Developments on Reusable TPS Materials Based Upon Shuttle Tile

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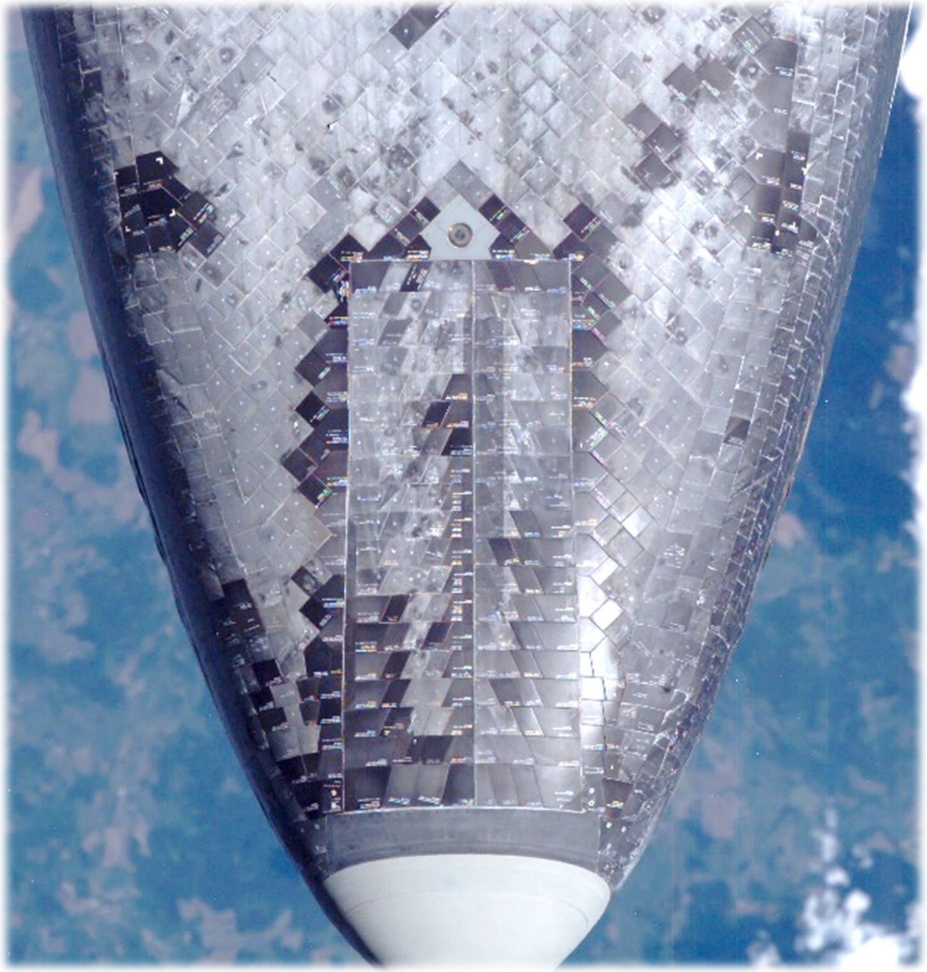
Outline

- Shuttle Tile Background and Modernization Effort
- AETB: Alumina-Enhanced Thermal Barrier
- AETB Processing and Trends
  - Raw Materials
  - Billet Casting Process
  - Billet Firing
- AETB Material Characterization
  - Composition
  - Meso-structure
  - Tensile Strength
- New vs Legacy Fibers
- Development/Exploratory Efforts



Background

- The Space Shuttle Orbiter's thermal protection systems (TPS's) are *the* flight proven starting point for reusable entry systems under development today
  - The Orion capsule also uses tile for its backshell but for fundamentally different reasons
  - The last decade has seen renewed interest in reusable TPS from the dramatic growth in commercial space
- Reusable, insulating TPS were developed extensively for Shuttle and some for the X-37B, but little else till recently
- Effectively there is a gap between the Shuttle efforts and now
  - Loss of personnel, know-how, supply chains, and equipment
- Aligned with NASA's strategic objective to support the commercial space sector, efforts at Ames Research Center include
  - IRAD to "restart" and modernize AETB processing
  - Technology transfer and arc jet testing support for commercial partners
  - Developmental, low TRL projects on novel solutions for reusable TPS
  - Overarching objective: R&D on all aspects of AETB processing and properties to aid the agency and partners

