Bringing the heat: thermal protection systems for low Earth orbit transportation and Lunar exploration

Nathaniel Olson / NASA Johnson Space Center

UCSB Materials Colloquium

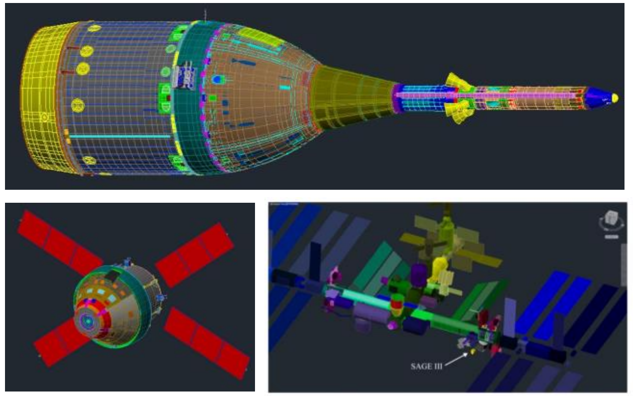
October 25th, 2024

NASA Johnson Space Center

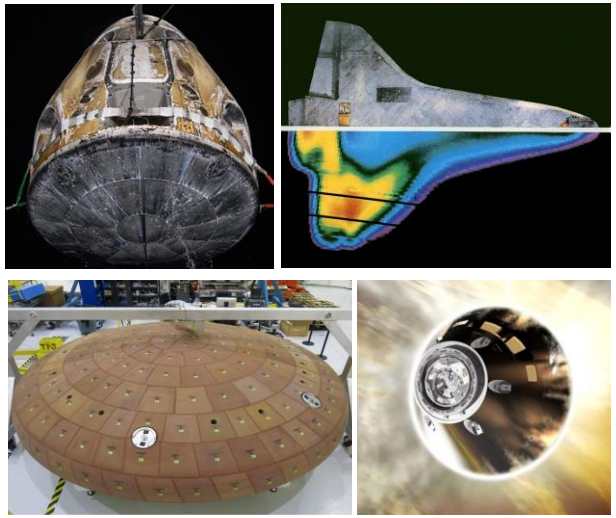
Located in Houston, TX
>3,200 civil servants and >11,000 contractors

Leads NASA's human spaceflight programs and associated research & development

NASA Johnson Space Center
↳ Engineering Directorate
↳ Structural Engineering Division
↳ Thermal Design Branch



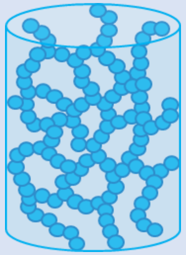
Passive thermal control maintains spacecraft temperatures without using pumped fluids



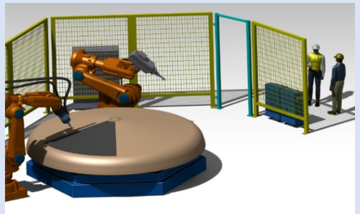
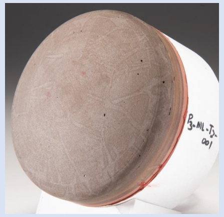
Thermal protection systems (TPS) protect a spacecraft from the heating encountered during hypersonic flight through a planetary atmosphere.

Background on TPS


Thermally Stable Aerogels

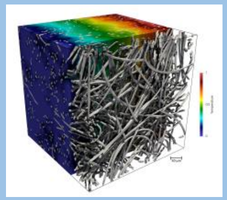
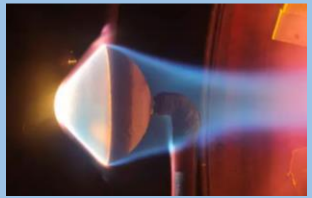



Additively Manufactured Thermal Protection Systems

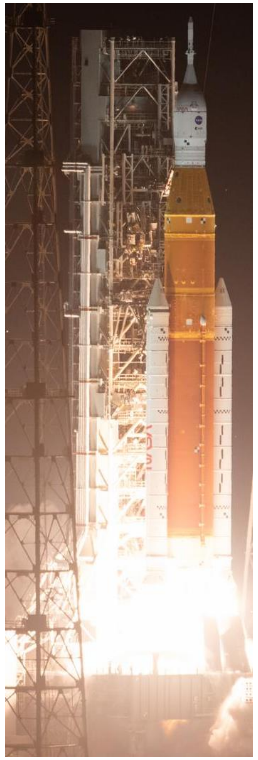
Silica Fiber Characterization for Reusable TPS


Program Support and Challenges Facing TPS

High velocities and extreme environments of spaceflight necessitate **thermal protection systems** (TPS)

Launch and Ascent



Aborts

Plume Heating
Aerodynamic Heating

Returning from Low-Earth-Orbit
7 km/sec

High
Aerodynamic Heating

Returning from Moon
11 km/sec

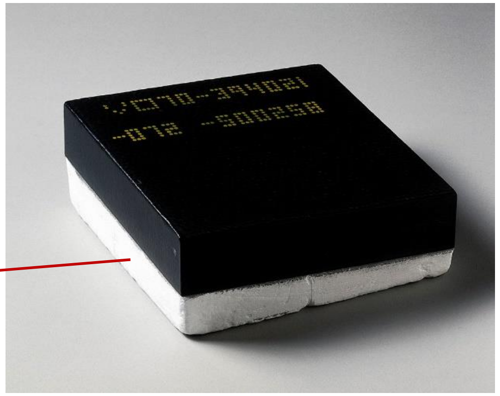
Very High
Aerodynamic Heating

Convective Heating Scales with Velocity^3
Shock-Layer Radiation Scales with Velocity^8

$$\dot{q}_{conv} \propto v^3 \left(\frac{\rho}{R}\right)^3$$

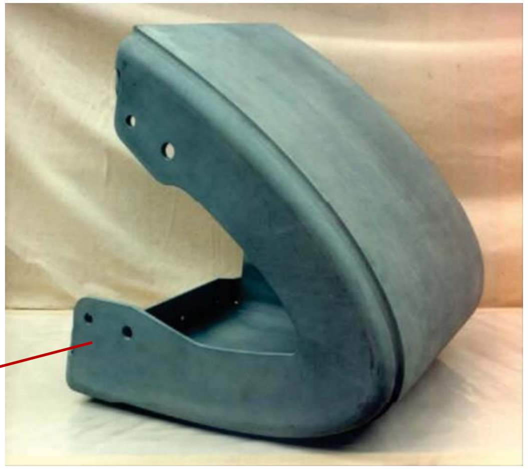
$$\dot{q}_{rad} \propto v^8 \rho^{1.2} \mathrm{R}^{0.5}$$

Insulative TPS



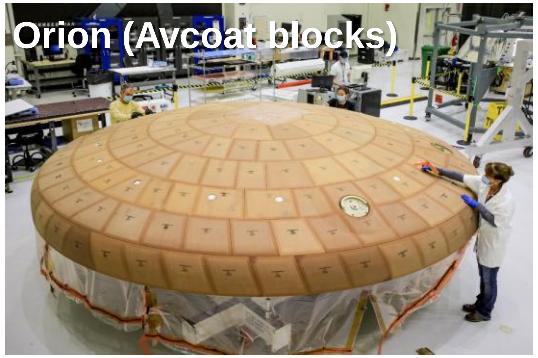
Energy management through **storage and re-radiation (material remains unchanged).**

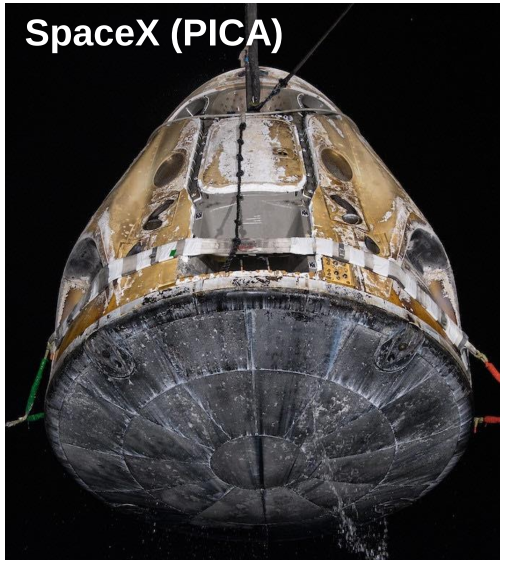
Hot Structures

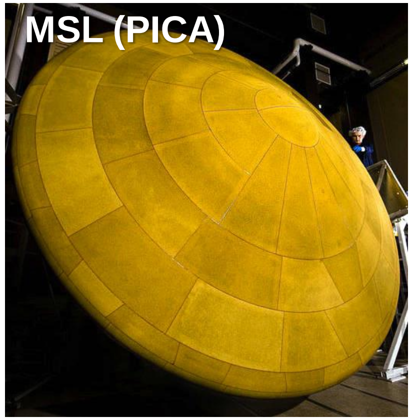


Energy management through **re-radiation of heat.**

Ablative TPS





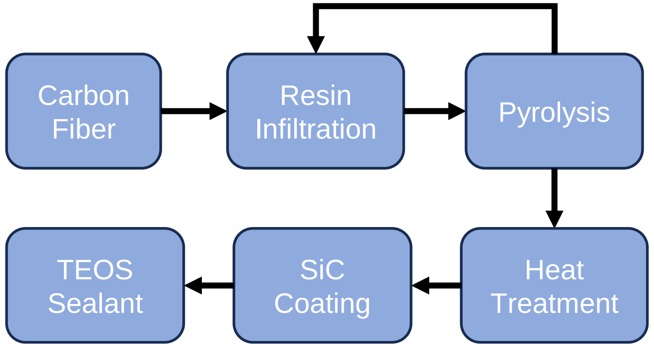


Energy management through **controlled material consumption, charring, and re-radiation.**

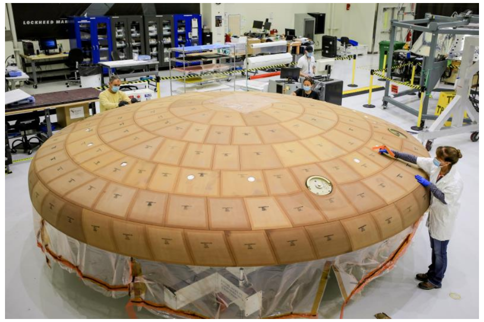
TPS = Thermal Protection **System**

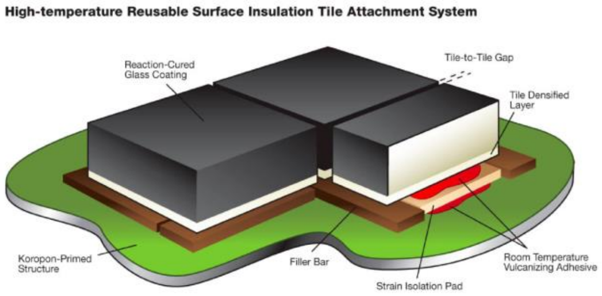
Material

Manufacturing



Integration



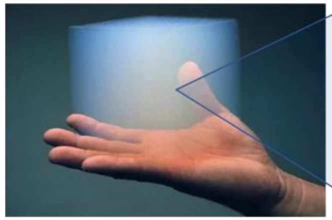


Operation

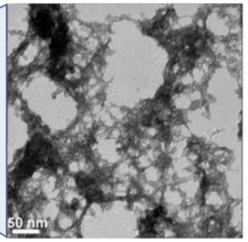
Improving aerogel thermal stability & understanding of structural evolution

Project Objective

Develop aerogel to maintain **porosity** and **surface area** at high temperatures (≥1200°C) for use as insulation in next-gen aerospace applications

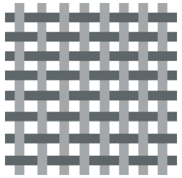


Silica aerogel block (NASA JPL)



Highly porous network of interconnected nanoparticles

Aerogels = Ultra-low thermal conductivity and weight



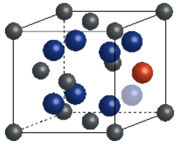
Development of Thermally Stable Aerogels

Composite Manufacturing & Testing

Aim 1

Composition & Material Properties

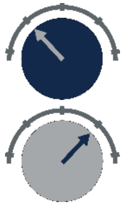
- Doped zirconia aerogels with Y, Yb, Gd, Ce, and Ca
- Study the evolution and connect to **material properties**



Aim 2

Synthetic Parameters

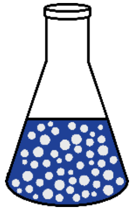
- Finely **tune pore structure** via synthetic parameters
- Impact of starting structure on subsequent evolution



Aim 3

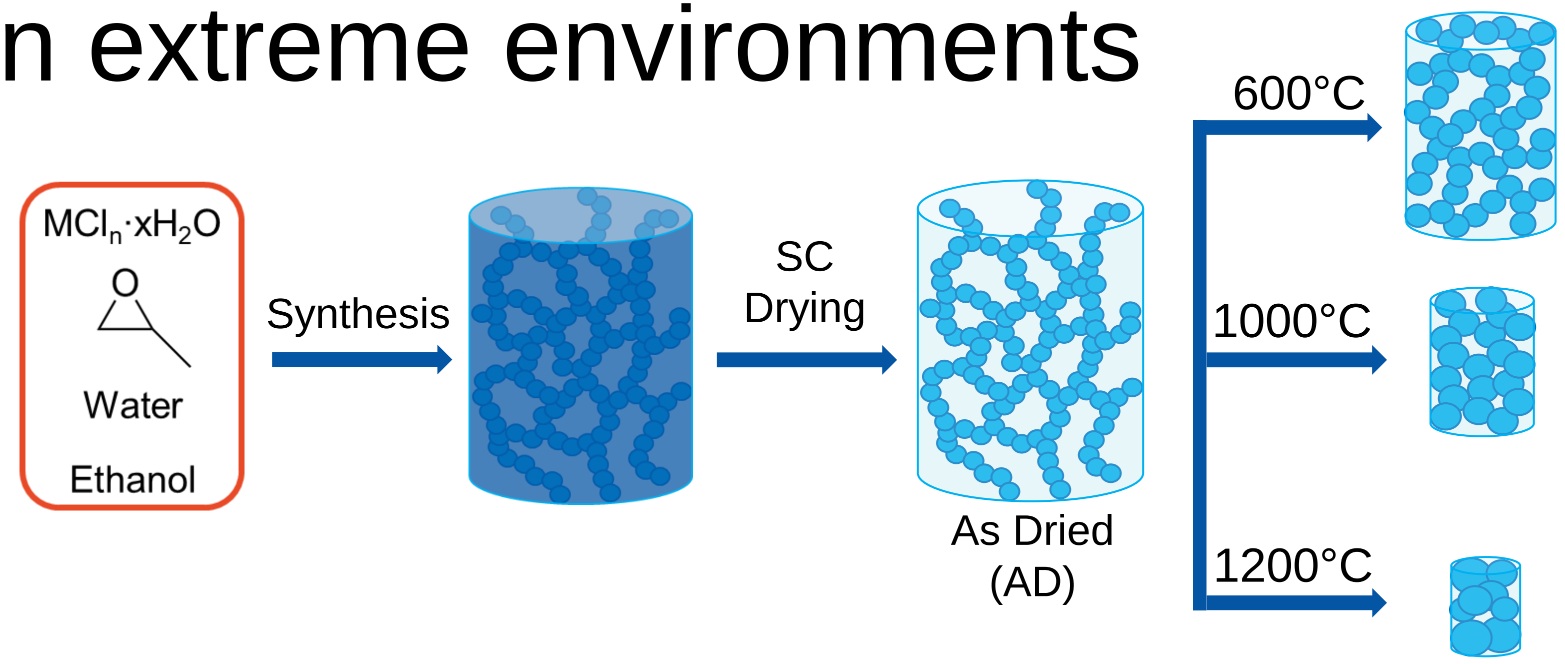
Synthetic Techniques

- Devise new synthetic techniques from **colloidal precursors**
- Post-synthetically modify aerogel with **MO_x coatings**



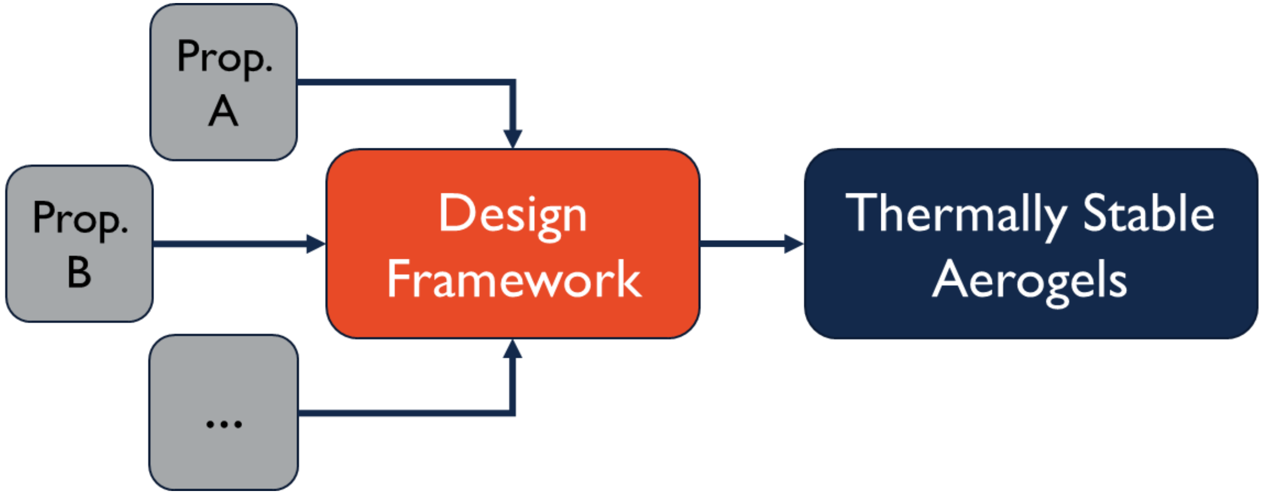
Aim 1: Olson, N., et al. (2021). *JACerS*, 104 (8), 4190-4202.
Olson, N., et al. (2023). *JACerS*, 106(12), 7745-7759.
Aim 2: Olson, N., Meyer, J., et al. (2024). *JSST*, 110, 607-618.
Aim 3: Olson, N., Stokes, J., et al. (2024). *JACerS*, 107(9), 6353-6368.

Characterizing the structural evolution of aerogels in extreme environments



Characterization via:
Nitrogen Physisorption
SEM
XRD
TEM
FTIR

Connect material performance to material properties to build a **kinetically & thermodynamically informed design framework** for thermally stable aerogels.

