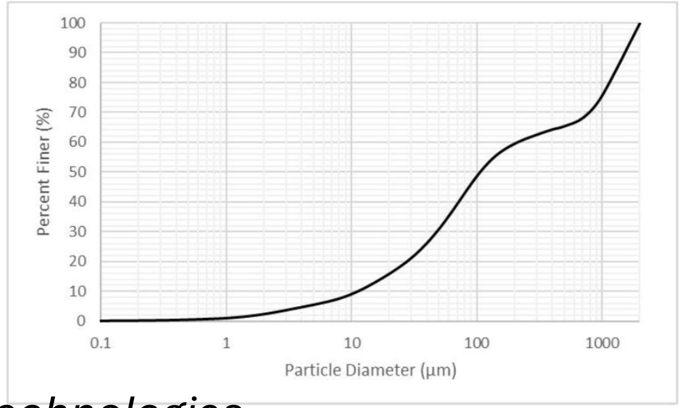
LMS-1 Lunar Mare Simulant | **Fact Sheet**
003-02-001-1225

Particle Size Distribution
Using a combination of laser and sieve analysis



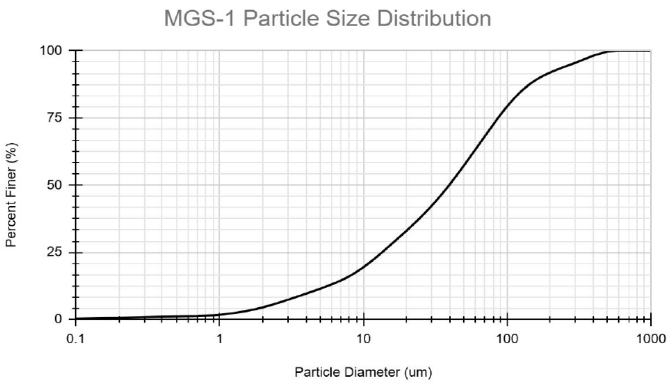
LSP-2 Lunar South Pole Simulant | **Fact Sheet**
001-12-001-1225