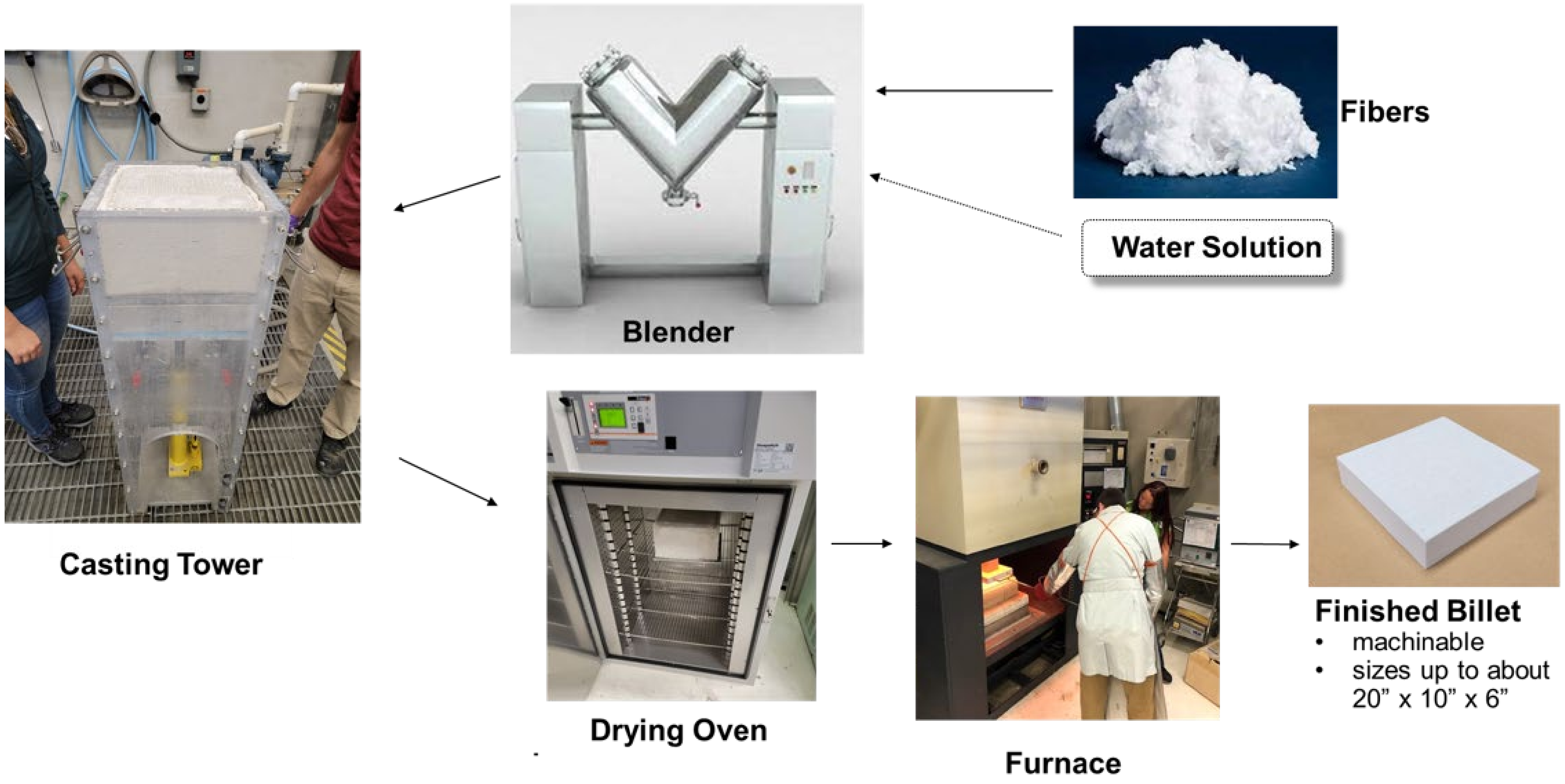
Ames AETB Restart and Modernization

- *What constitutes good AETB?*
  - Without a defined vehicle and flight profile, "what works" become ambiguous
  - Instead, can we develop and leverage materials and capabilities which are generally applicable
- After ~20yrs, does following the specification and work instructions still give the same result?
  - Shuttle-era documentation does not "stand alone" as an AETB blueprint in different contexts
- Are raw materials available today the same as they were >20yrs ago?
  - Both Shuttle and Orion operate on legacy stockpiles

- More than 67 billets produced starting since 2022 for R&D
  - Characterization and process-property correlations
  - New raw materials
  - Replacing worn/retired equipment
- Key processing parameters
  - Chop time, target cake thickness, compression fraction, etc
- Characterization: microstructure, phase, density, strength