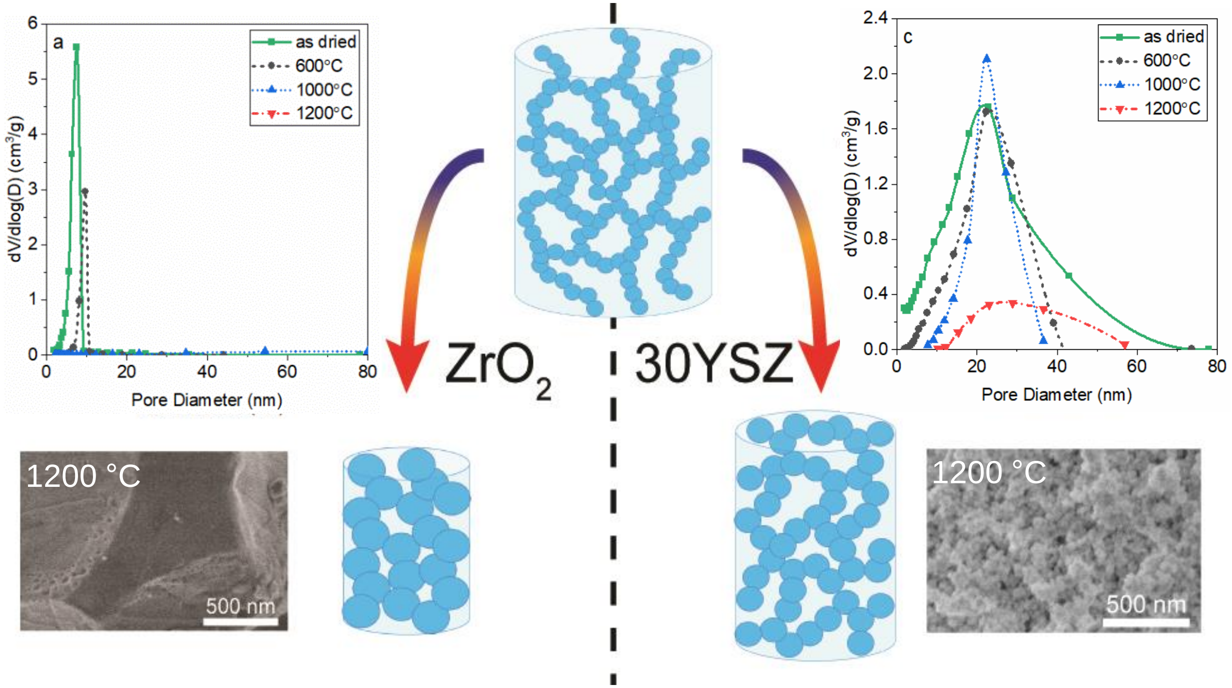


High yttria concentration improves thermal stability in yttria-stabilized zirconia (YSZ) aerogels

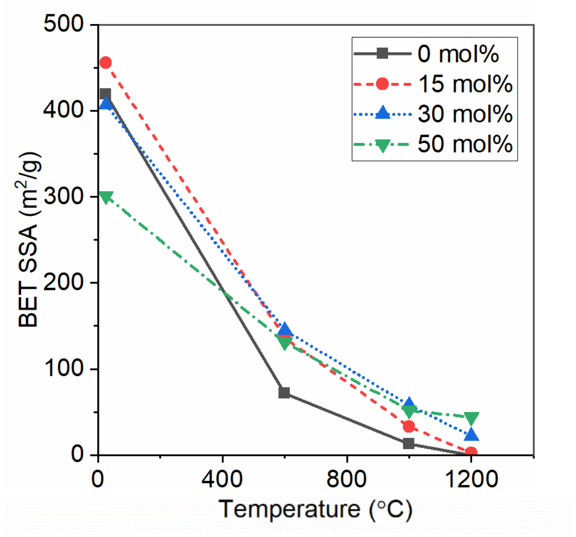
Studied YSZ aerogels at 0, 15, 30, and 50 mol% $YO_{1.5}$ in ZrO_2

Mesoporosity eliminated at 1000 °C

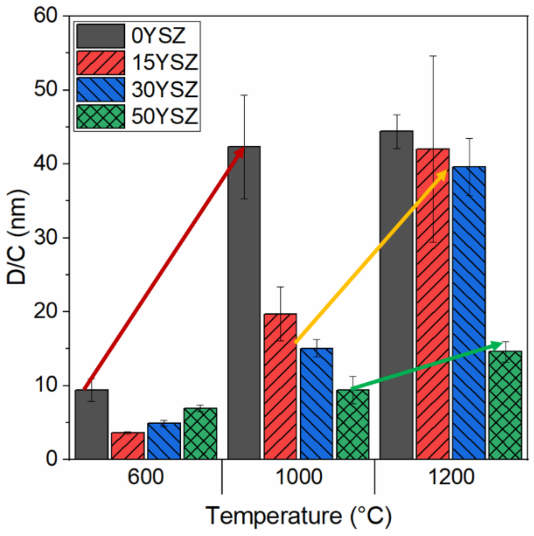
Mesoporosity remains to 1200 °C



Increases BET SSA at high temperatures



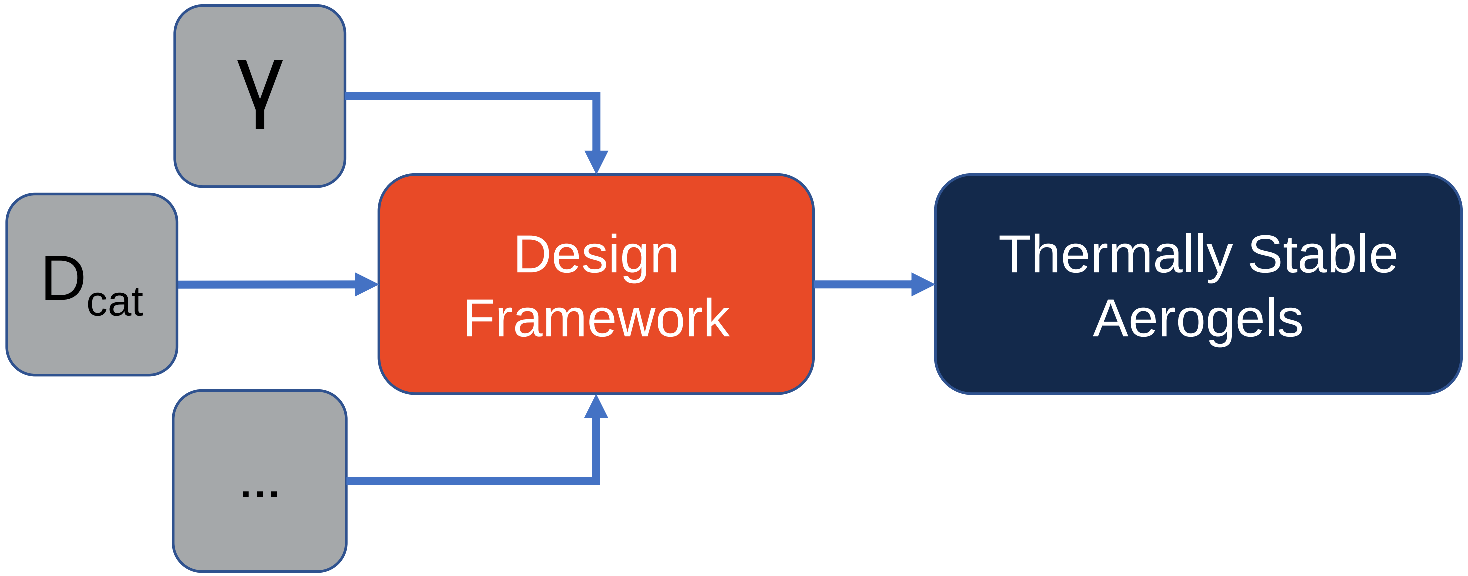
Inhibits crystallite growth



Doping ZrO_2 with > 30 mol% $YO_{1.5}$ improved the stability of the pore structure to 1200 °C.

A design framework for thermally stable aerogels informed by kinetics and thermodynamics

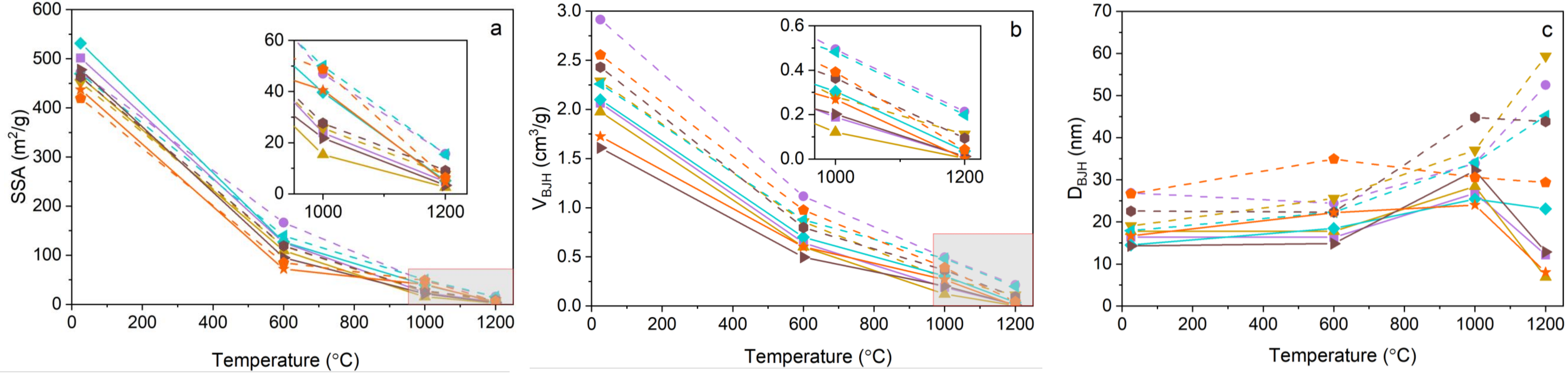
1. **Decreased cation diffusivity**[1,2,3] of Zr^{4+}, Y^{3+} in YSZ with increased Y_2O_3 doping responsible for decreased mass flow
   - *Less densification AND crystallite/grain growth* (reduced rate of mass flow)
2. **Lower surface energy**[4] with increased yttria content ($\gamma_c < \gamma_t < \gamma_m$) leads to:
   - *Improved stability of pore structure* (lower driving force for densification)

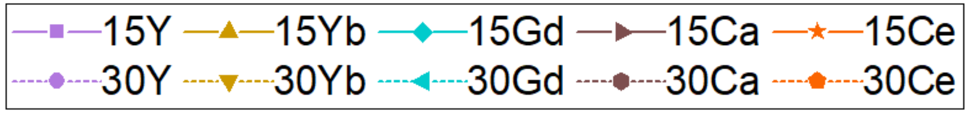


1. Kilo, M., et al. (2000). *Journal of the European Ceramic Society*, 20(12), 2069-2077.
2. Kilo, M., et al. (1997). *Berichte der Bunsen-Gesellschaft*, 101(9), 1361-1365.
3. Kilo, M., et al. (2003). *Journal of applied physics*, 94(12), 7547-7552.
4. Drazin, J. W., & Castro, R. H. (2015). *Journal of the American Ceramic Society*, *98*(4), 1377-1384.

Evaluation of pore structure with nitrogen physisorption quantifies change in performance

SSA | Pore Volume | Pore Size





<u>Best Performers*</u>

1000 °C: 30Y, 30Gd, 30Ce

1200 °C: 30Y, 30Gd

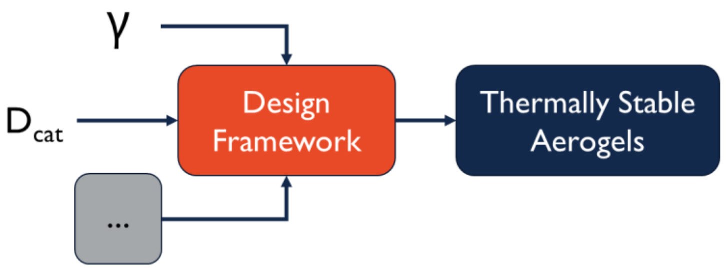
Increased dopant concentration leads to reduced densification.

Attempts to connect material properties to thermal stability thwarted by lack of material property data.

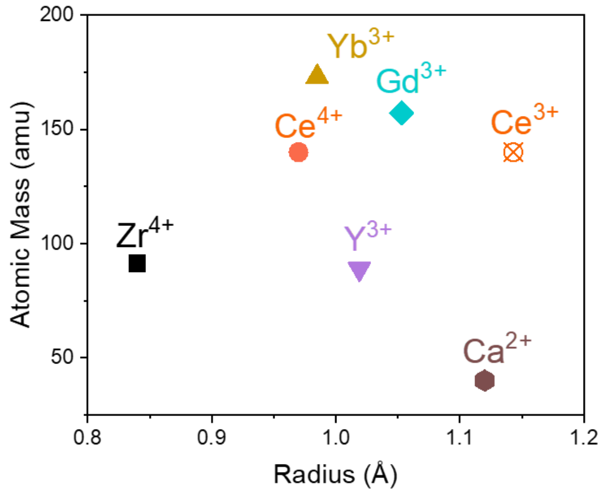
*Best performance dictated by maximum SSA and pore volume at a given temperature

Olson, N.S., et al. (2023). *Journal of the American Ceramic Society*, 106(12), 7745-7759.

Connecting material properties to thermal stability



Given this absence, we turned to something readily available: cation properties (mass, radius, charge)



From our work on YSZ, we were able to connect our results to others' measurements of surface energy and cation diffusivity.

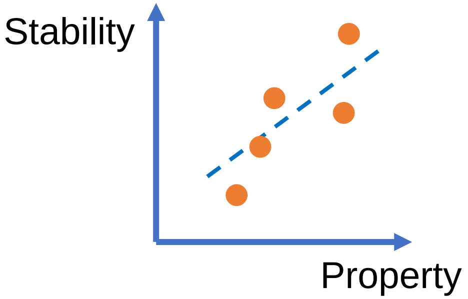
Next, we calculated a weighted average for each material

$$\text{Weighted Property} = x_{Zr}P_{Zr} + x_M P_M$$

$$x_M = \text{mole fraction } MO_y$$
$$P_M = \text{property of dopant } M^{2y+}$$

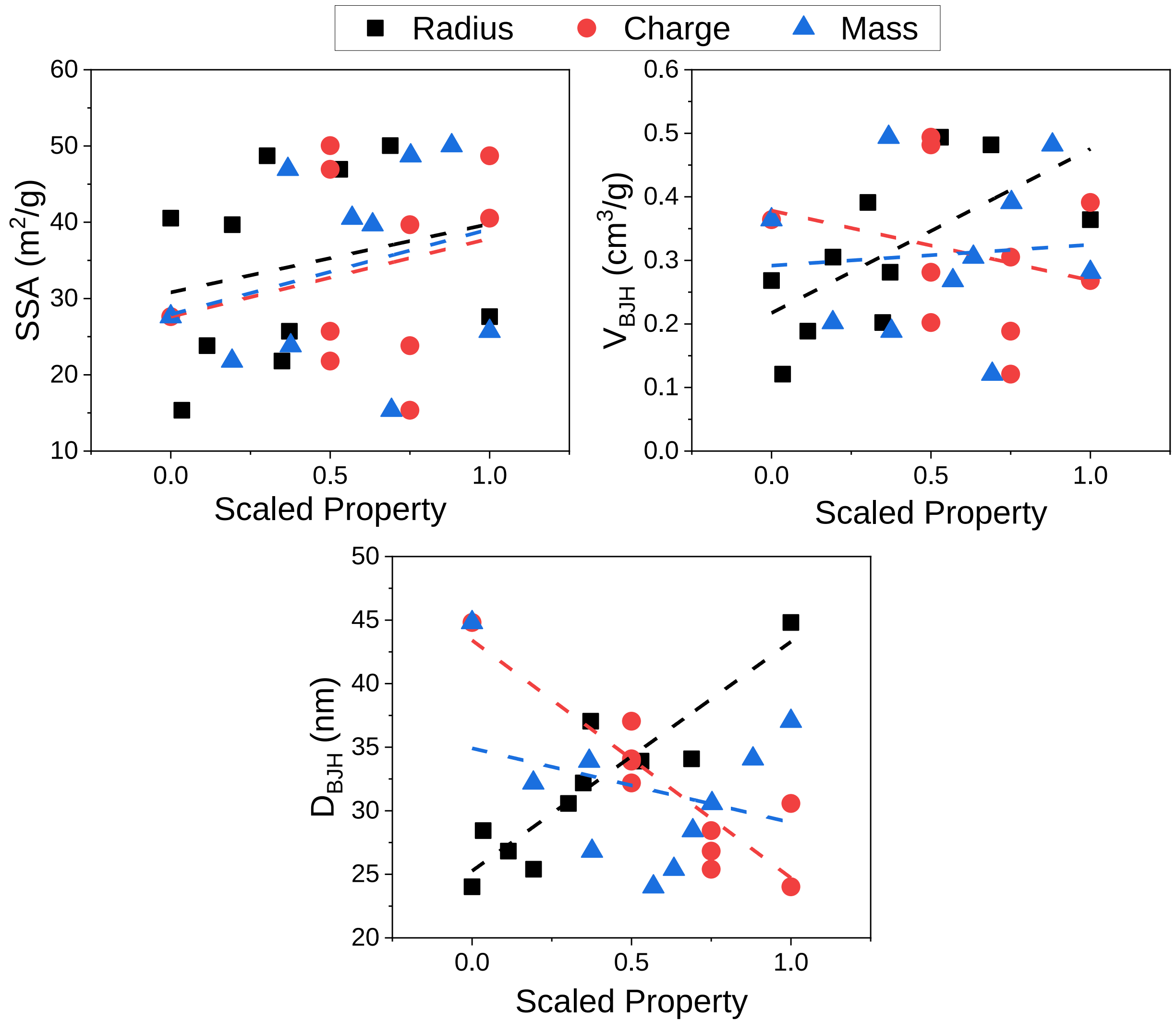
But… neither those properties nor others are available for wider ranges of dopants and concentrations

We then performed linear regression on the **absolute** (property at a given temperature) and **relative** (percent change) thermal stability.



$$\%\ \text{Change} = \frac{x_f - x_i}{x_i}$$

Cation properties are not clearly related to thermal stability (<u>**absolute**</u> or relative)



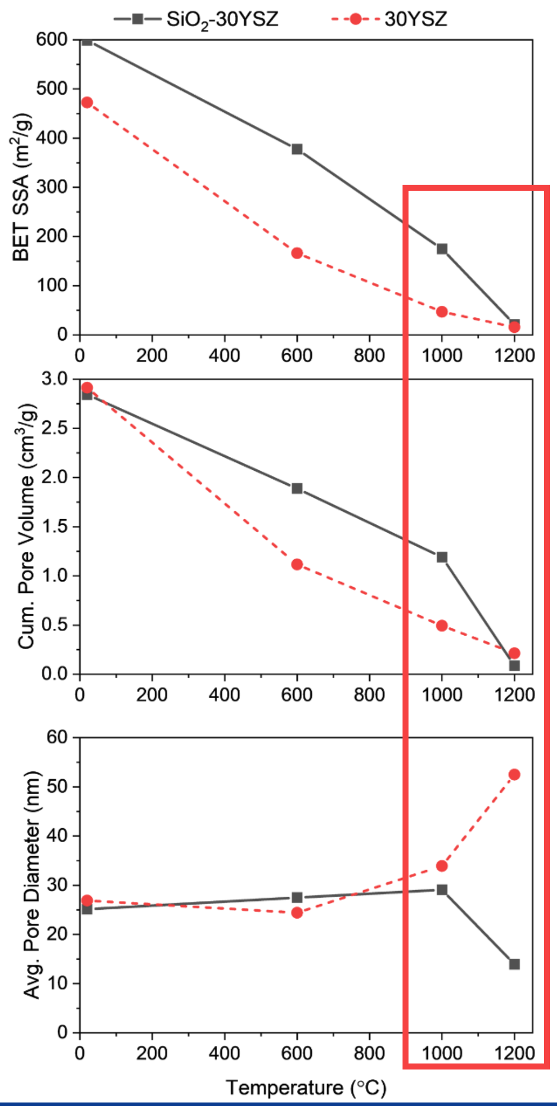
Property	Response		p-value
Radius			0.54
Charge	SSA	1000	0.50
Mass			0.44
Radius			0.04
Charge	V_{BJH}	1000	0.47
Mass			0.8
Radius			3.0E-04
Charge	D_{BJH}	1000	5.9E-04
Mass			0.41

Cation properties are not clearly related to thermal stability (**absolute** or relative)

Property	Response		p-value
Radius			0.54
		1000	0.50
			0.44
			0.04
		1000	0.47
Mass			0.8
Radius			3.0E-04
Charge	D_{BJH}	1000	5.9E-04
Mass			0.41

Wider availability of material property data (such as surface energy, cation diffusivity, etc.) would enable more thorough analysis of property-stability relationships.