Particle Size Distribution
Using a combination of laser and sieve analysis



MGS-1 Mars Global Simulant | **Fact Sheet**
003-05-001-1225

Particle Size Distribution
Using a combination of laser and sieve analysis



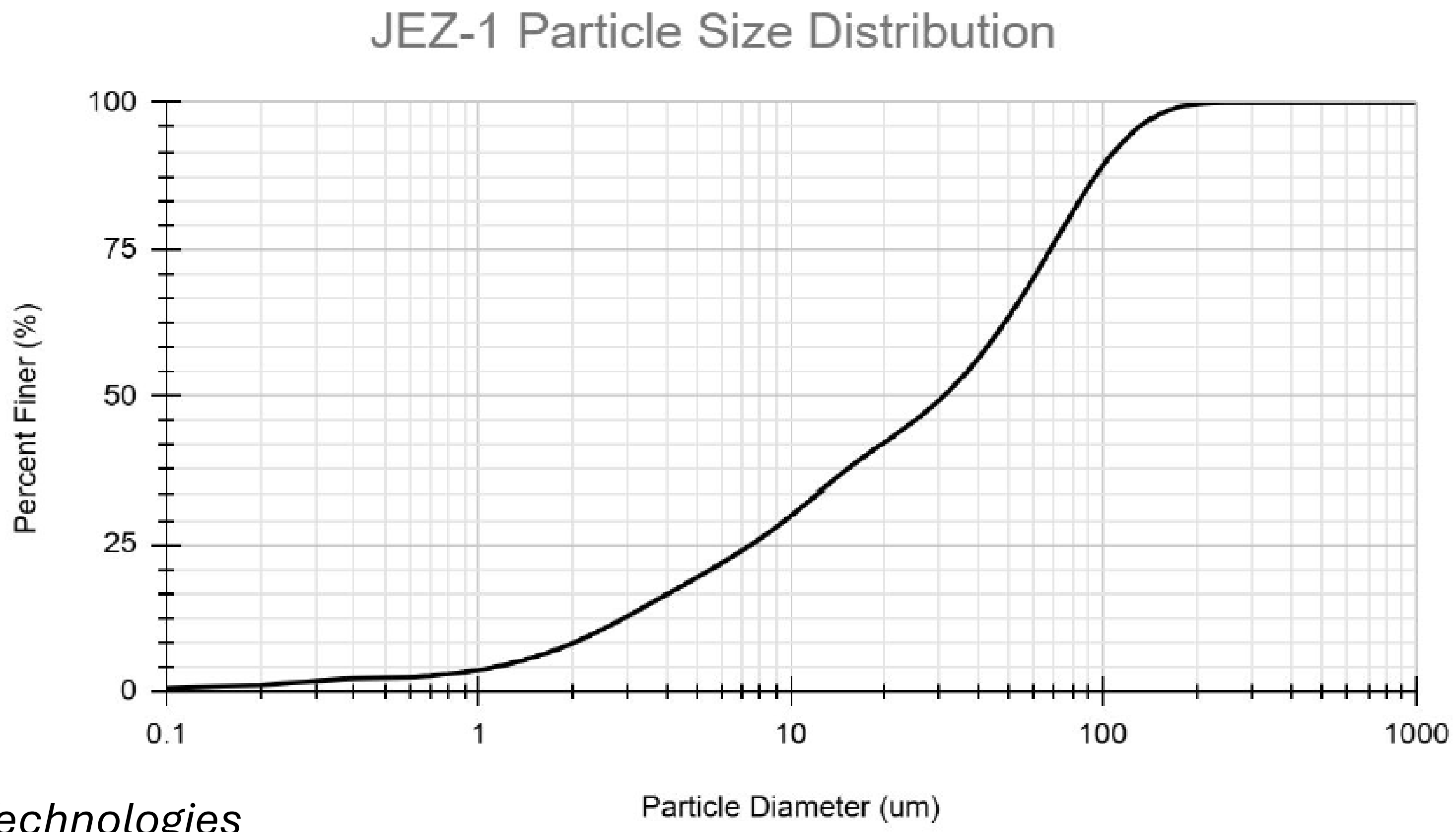
Source: Space Resource Technologies

JEZ-1 Jezero Delta Simulant | **Fact Sheet**

003-08-001-1225

Particle Size Distribution