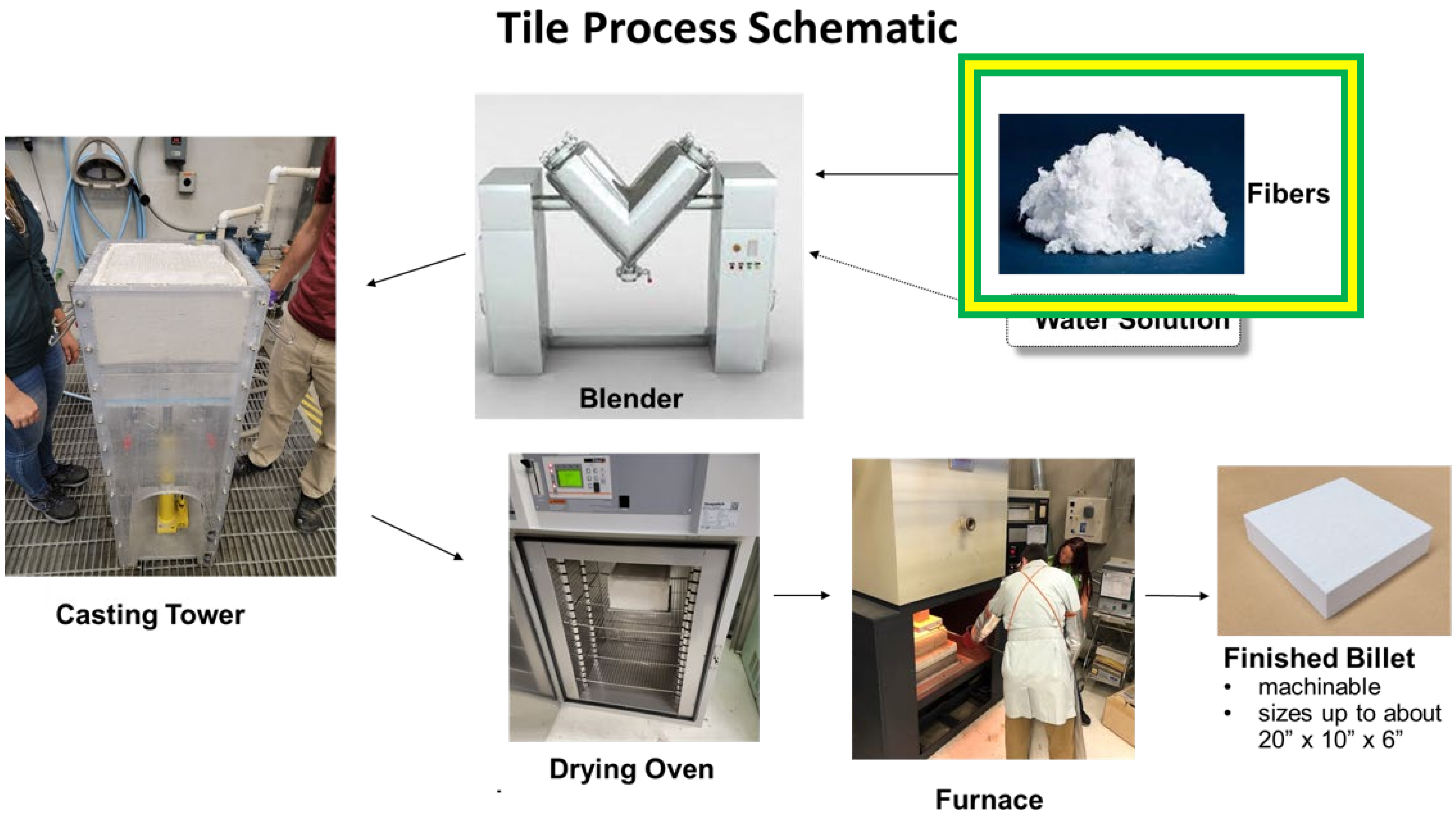
AETB Billet Process - Overview

Tile Process Schematic



- Multiple fiber constituents
- Wet slurry
- Blend
  - Mix constituents
  - Maintain fibers
- Cast
  - De-water
  - Form/compress
- Partial sinter
  - Develop strength and stability