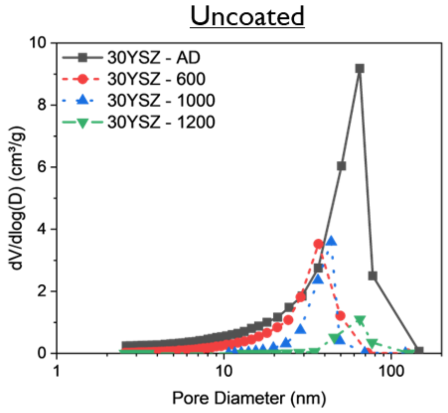
Application of SiO_2 coatings to best YSZ compositions show excellent stability to 1000 °C

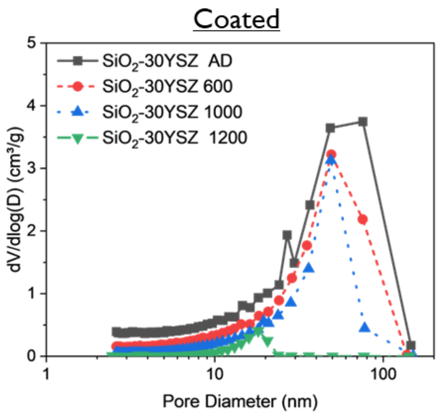


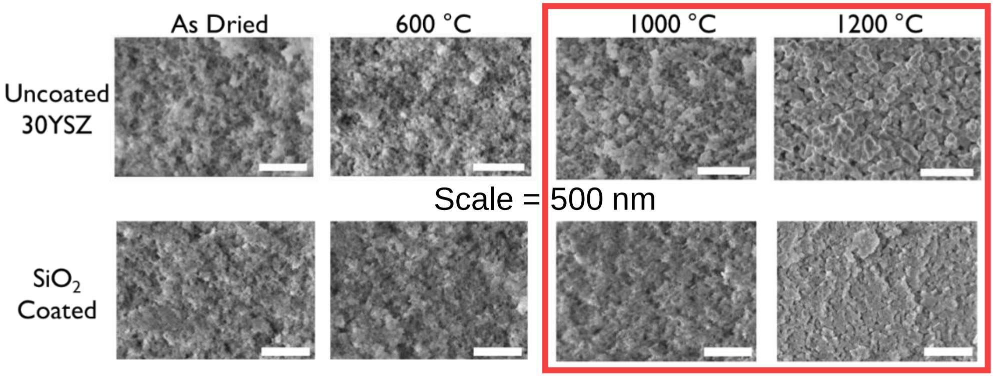
Liquid phase deposition from tetraethyl orthosilicate (TEOS)†

According to EDS, the molar ratio for 30YSZ is:

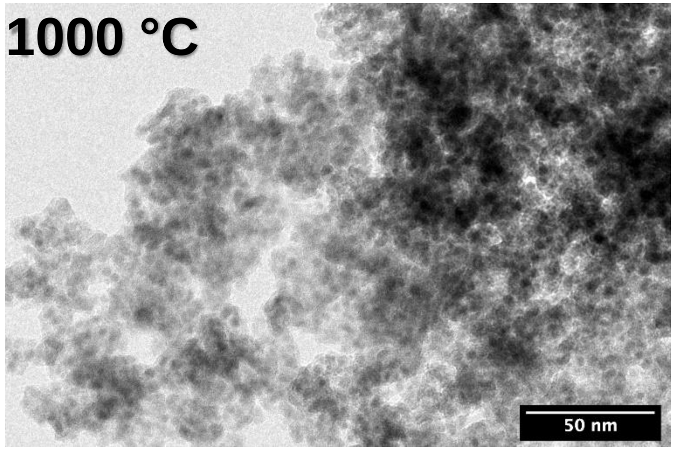
Zr	Y	Si
1.00	0.55	3.01

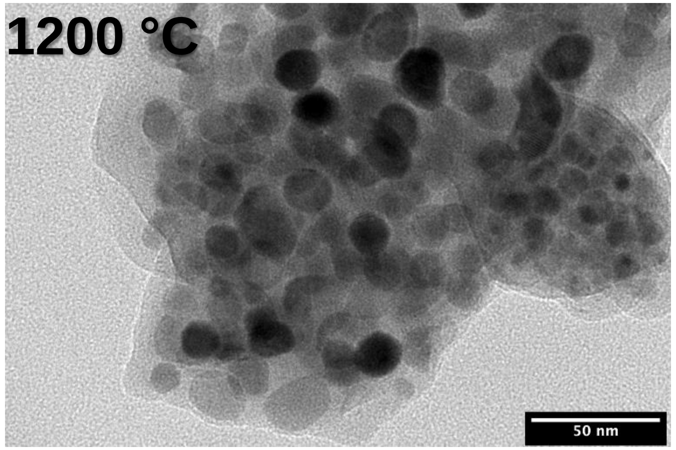






SiO_2-coated 30YSZ

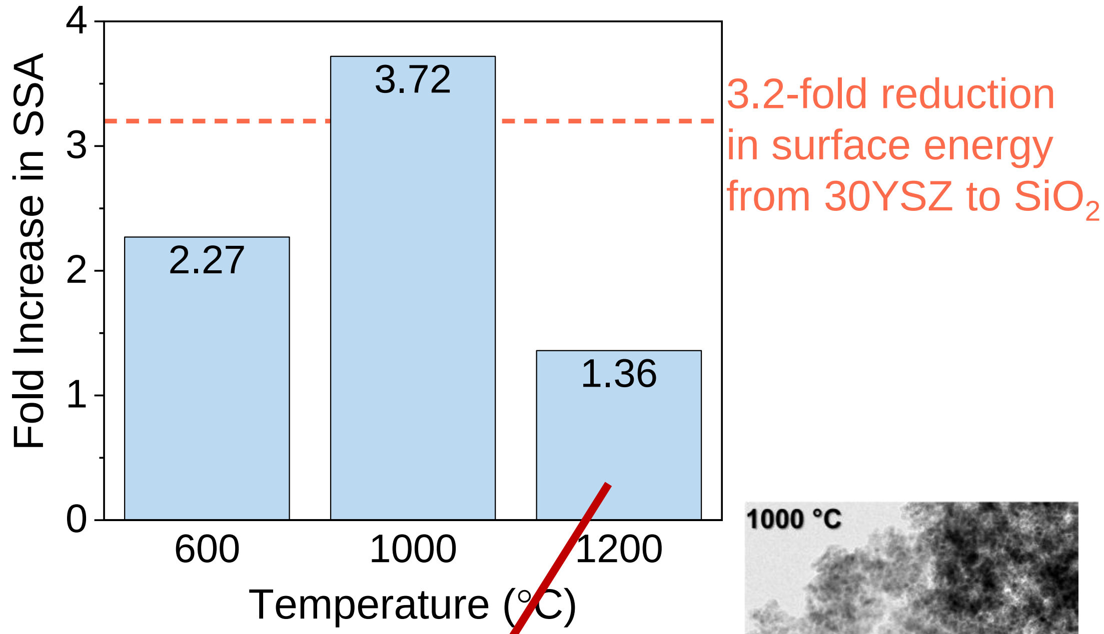


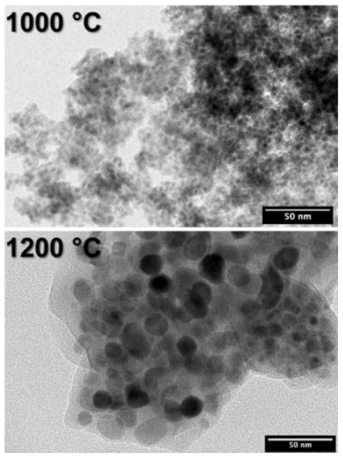


From 1000 to 1200 °C, SiO_2 coating enables viscous sintering and *enhances* densification.

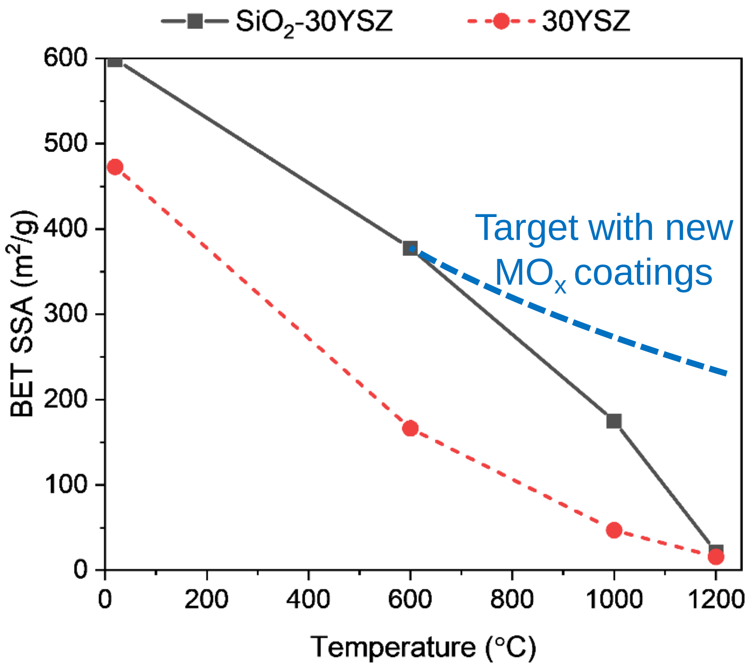
Over a factor of three reduction in surface energy from 30YSZ (0.83 J/m²) to amorphous SiO_2 (0.259 J/m²)

- Factor of 2.27 to 3.72 *increase* in SSA at 600, 1000 °C
- Searching for metal oxides with a range of surface energies could further test this hypothesis





Beyond 1000 °C, viscous sintering enhances densification, providing an upper use temperature for silica-coated aerogels

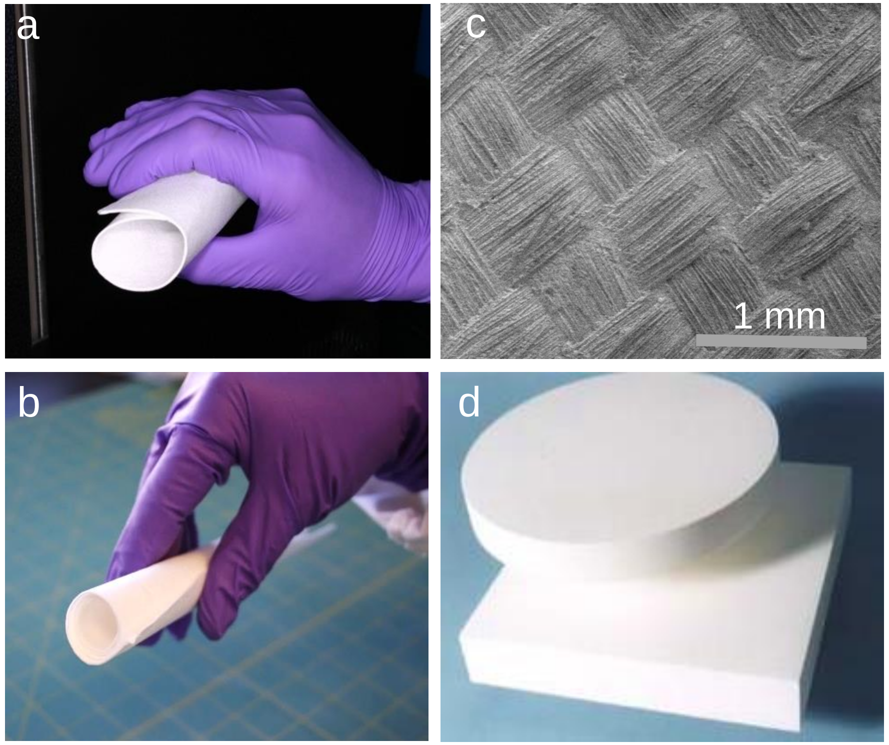


Can different metal alkoxide coatings still achieve improvement in thermal stability, but avoid this viscous flow behavior?

A key challenge is the reactivity of alkoxides beyond tetraethyl orthosilicate (TEOS)

Olson, N.S., and Stokes, J.L., et al. (2024). *Journal of the American Ceramic Society*, 107(9), 6353-6368.
Brunauer, S., et al. (1956). *Canadian Journal of Chemistry*, *34*(10), 1483-1496.
Drazin, J. W., & Castro, R. H. (2015). *Journal of the American Ceramic Society*, *98*(4), 1377-1384.

Select most favorable aerogel formulations and prepare composite materials for characterization



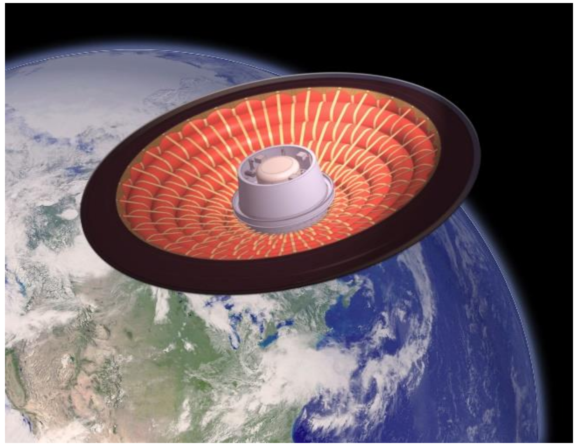
(a) Alumina papers

(b) Aluminosilicate papers

(c) Woven fabric

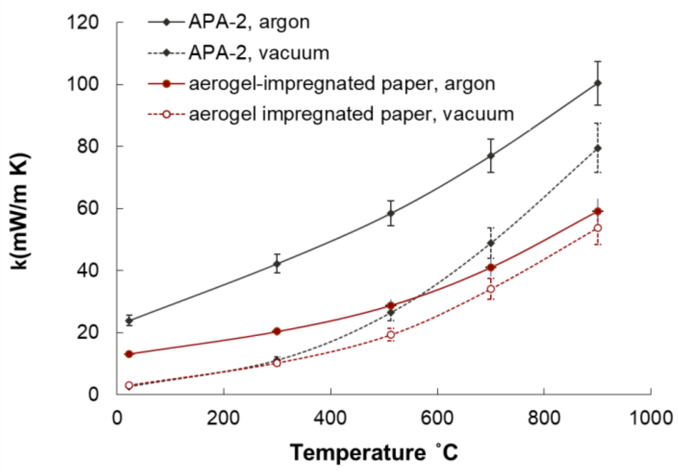
(d) Aerogel-filled foams

Spring seals utilizing aerogel-based composites

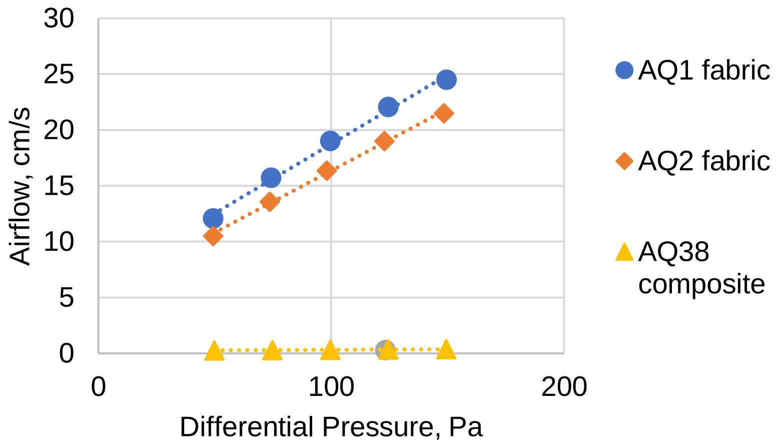


Flexible composite layers in inflatable decelerators

Thermal Conductivity of Composite



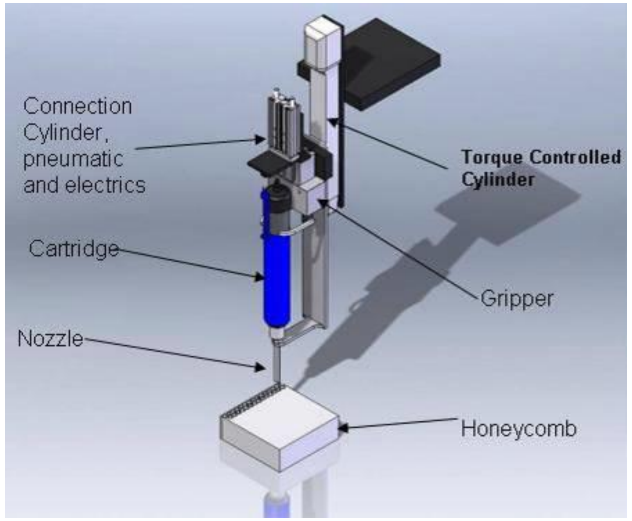
Permeability of Composite



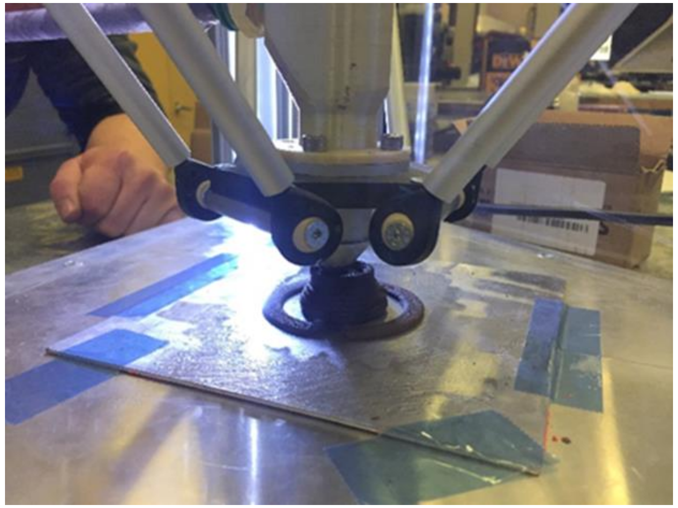
AMTPS at NASA

Additive Manufacturing of Thermal Protection Systems

- **2007 – 2009:** Explored automation for TPS
  - TPS Advanced Development Project developed automated gunning of honeycomb Avcoat on flat surfaces
- **2018:** AM manufacturing successes, especially in composite structures, led to exploratory efforts in AMTPS with internal funds at JSC, augmented with DOE funding through Oak Ridge National Lab
  - 3D Printed Heat Shields (FY18-FY20 CIF Project)
- **2019 – Present:** NASA continued development both internally & externally
  - **AMTPS Early Career Initiative (ECI)**
  - SBIR/STTR Program
    - 11 Phase 1 awards ($150K / 13 months)
    - 4 Phase 2 awards ($750K / 2 years)



Automation explored for honeycomb Avcoat under TPS ADP (2008)


Initial printing trials under JSC CIF project, 3D Printed Heat Shields (2017-2019)

Why additive manufacturing?