Using a combination of laser and sieve analysis



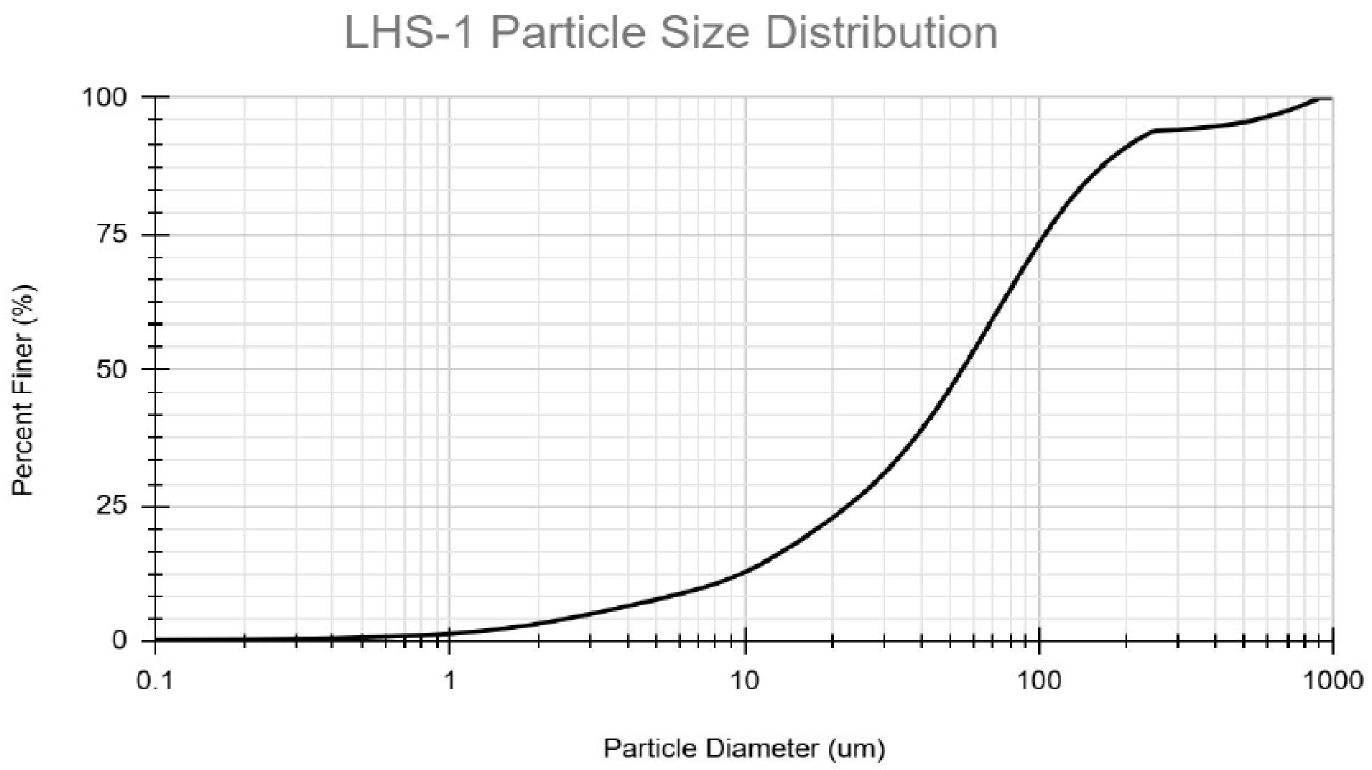
Source: Space Resource Technologies

LHS-1 Lunar Highlands Simulant | **Fact Sheet**

003-01-001-1225

Particle Size Distribution

Using a combination of laser and sieve analysis



Source: Space Resource Technologies

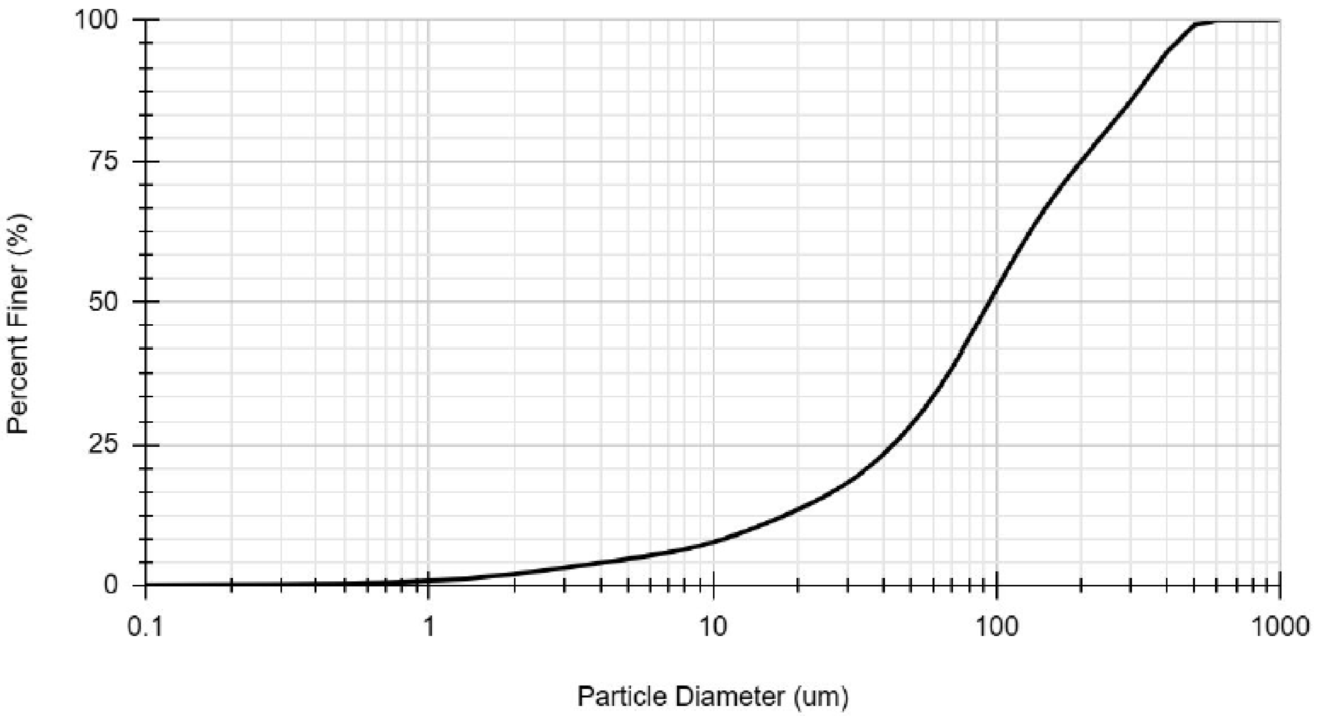
LMS-1 Lunar Mare Simulant | **Fact Sheet**