AETB Billet Process



Raw materials/fibers

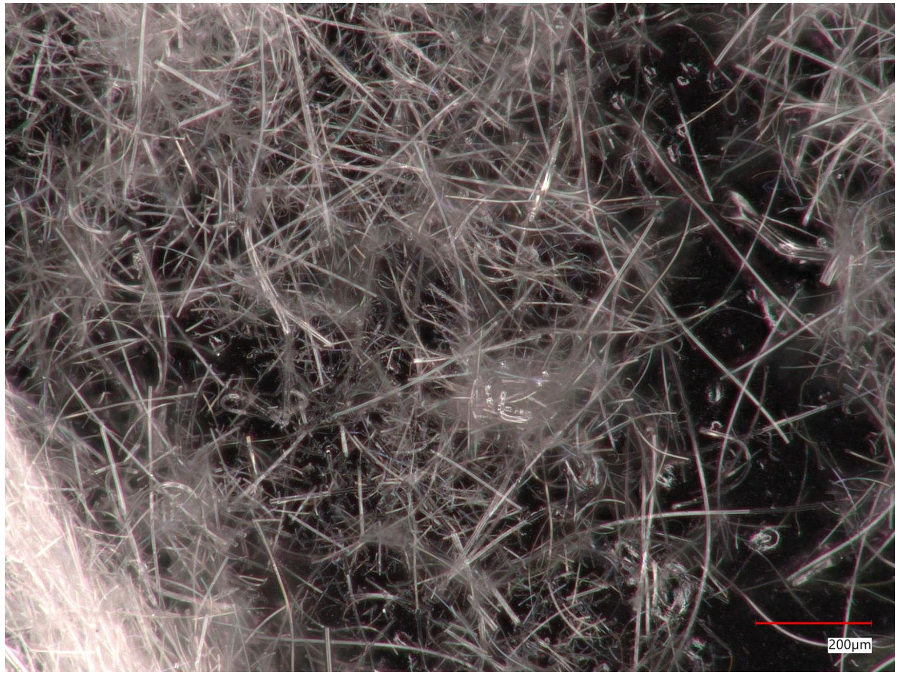
- High purity silica
- Alumina
- Aluminoborosilicate

- How the fibers are supplied and prepared affects downstream processing
- In many ways, an open-ended question