Build time reduced from months/weeks to weeks/days

Traditional Approaches

Manual fabrication, bonding in segments, single formulation

Orion

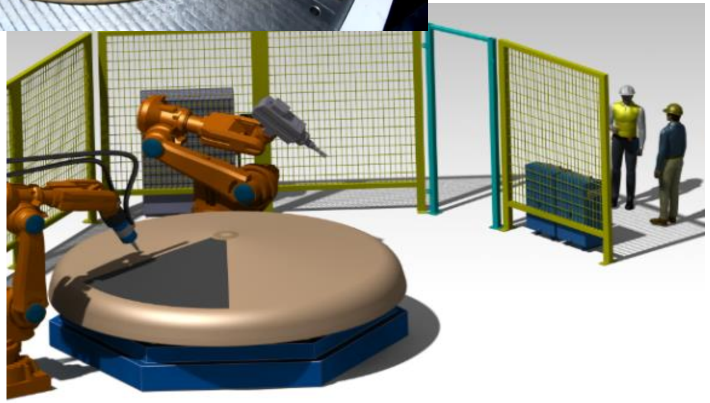
Apollo

Mars Science Laboratory

Photo Credits
Left: B. Anthony Stewart/National Geographic/Getty Images, The Amazing Handmade Tech That Powered Apollo 11's Moon Voyage – HISTORY
Top right: NASA/Isaac Watson, Heat Shield Milestone Complete for First Orion Mission with Crew | NASA
Bot right: NASA/JPL-Caltech/Lockheed Martin, Large Heat Shield for Mars Science Laboratory – NASA's Mars Exploration Program

AMTPS

Automated, monolithic fabrication, graded formulation



Goal: To develop an automated, additive approach for heat shield manufacturing

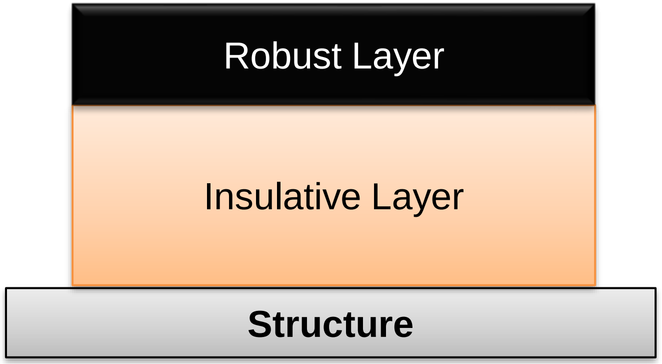
Why? Reduce cost and improve consistency over traditional manufacturing by automating and accelerating production; direct integration onto structure during processing simplifies integration

Internal R&D (pre-cursor project)

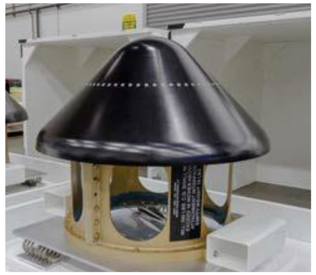
Focus on ablative TPS

Project Deliverables

(1) Develop and characterize a printable, graded TPS architecture



(2) Build and test a mid-scale MDU (up to 1.0m dia.)



(3) Design and build AM capsule for flight testing

FY18-20 | FY21 | FY22 | FY23

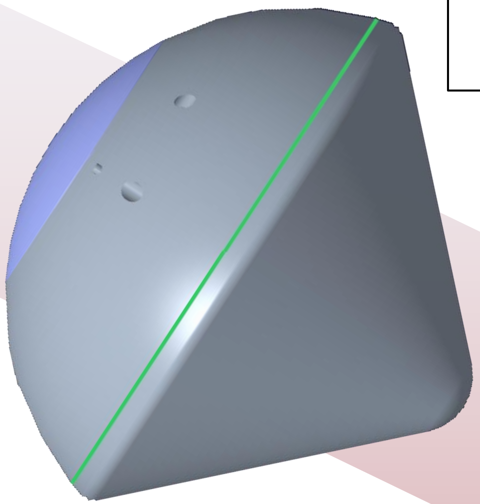
Two printable TPS material systems

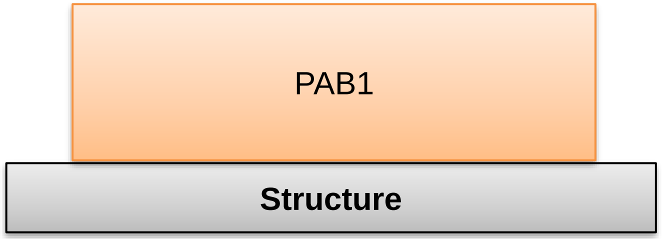
Forebody (PAF1)

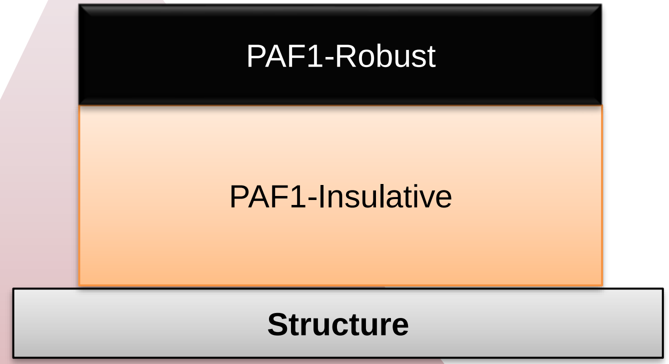
- **P**rintable, mid-density **A**blator for **F**orebody
- Phenolic-based resin / dual layer
  - *Robust*: higher density; higher temp capability ablative layer
  - *Insulative*: lower density, more insulating internal layer

Backshell (PAB1)

- **P**rintable, low-density **A**blator for **B**ackshell
- Several resin options explored during course of project







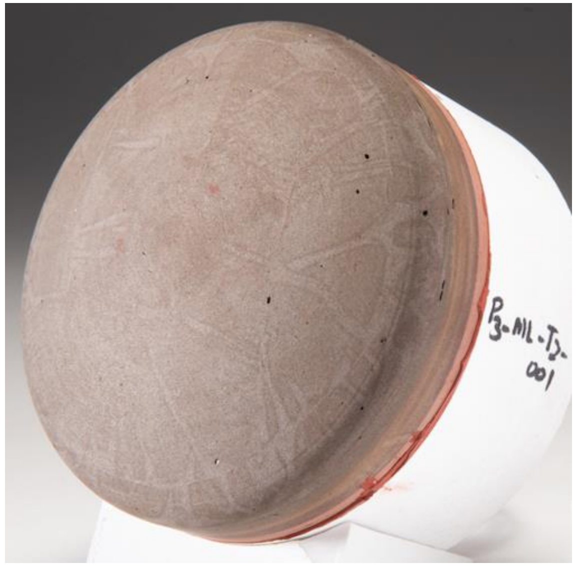
Dual layer TPS layer configuration

Good performance in arc jet testing

- Two rounds of arc jet testing at NASA Ames AHF facility in 2021 and 2022
- 4” diameter iso-q models
- 30 second exposures

Pre-test

4” diameter AMTPS iso-q
Multi-layer (insulative → robust)

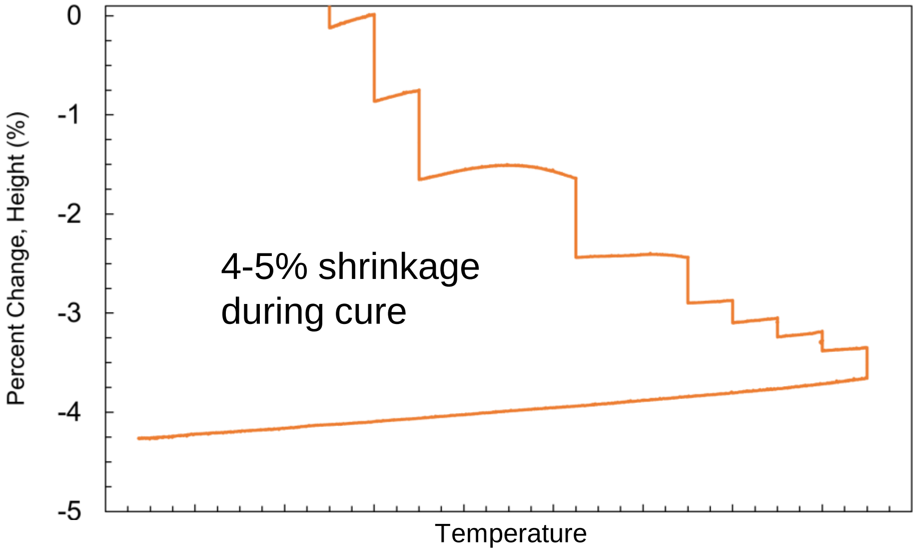


During test

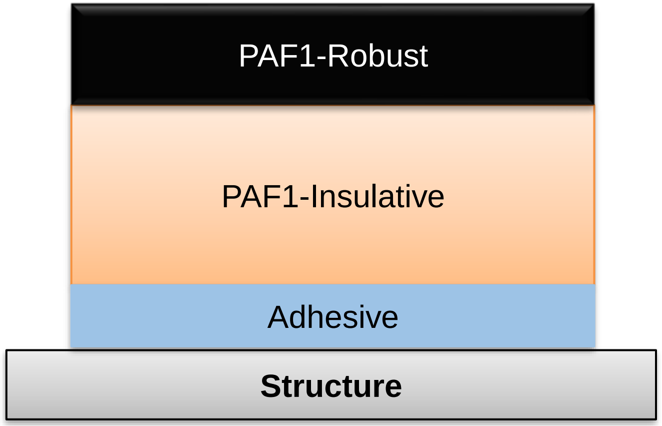
Thermoset resin shrinkage leads to disbond during cure

Goal: Print TPS directly onto capsule.
Cure and bond in a single step.

3D printed ablative material does not stick to aluminum or titanium

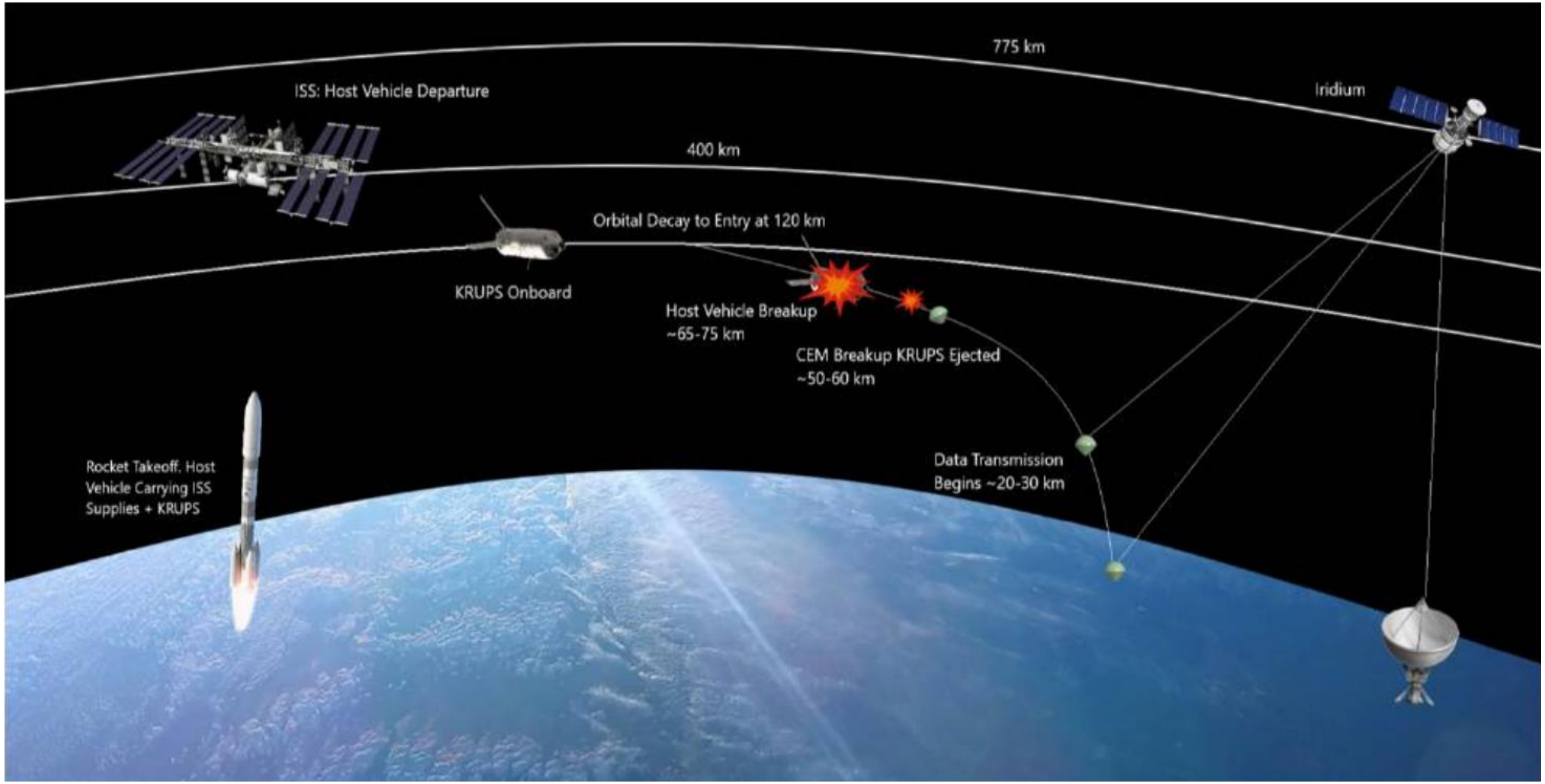


To cure ablative TPS material directly onto a structure, an adhesive or mechanical solution must be implemented.



Flight test collaboration with University of Kentucky

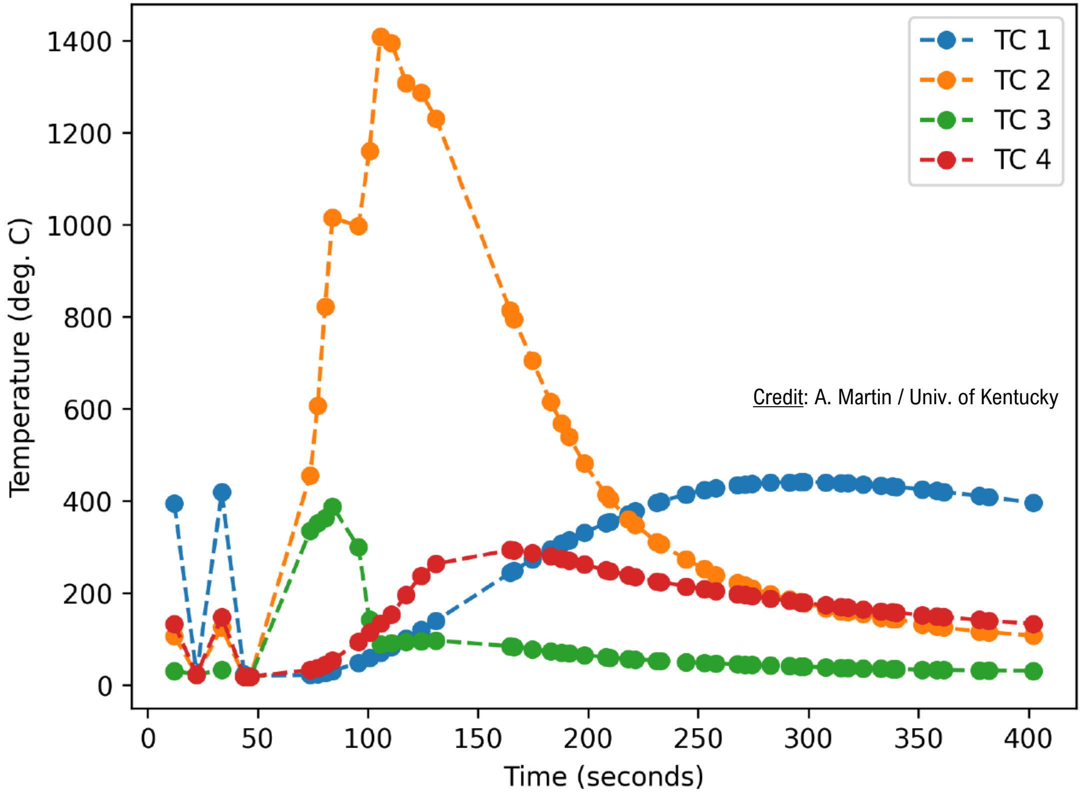
- KREPE capsules launched to ISS onboard Cygnus re-supply vehicle
- Capsules depart ISS onboard Cygnus (ISS trash on-board)
- Re-entry and breakup of Cygnus; capsule fly free to ground and telemeter data