003-02-001-1225

Particle Size Distribution

Using a combination of laser and sieve analysis

LMS-1 Particle Size Distribution



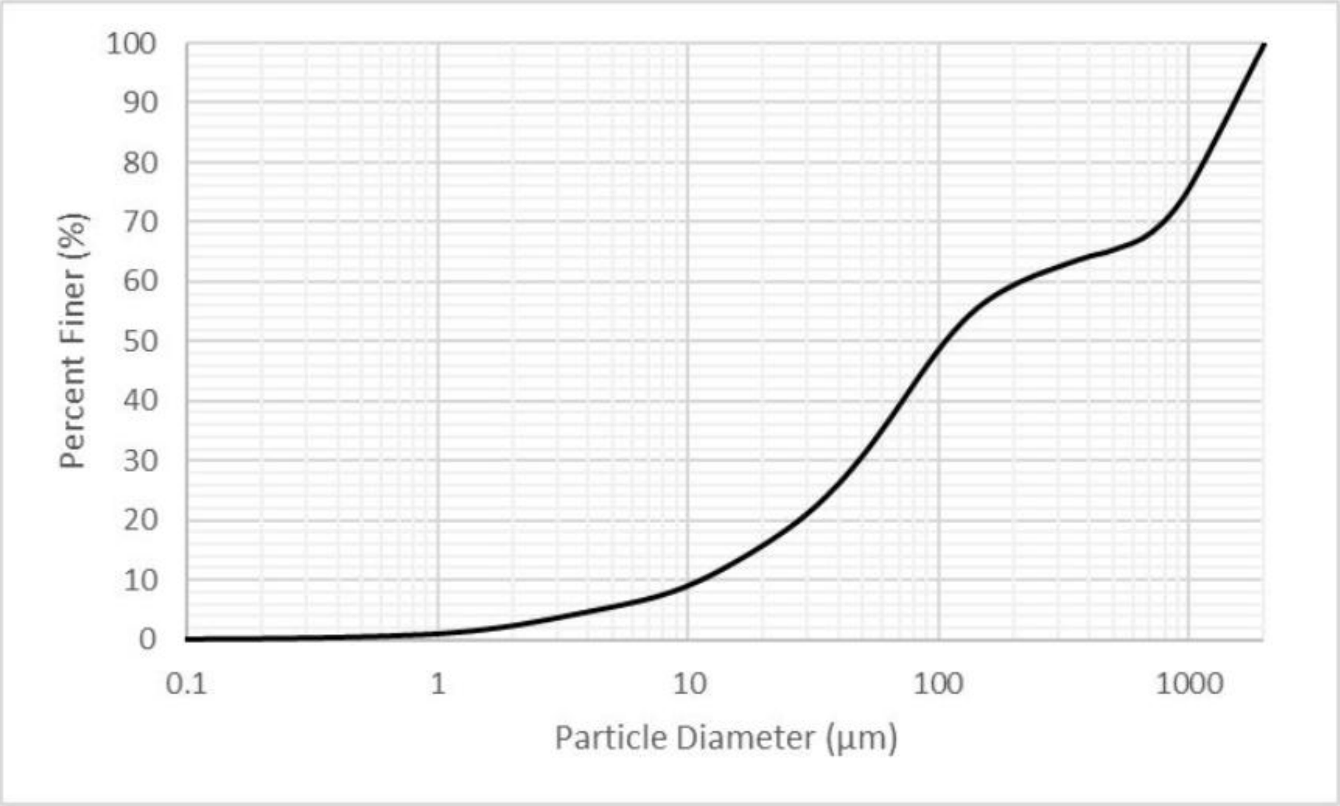
Source: Space Resource Technologies

LSP-2 Lunar South Pole Simulant | **Fact Sheet**

001-12-001-1225

Particle Size Distribution