Fiber Raw Materials

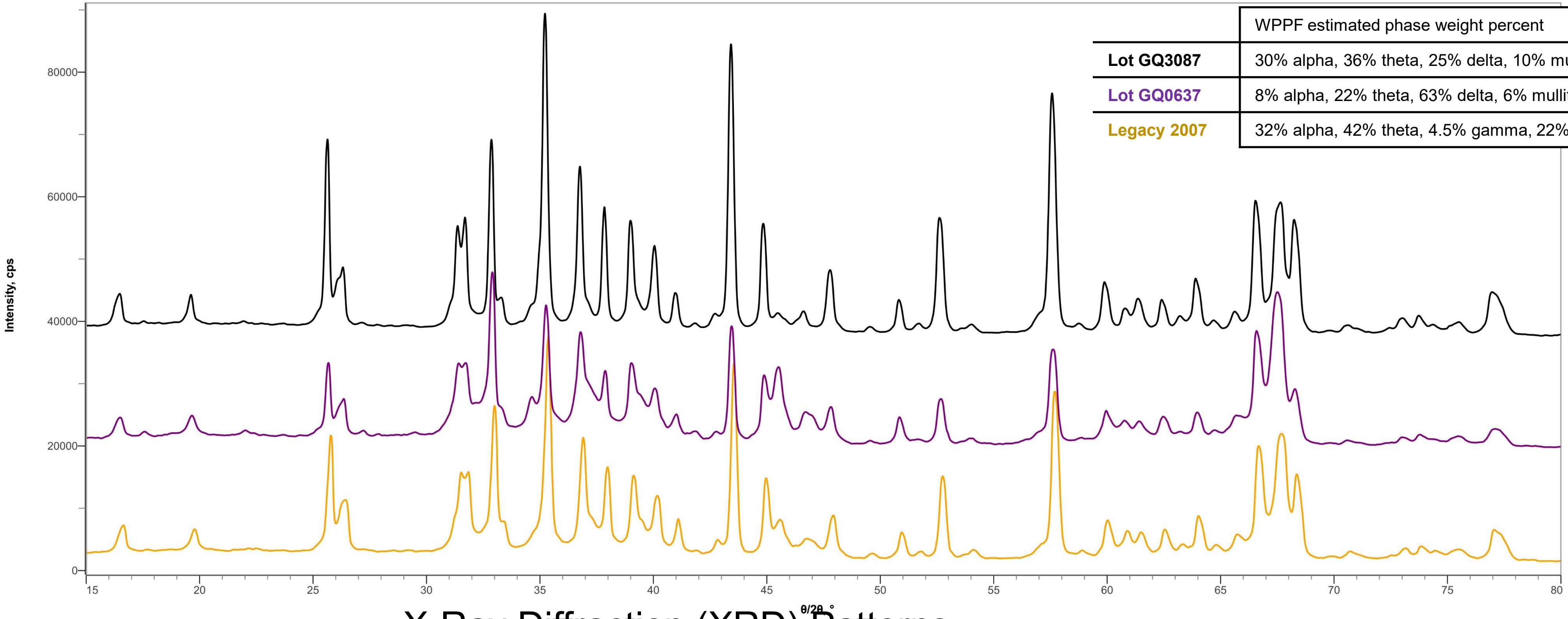
- Ceramic fibers are challenging to characterize
  - Length, diameter, uniformity
- Indirect metrics (i.e. final billet properties) will be correlated back to fibers used
- Highlight a few findings on Q-Fiber, Saffil, and Nextel fibers

- High purity silica (Q-Fiber)
  - Direct characterization limited in this project as others (within NASA and partners) have efforts underway



Fiber Raw Materials - Alumina

- Saffil HA has significant fraction of metastable/transition alumina phases
  - These are typically more accommodating of processing than the alpha/corundum phase
  - Do transition to the alpha phase on firing
  - To be determined if the different transition phases have an effect on chop or casting