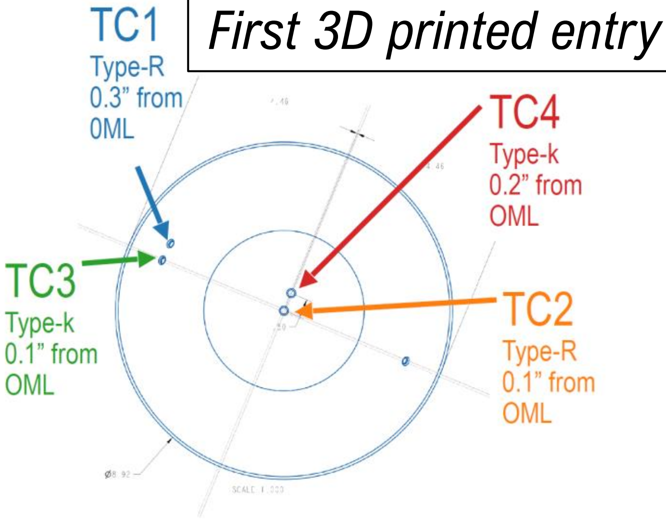
KREPE1 Mission: 1st AMTPS Heat Shield

University of Kentucky

- 1 of 3 KREPE capsules protected by AMTPS heat shield
  - 11” diameter, 45-degree sphere-cone
  - Built in ~2 weeks, single piece, multi-layer construction
- Cyanate ester-based printable ablator
- **Successfully returned in Dec 2021**

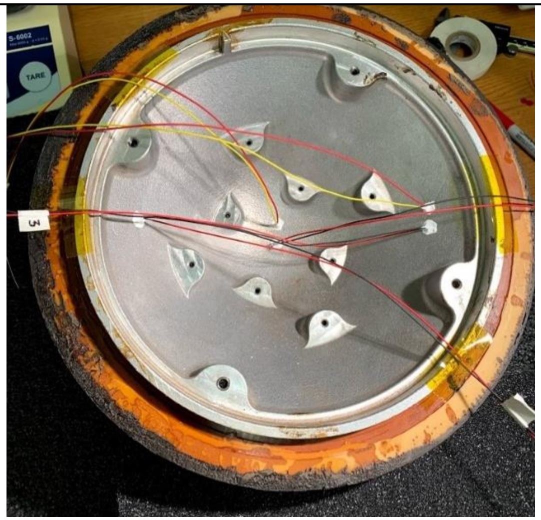


*First 3D printed entry flight heat shield in history**



Credit: A. Martin / Univ. of Kentucky

T/C instrumentation

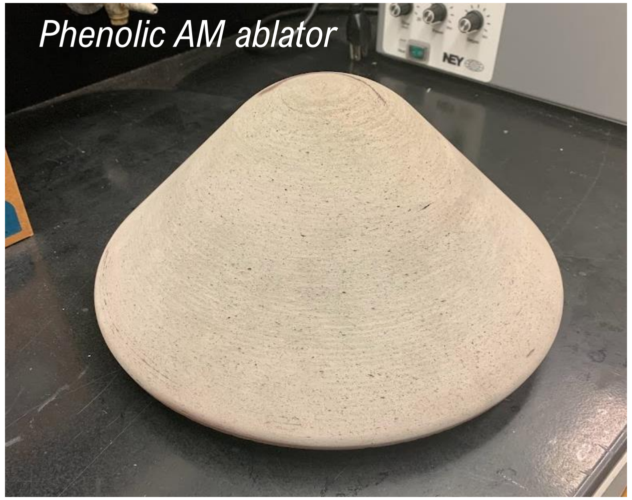


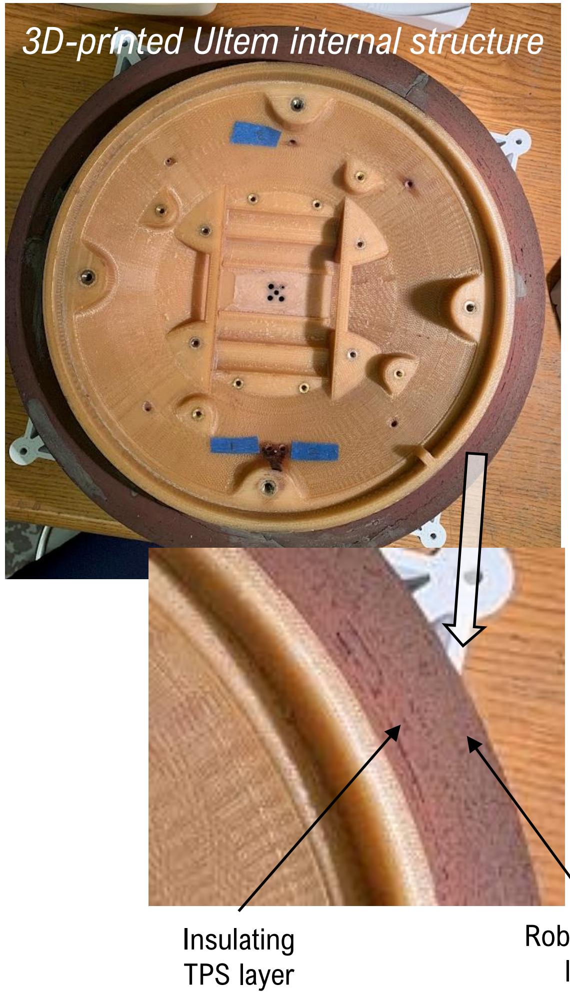
** To the authors’ knowledge*

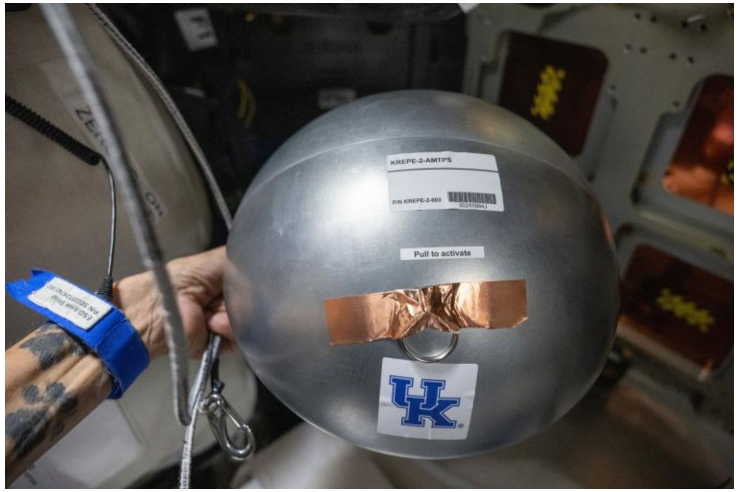
KREPE2 Mission: Same mission, new material

- 1 of 5 KREPE capsules protected by AMTPS heat shield
  - Same geometry: 11” diameter, 45-degree sphere-cone
  - Printed in ***2 days***
- <u>Phenolic-based</u> printable ablator
  - Dual layer system (robust + insulative)
  - Adhesive layer for bond
- Instrumentation
  - 6 thermocouples
  - 5 forebody pressure sensors
  - 1 spectrometer
  - GPS / IMU for reconstruction
- **Launched on NG-20 in Jan 2024, successfully returned July 2024! Awaiting data…**

Second 3D printed flight heat shield successfully flown in 2024



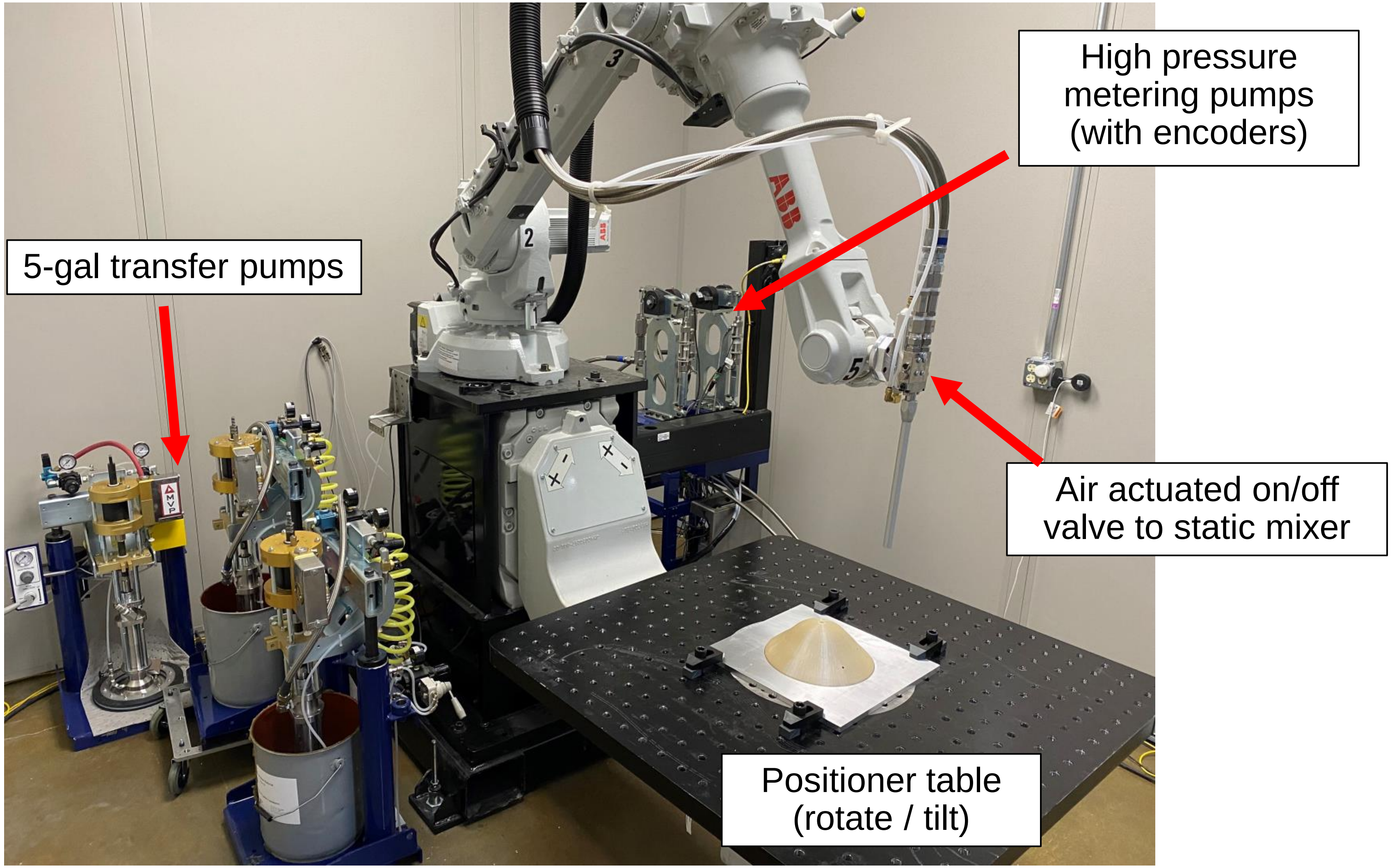




Manufacturing an AMTPS heat shield

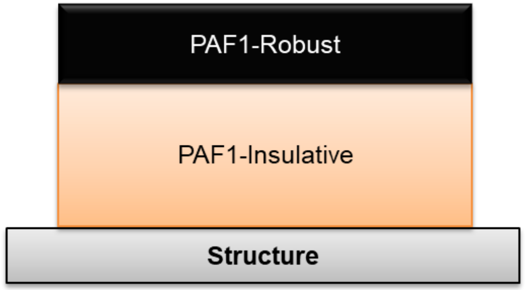
- *Positioner table* affords toolpath flexibility (**6+2 axis printer**)
  - Concentric/spiral
  - Rectilinear/crosshatch (e.g., 0/90 or 0/45/90/45/0)
  - Combo of concentric/rectilinear
- Software tools translate to manufacturing cell
  - Hypermill
    - Machining focus with AM capabilities) will output position and orientation vector, post process for robot motion planning
  - ROS rviz and Gazebo
    - Robot motion planning and simulation

1 or 2K system; expandable to multi material systems

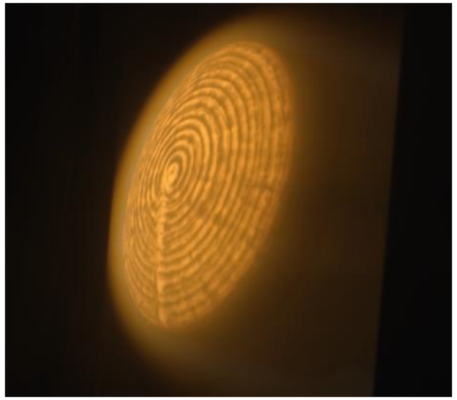


Development of Additive Manufacturing of Thermal Protection Systems (AMTPS)

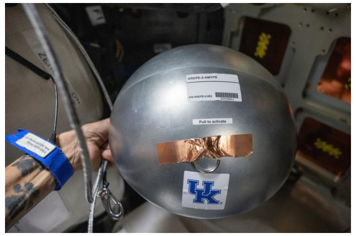
<u>Goal</u>: Reduce cost and time required to manufacture ablative TPS.



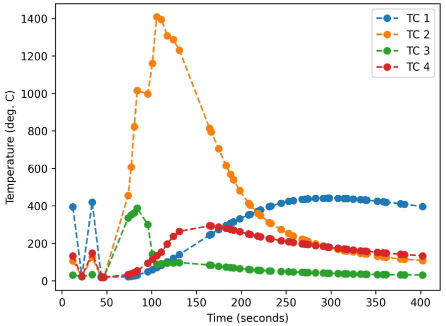
Dual layer TPS **material system developed**



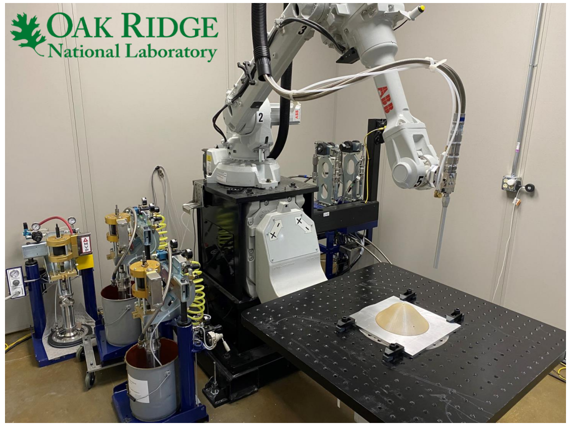
High enthalpy ground testing completed in arc jet facility at NASA Ames Research Center



University of Kentucky



First **flight test** of an additively manufactured ablative heat shield.



Scale-up of manufacturing with Oak Ridge National Laboratory.

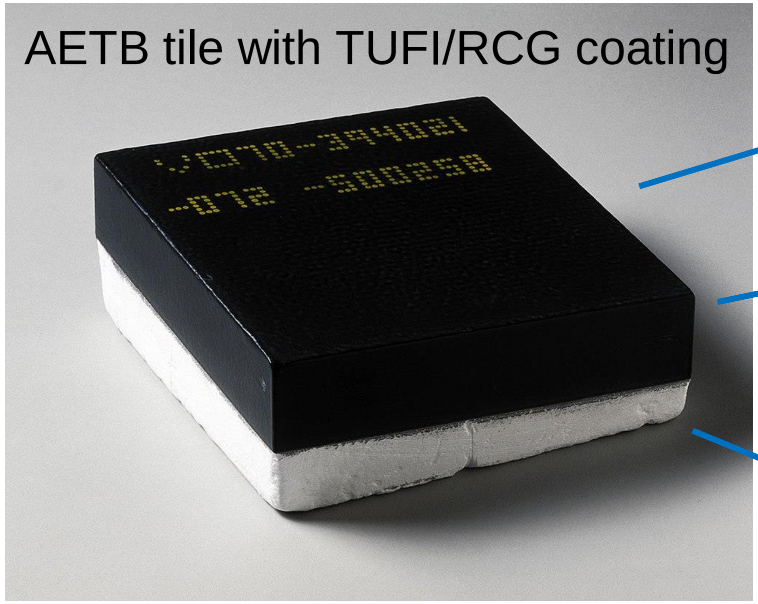
<u>Future Challenges:</u>

1. Overcoming cure shrinkage issues that contribute to dis-bonding and cracking

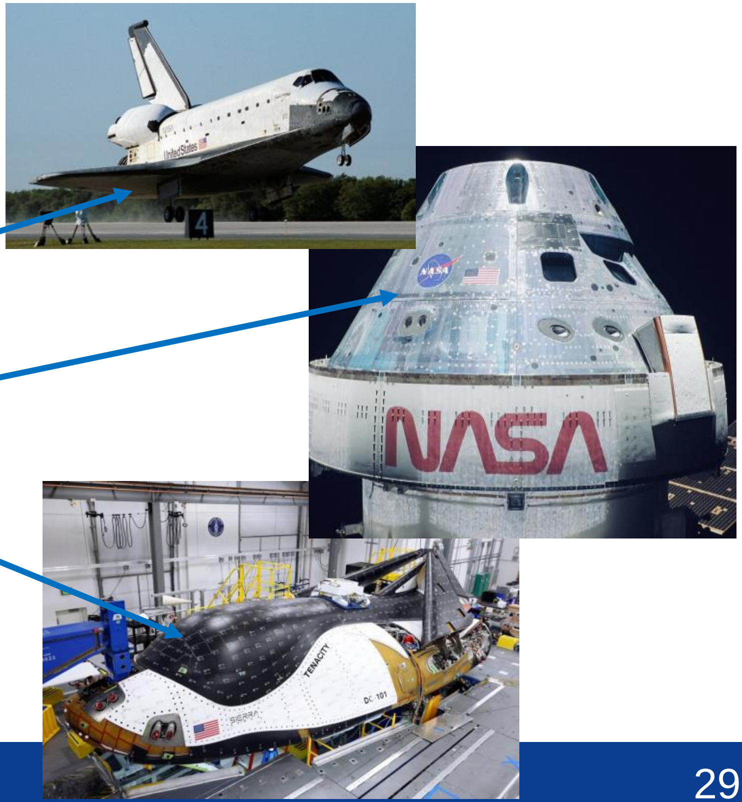
2. Robotic control for large-scale manufacturing.