Using a combination of laser and sieve analysis

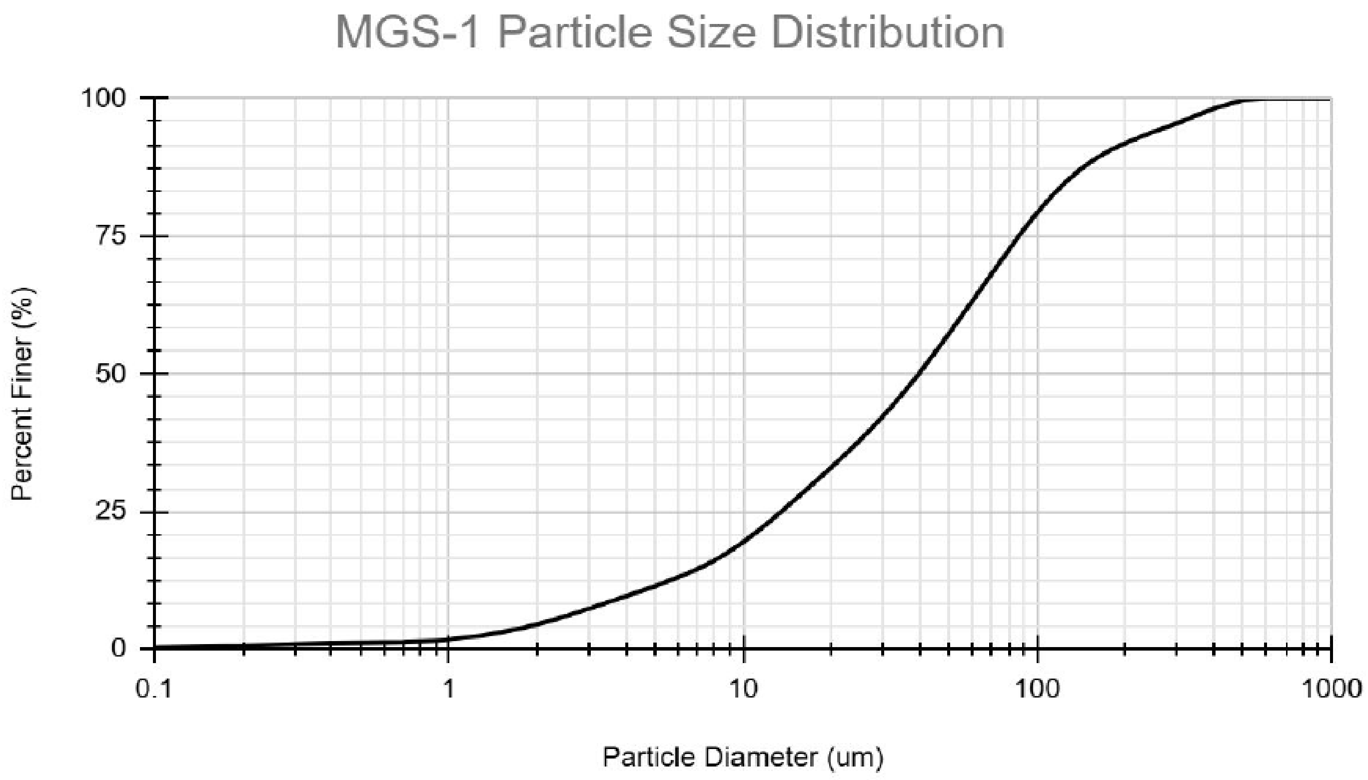


Source: Space Resource Technologies

MGS-1 Mars Global Simulant | **Fact Sheet**
003-05-001-1225

Particle Size Distribution

Using a combination of laser and sieve analysis



Source: Space Resource Technologies