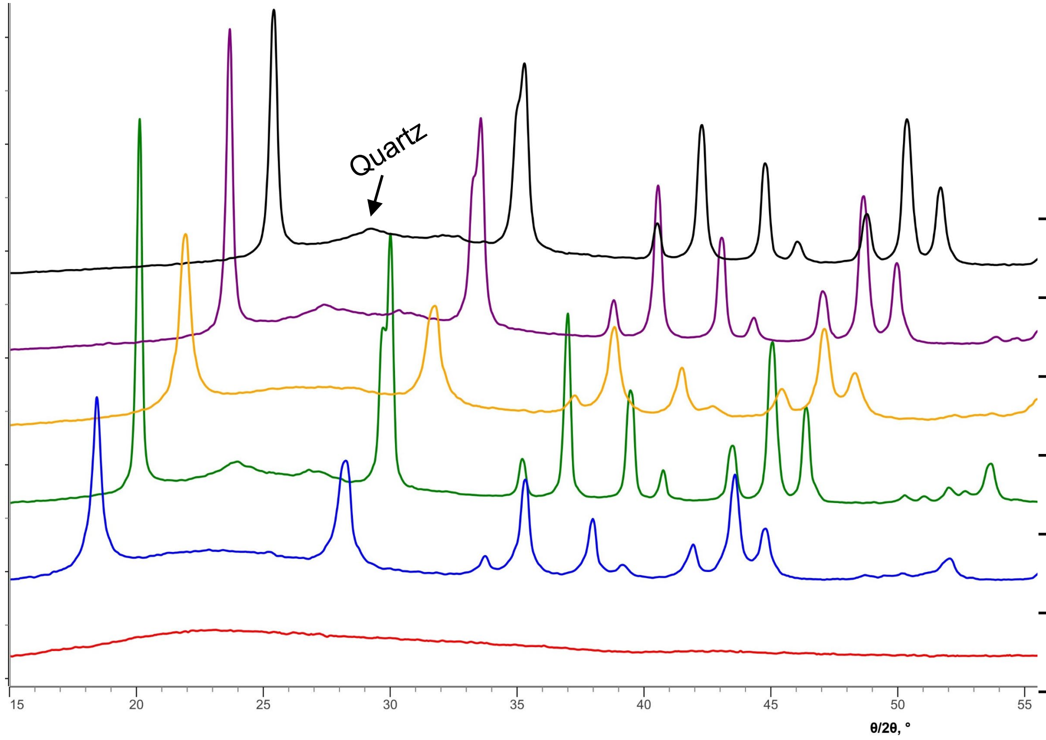


	WPPF estimated phase weight percent
Lot GQ3087	30% alpha, 36% theta, 25% delta, 10% mullite
Lot GQ0637	8% alpha, 22% theta, 63% delta, 6% mullite
Legacy 2007	32% alpha, 42% theta, 4.5% gamma, 22% mullite

X-Ray Diffraction (XRD) Patterns

Fiber Raw Materials - Aluminoborosilicate

- Nextel 312 provides boria to AETB as well as being refractory
- Fiber sizing is removed before use, but how hot it is fired (organic burn off) can effect chemistry and phase composition
  - Chemistry (by ICP) shows no significant change in boron up to 1200C
  - Firing does develop crystalline phases with some quartz devitrifaciton at 1200C



	WPPF estimated phase weight percent
Lot 5735 Box 2 Boat 4	88% (boro)mullite, 4.7% amorphous, 7% quartz
Lot 5735 Box 1 Boat 1	85% (boro)mullite, 7.1% amorphous, 8% quartz
Lot 439 Box 14	87.7% (boro)mullite, 12.3% amorphous
Lot N037098 1205C 90m	83% (boro)mullite, 7.9% amorphous, 9% quartz
Lot N037098 900C 4hr	89.5% (boro)mullite, 10.5% amorphous
Lot N037098 as received	Fully amorphous