Characterizing silica fiber for AETB production

- **AETB** = Alumina Enhanced Thermal Barrier
  - Reusable aluminoborosilicate tile
- Silica fiber is primary component of AETB and is produced by Johns Manville
  - Heritage raw material stockpile being used for production of AETB for Orion
  - Process change from Shuttle-era to today



AETB tile with TUFI/RCG coating



Characterizing modern and heritage silica fiber

Total of 16 fiber lots: 12 modern, 4 heritage

Two aims of work:

(1) Evaluate changes in physical properties of Q-fiber from heritage to modern version
(2) Identify fiber properties that drive differences in fiber performance

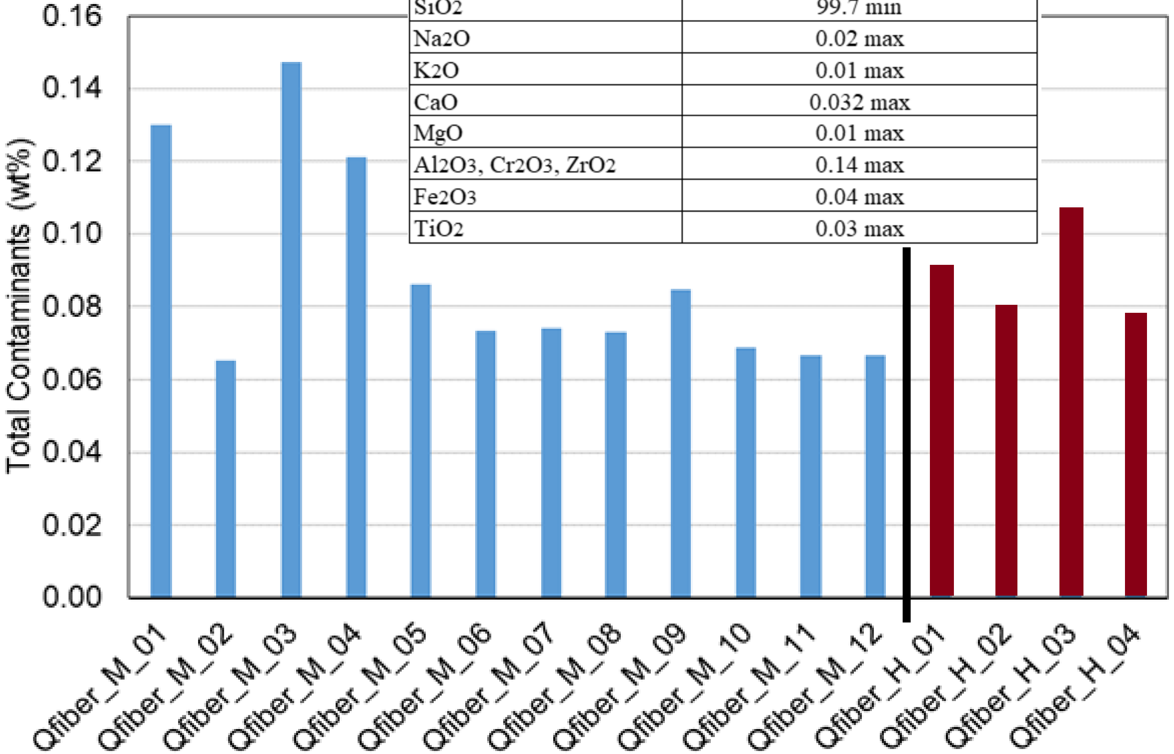
- Measurements of **fiber properties** include:
  - Morphology: SEM
  - Diameter: SEM + ImageJ, SEM + GIFT, DiamScope, Williams Freeness
  - Composition: ICP-MS (trace), ICP-OES (silica), silica volatilization
  - Specific surface area: Nitrogen Physisorption (BET method)
- Measurements of **fiber performance** include
  - Crystallinity: XRD (2500 °F, 4 h)
  - Shrinkage:
  - Fired Density:
  - Dimples:

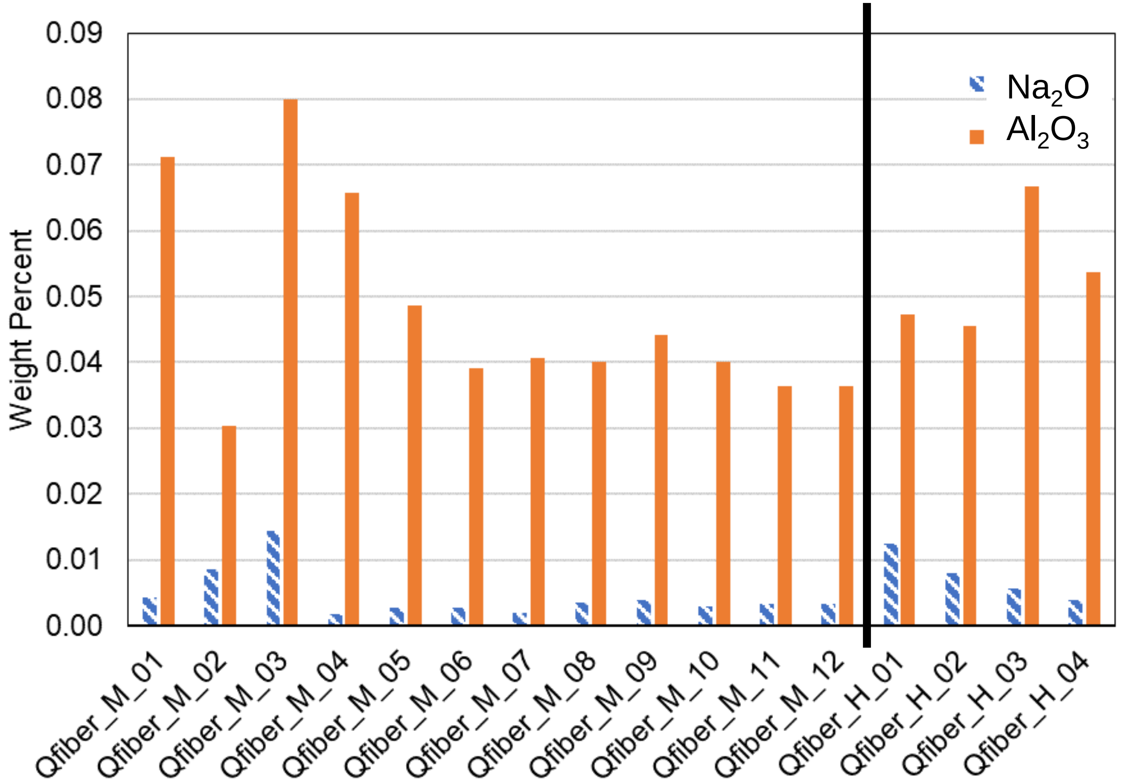
(Shrinkage, Fired Density, Dimples:) Cast Thermal Stability Samples (2500 °F, 15 min)

Compositional purity strictly controlled for Shuttle program to prevent devitrification

Table I. Chemical Composition of Silica Fibers

Component	Requirement (Weight Percent)
SiO_2	99.7 min
Na_2O	0.02 max
K_2O	0.01 max
CaO	0.032 max
MgO	0.01 max
Al_2O_3, Cr_2O_3, ZrO_2	0.14 max
Fe_2O_3	0.04 max
TiO_2	0.03 max



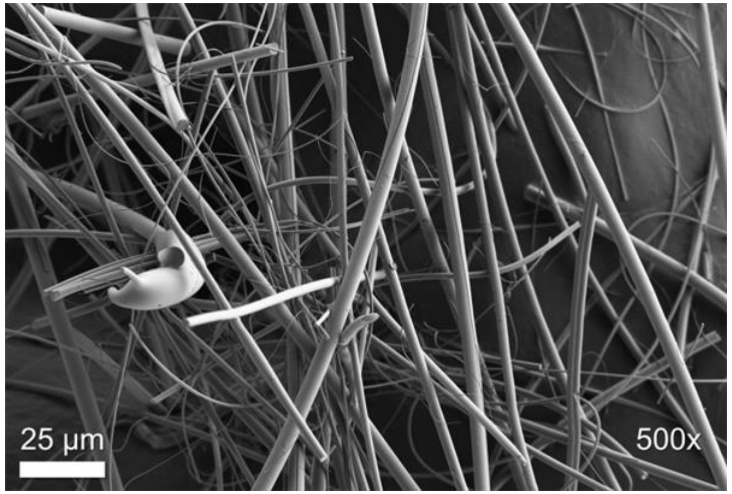


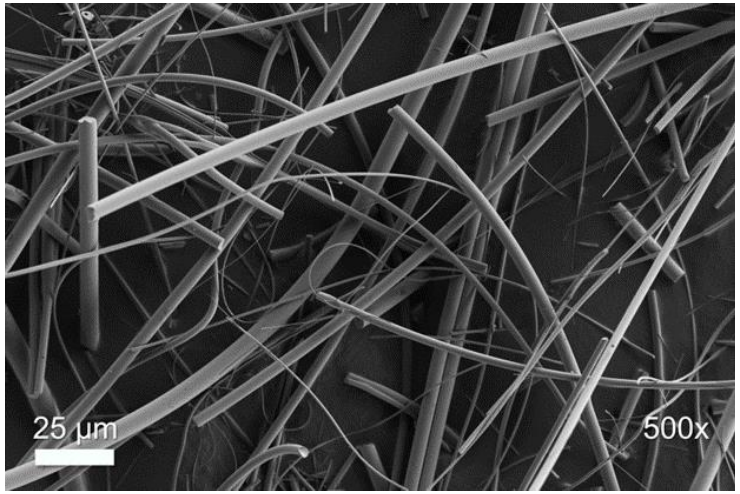
Average Total Trace Metal Oxides
Heritage: 0.0891 ± 0.014 wt.%
Modern: 0.0881 ± 0.028 wt.%

No significant difference in trace oxide content between Heritage and Modern fibers from NASA except for Cr_2O_3

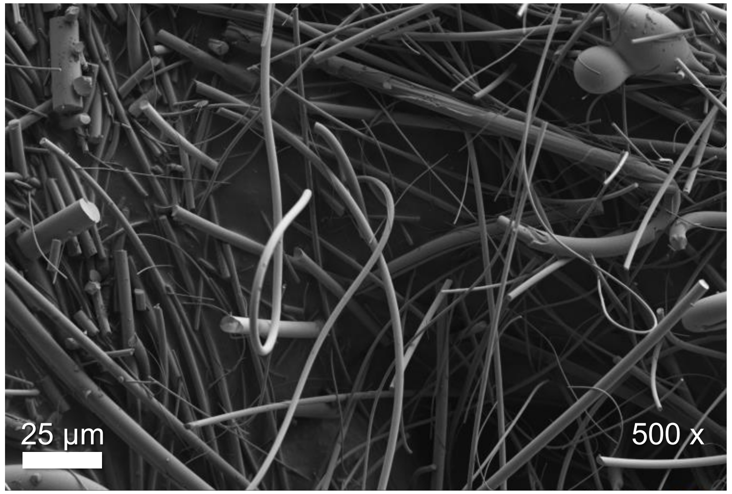
Modern had significantly more Cr_2O_3 than Heritage fibers (but within spec)

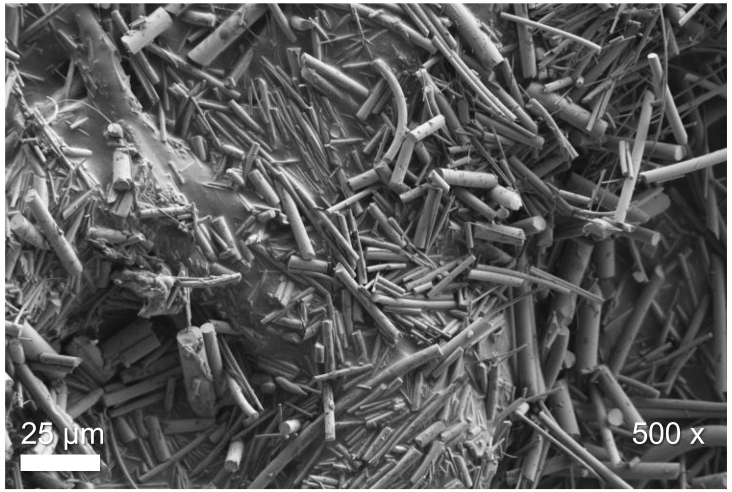
Modern

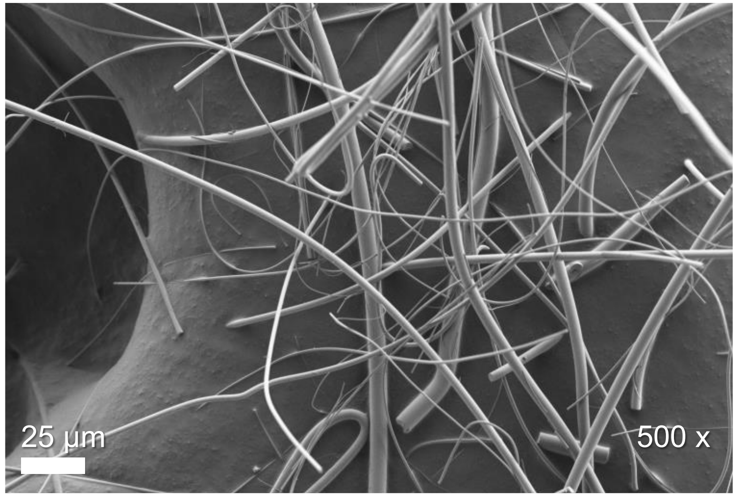




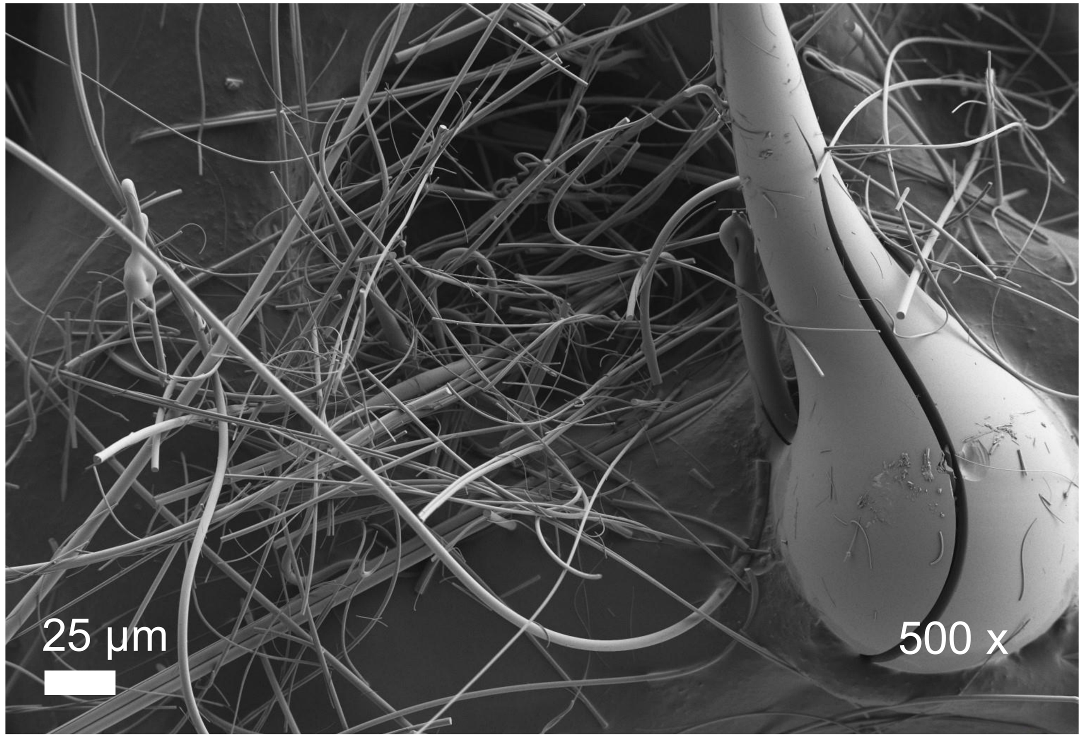
Heritage

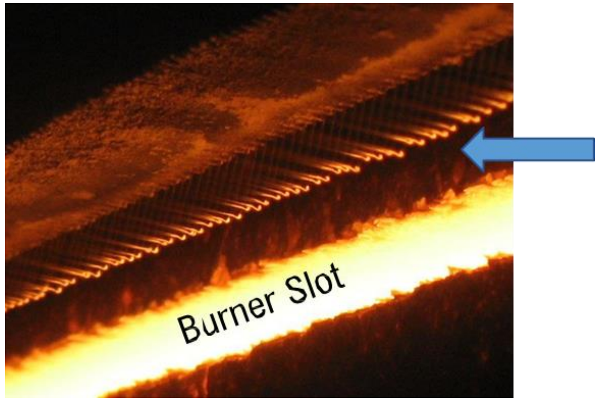






“Shot” are large pieces of un-fiberized glass





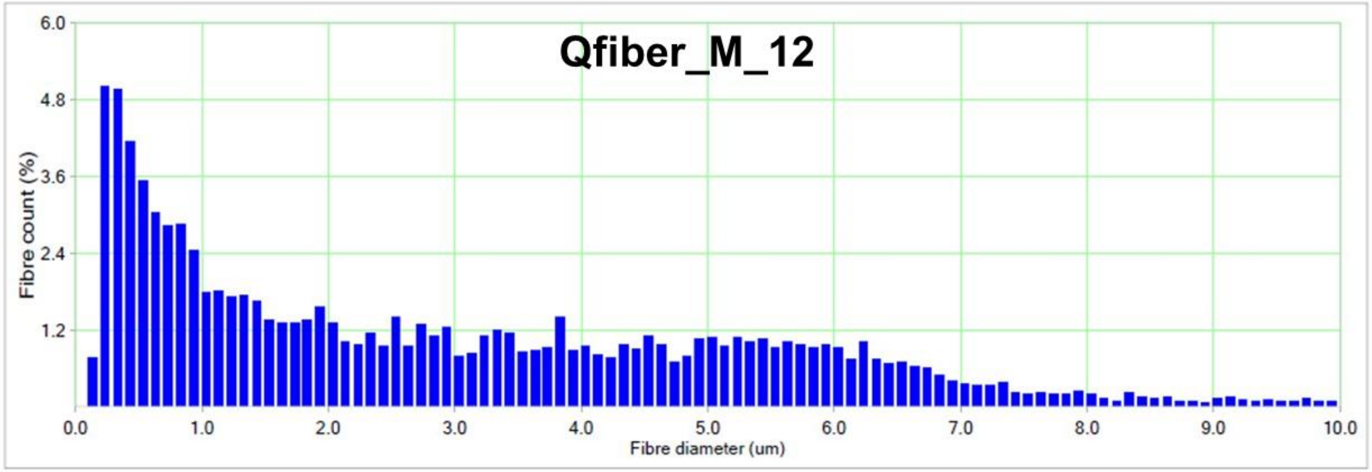
Sodium-rich glass is melted and fiberized in flame attenuator. Some glass makes it through without becoming fiber (shot)

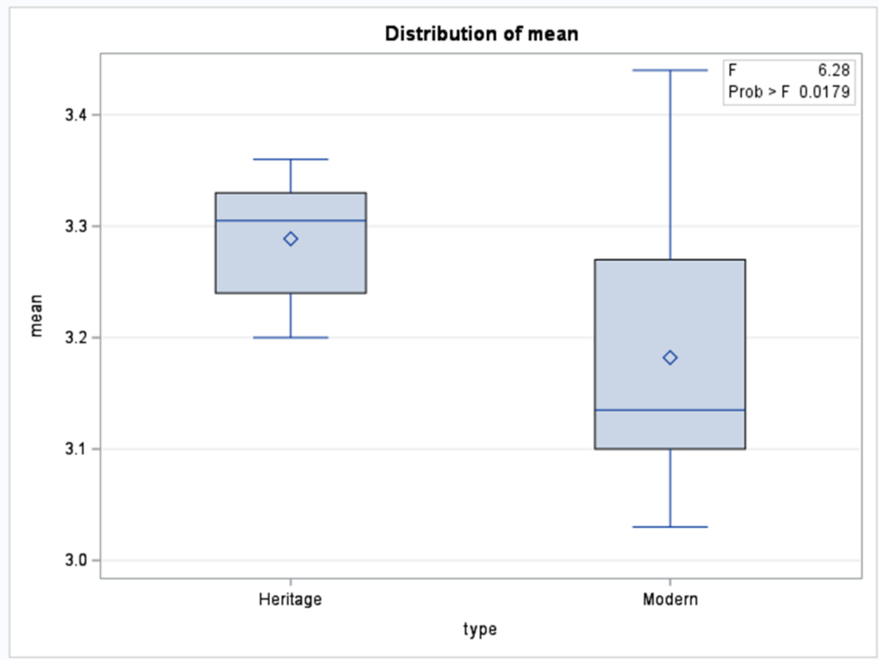
Shot remains **sodium-rich** during downstream acid leaching process.

Measurement of fiber diameter

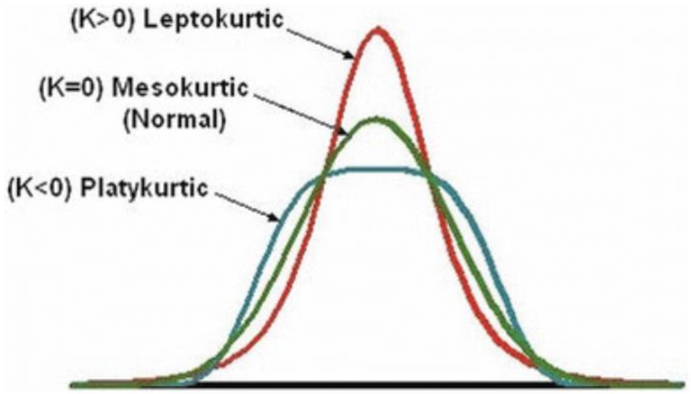
Fibremetrics DiamScope

1. Fibers dispersed in water bath
2. Optical images taken
3. Automatic image analysis





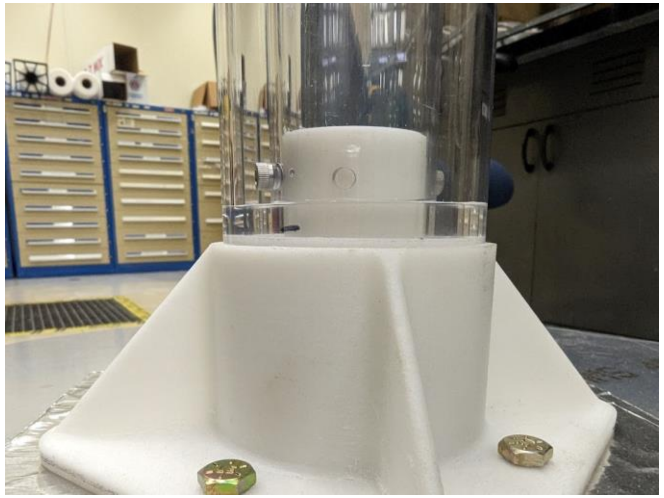
Mean diameter of Heritage fibers significantly larger than Modern fibers



Significantly more platykurtic fiber diameter distribution for Modern than Heritage fibers (thinner tails on distributions)

These differences may **influence manufacturing processes** and require modifications to current procedures.

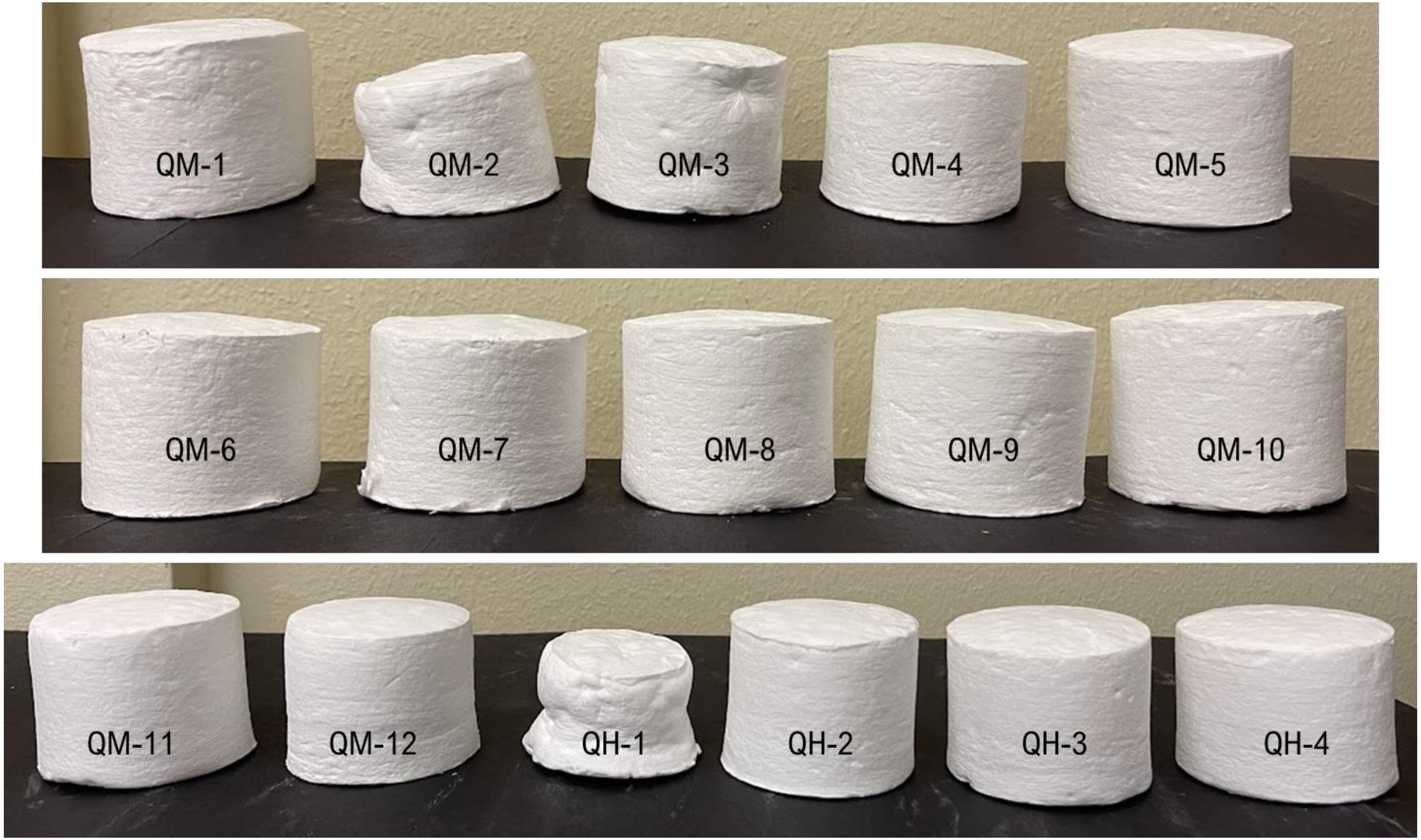
Preparation of thermal stability articles



Diameter Measurement

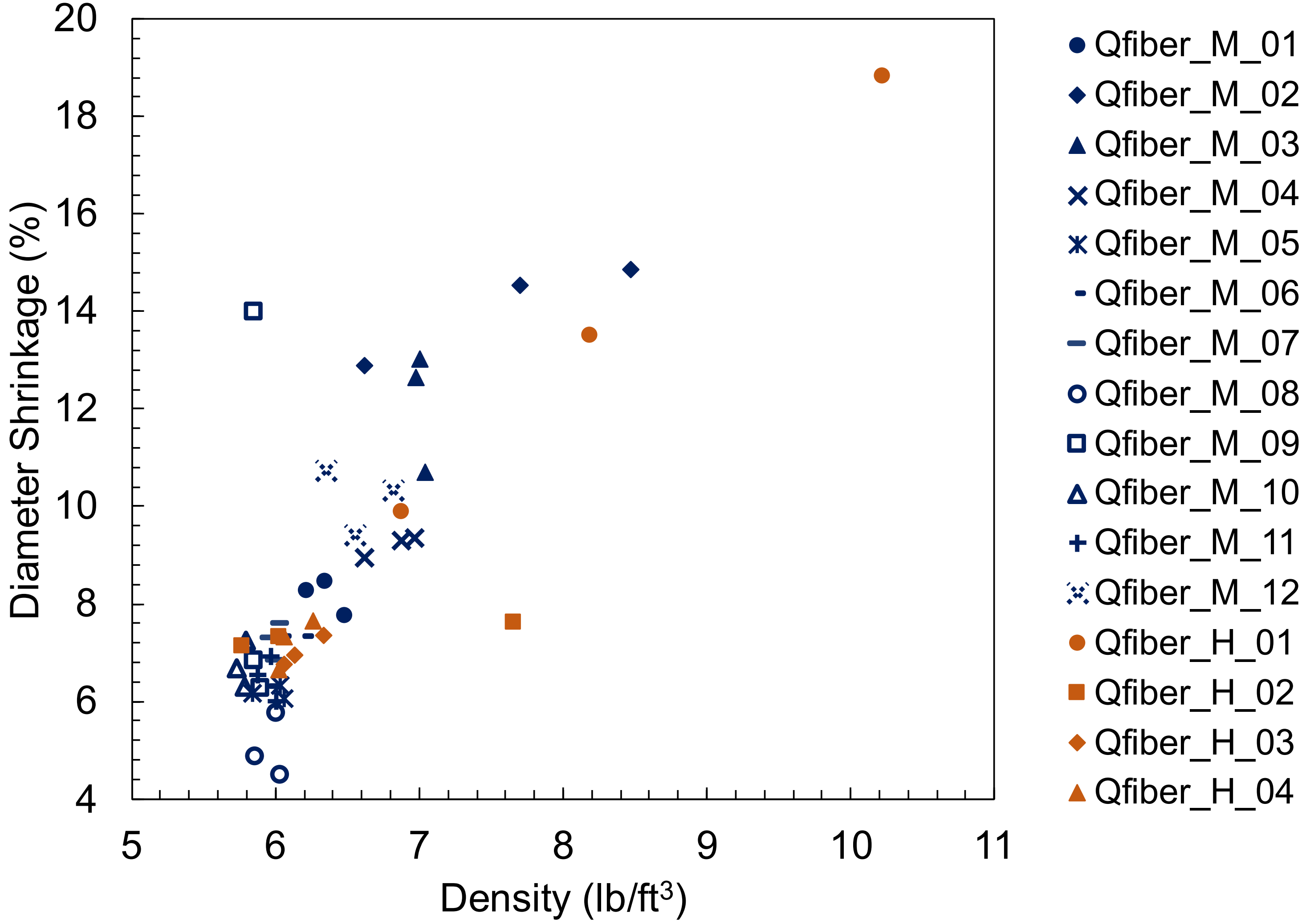
Volume (Density) Measurement

Sintered thermal stability articles



N=4 for each (3 sintered, 1 left dried)

Differences in performance between heritage and modern silica fiber



Average Diameter Shrinkage

Modern: 8.5 ± 2.6 %

Heritage: 8.3 ± 2.2 %

Average Density

Modern: 6.33 ± 0.56 lb/ft^3

Heritage: 6.57 ± 0.66 lb/ft^3

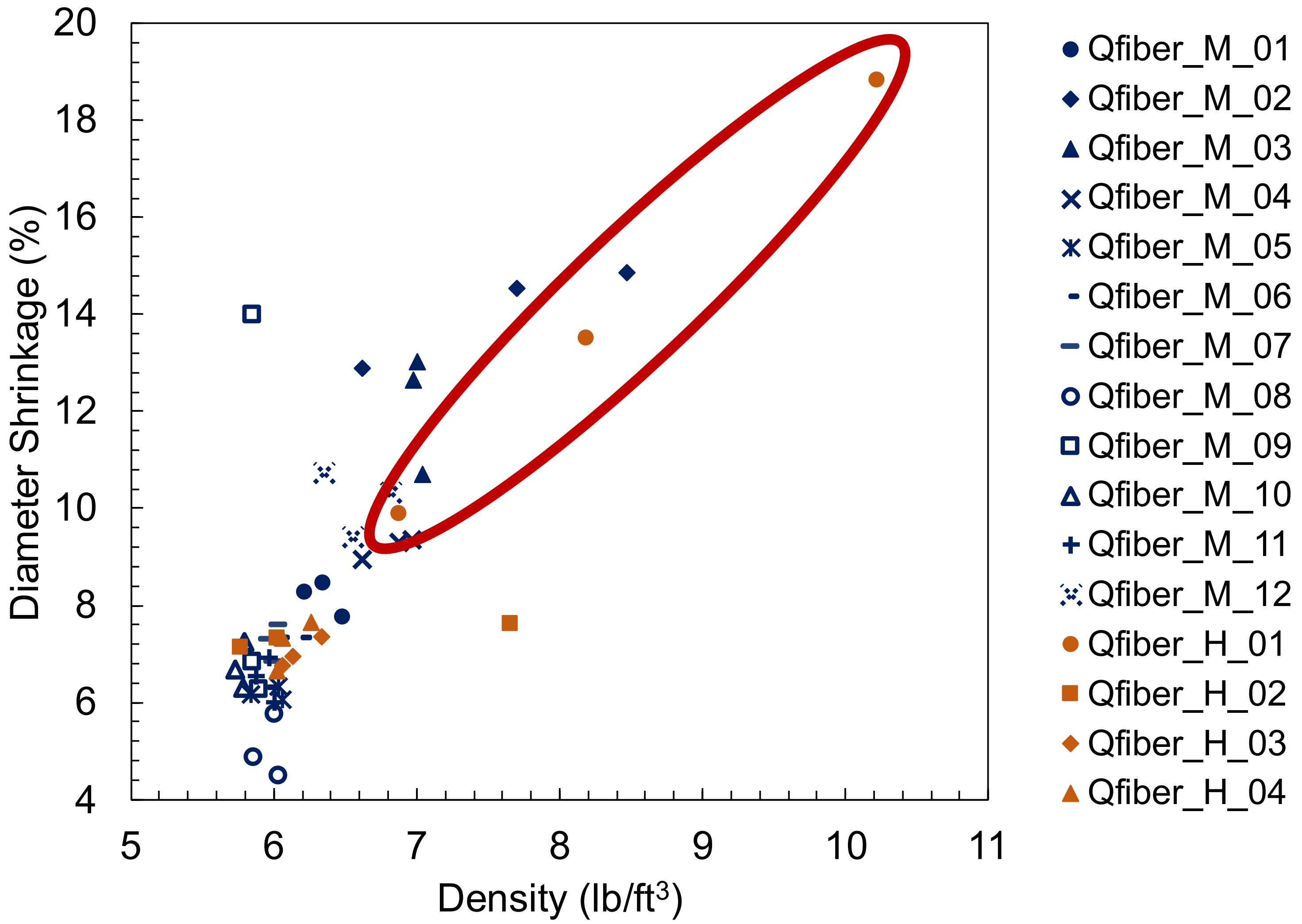
Average Dimples

Modern: 0.8 ± 1.3

Heritage: 0.8 ± 1.7

Statistical analysis in progress comparing modern and heritage performance.

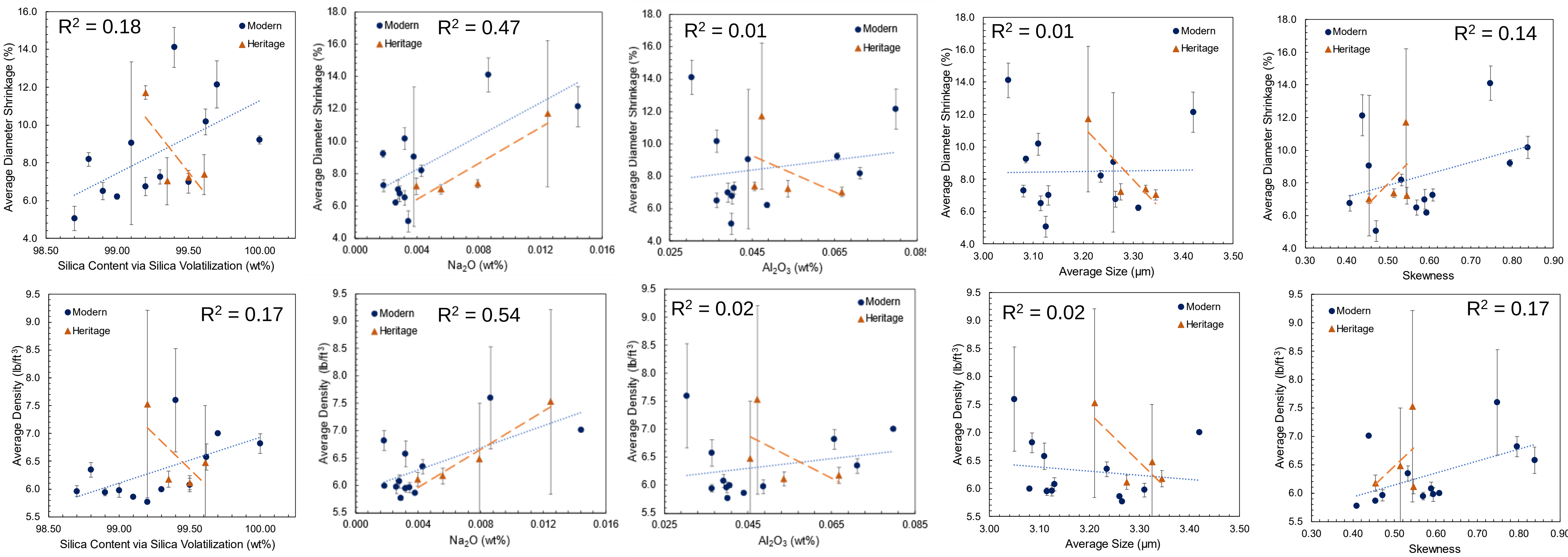
Differences in performance between heritage and modern silica fiber



Qfiber_H_01

Relationship between fiber properties and performance

	Silica Content	Na_2O	Al_2O_3	Average Fiber Size	Fiber Size Skewness
Shrinkage					
Sintered Density					

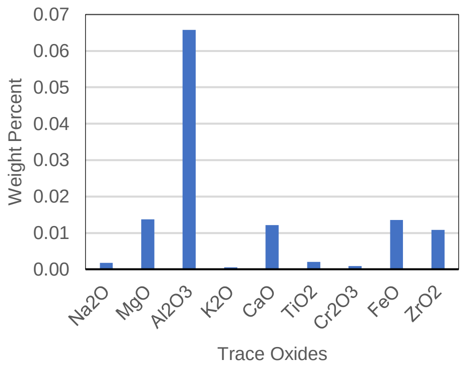


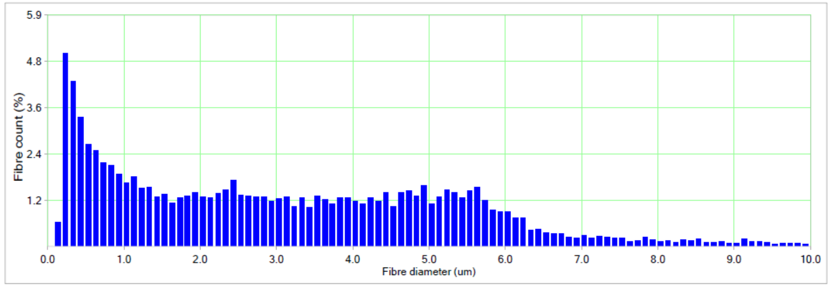
No clear relationships yet, but more data and statistical analysis is being pursued.

Characterization of raw materials for reusable TPS manufacturing

Change to primary constituent of AETB tile, which was used on Space Shuttle and now on Orion and Dream Chaser.

Fiber Properties



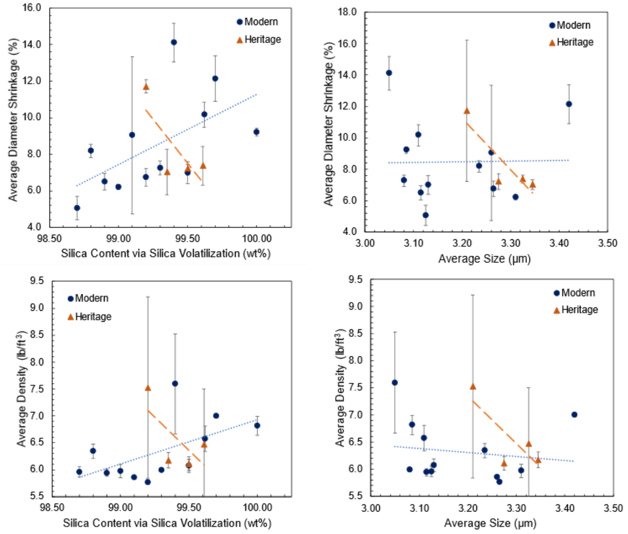


Silica Content

Average Fiber Size

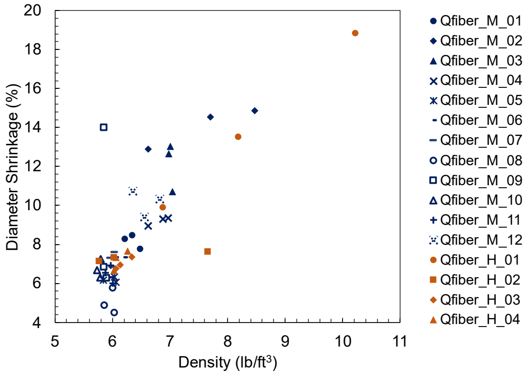
Shrinkage

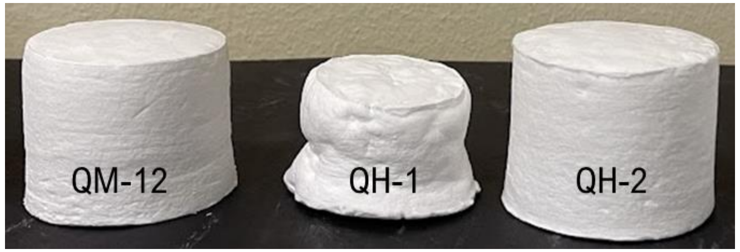
Sintered Density



Evaluating links between fiber properties and performance. What material properties are relevant for TPS performance?

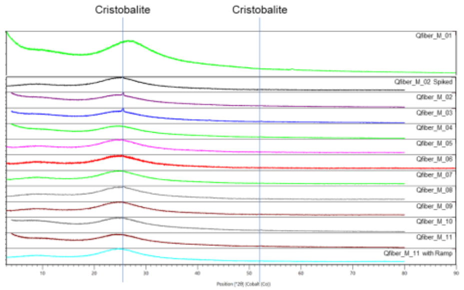
Fiber Performance



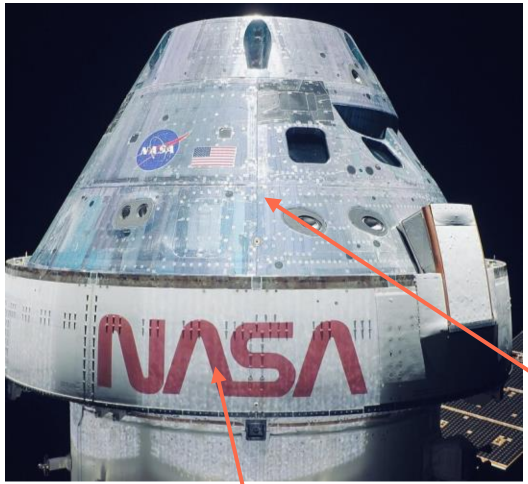


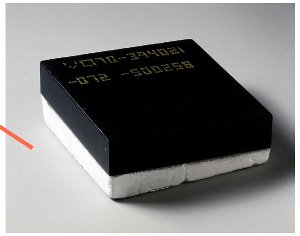
Future Work:

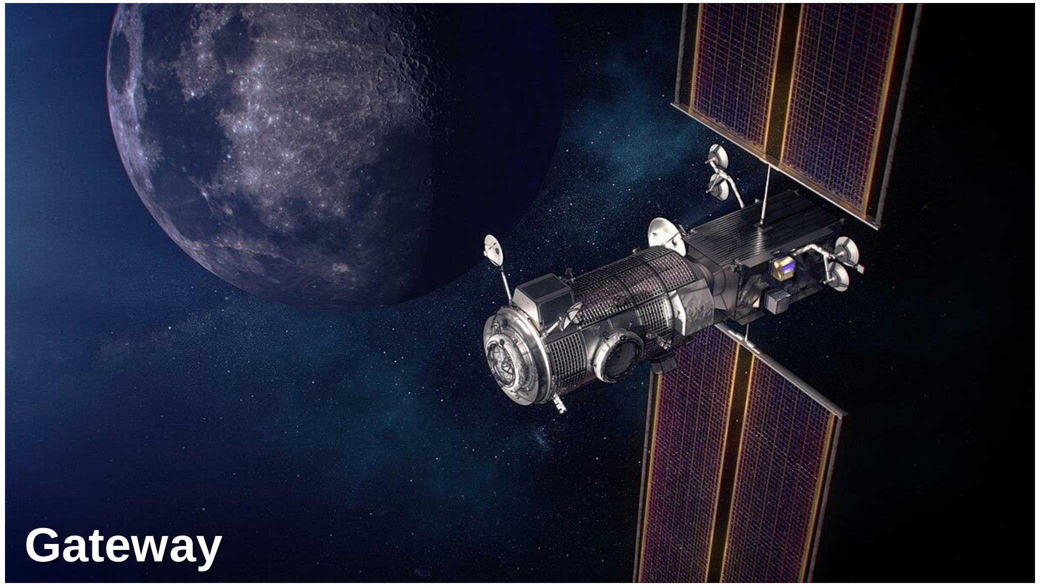
1. Finalize material characterization, including crystallinity via XRD.
2. Work with TPSF at NASA KSC to understand impact of modern fiber on manufacturing.
3. Begin qualification process for AETB tiles made with modern silica fiber.

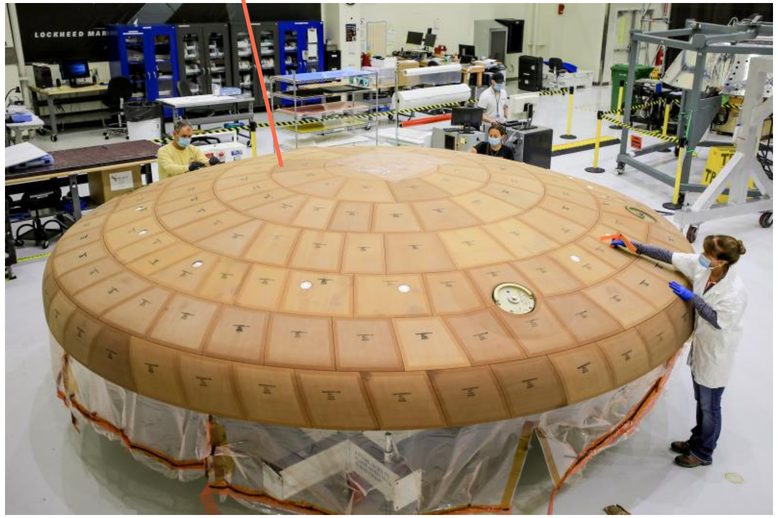


Artemis Program and the Orion spacecraft



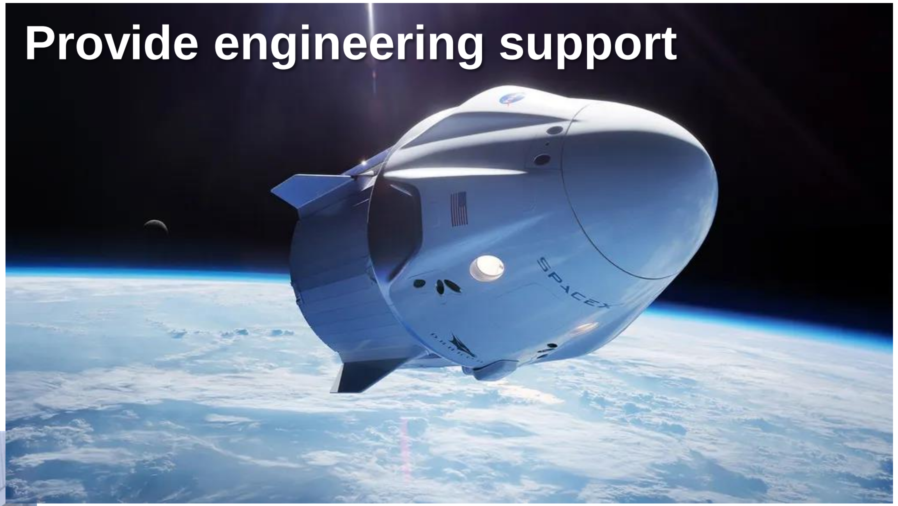


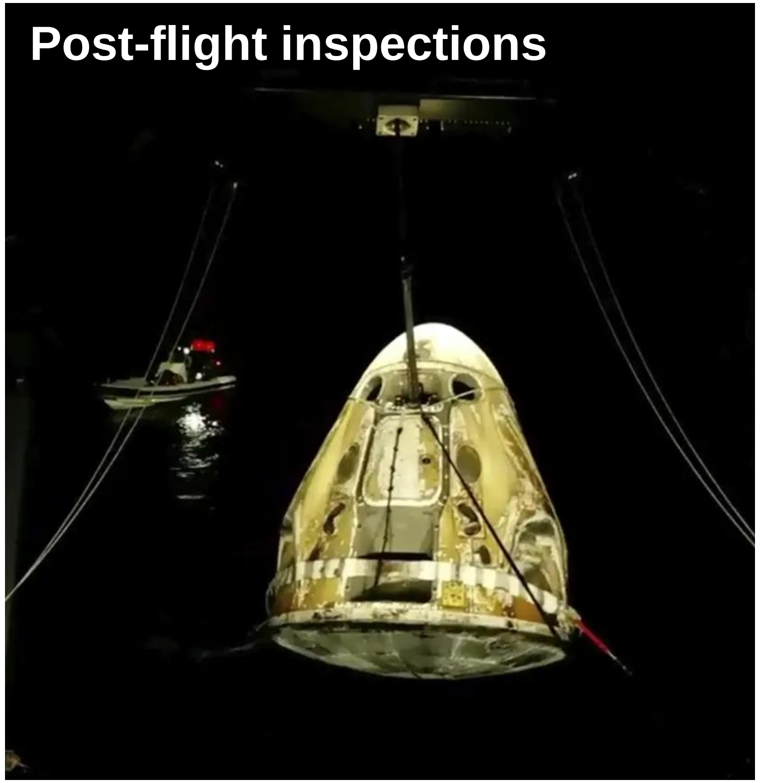


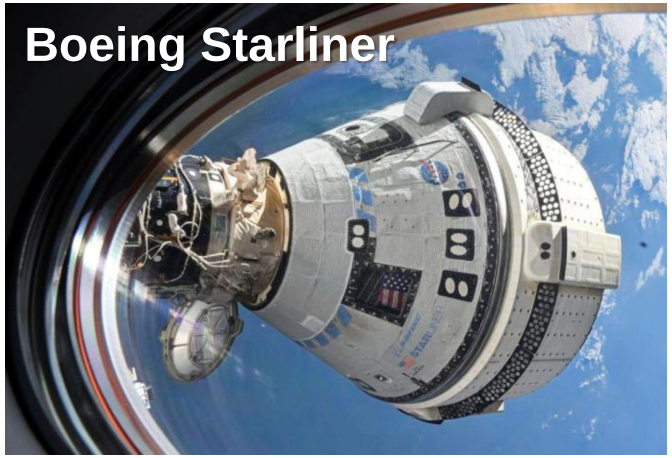


Commercial Crew Program

"…goal of safe, reliable, and cost-effective human transportation to and from the International Space Station from the United States through a partnership with American private industry."







Dragon has flown **36 astronauts** to the ISS on NASA missions (two crew rotations per year, four crew per flight)

Working with commercial partners on TPS

SpaceX Starship

Sierra Space Dream Chaser

Opportunities to work with NASA

Students:

- OSTEM Internships
  - Contractor; one-time internship
- Pathways Internships
  - Civil servant; co-op / rotations; eligible for conversion to full-time
- Graduate Fellowships
  - NSTGRO, MUREP, and GEM

https://www.nasa.gov/learning-resources/internship-programs/
→ Or search “NASA Intern”

Faculty:

- Small Business Technology Transfer (STTR) program → *Expected to open January 2025*
- Center Faculty Fellowships (JPL, GRC, …)
- NASA Early Career Faculty (ECF) program
- NASA Solicitation and Proposal Integrated Review and Evaluation System (NSPIRES)
- Partner with NASA PIs on their projects

For all opportunities, must be a US Citizen.

Challenges facing TPS

Changing Mindset

- Government operated vehicles for government-defined needs
- Infrequent flights

- Commercially operated vehicles
- Routine flights

Supply Chain

Limited Raw Material Supply

Limited TPS Vendors

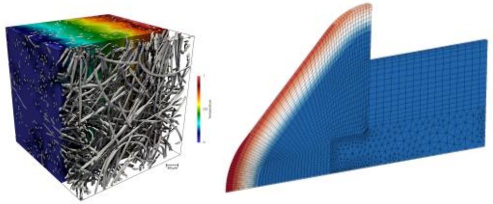
SPIRIT AEROSYSTEMS

TEXTRON

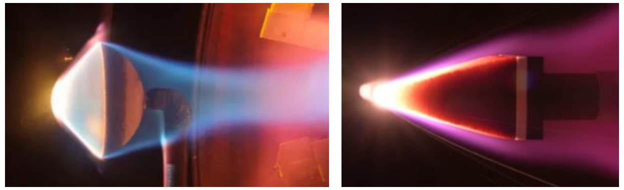
<u>Consequence</u>: Commercial space develops own infrastructure and capability to produce TPS at significant cost.

Testing

High Temperature Material Property Testing



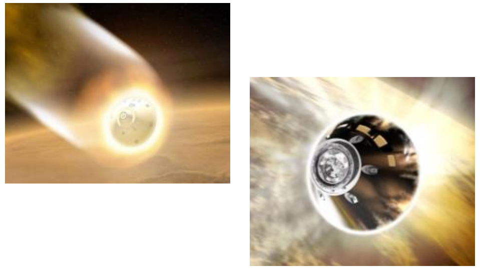
High Enthalpy Testing



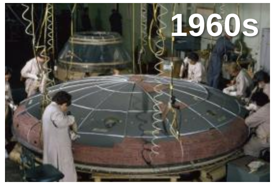
<u>Consequence</u>: Hesitancy to develop new TPS and/or reduced testing with increased risk of flight test failure.

Cost and Production Rate

Performance Over Cost



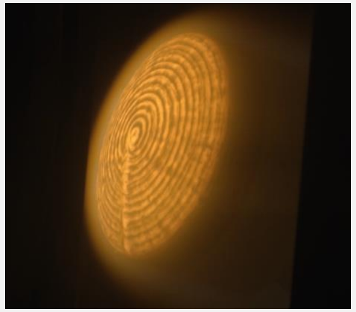
Lack of Manufacturing Innovation



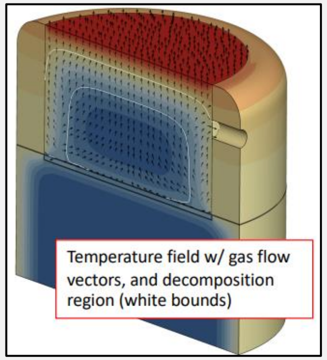
<u>Consequence</u>: Delays in vehicle development and flights. Fewer commercial companies prove successful.

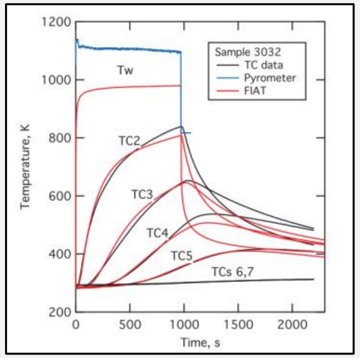
Thank you for your time!

Material Development



Material Response Modeling

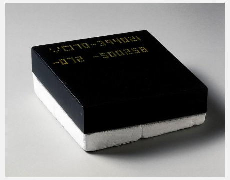


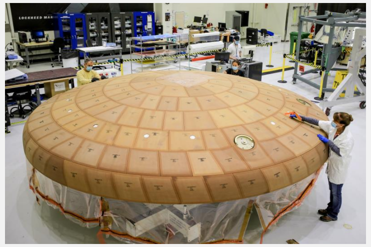


Commercial Partnerships

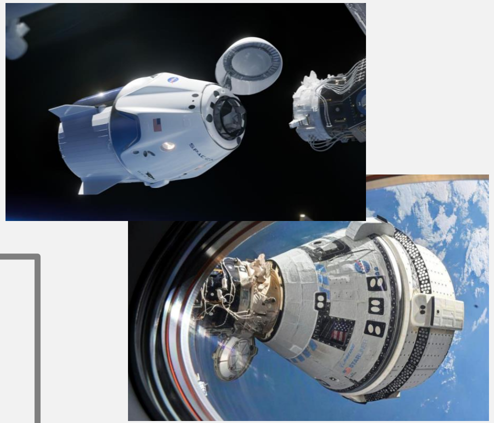
Orion TPS Management

Thermal Protection Systems at NASA JSC





Commercial Crew Program Support



Human Landing System (HLS) TPS Management

Community Engagement

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