AETB Billet Process

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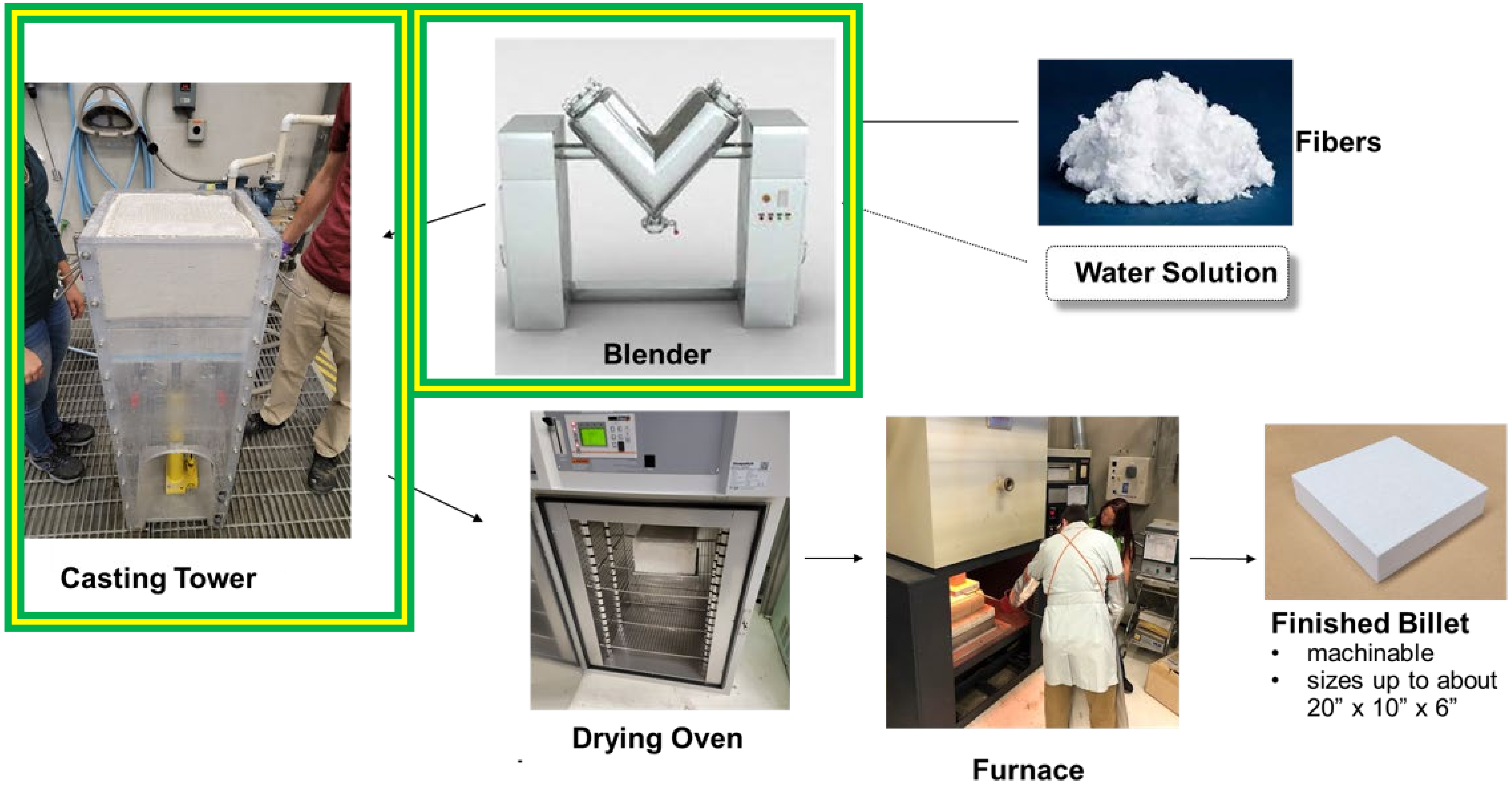


Blending/Casting

- Add-ins (surfactant, pH adjustment)
- Blend/chop process
- Casting process

- Mixing raw materials
- "Chopping"
- Cake formation
- Compressing wet billet for target density and anisotropy

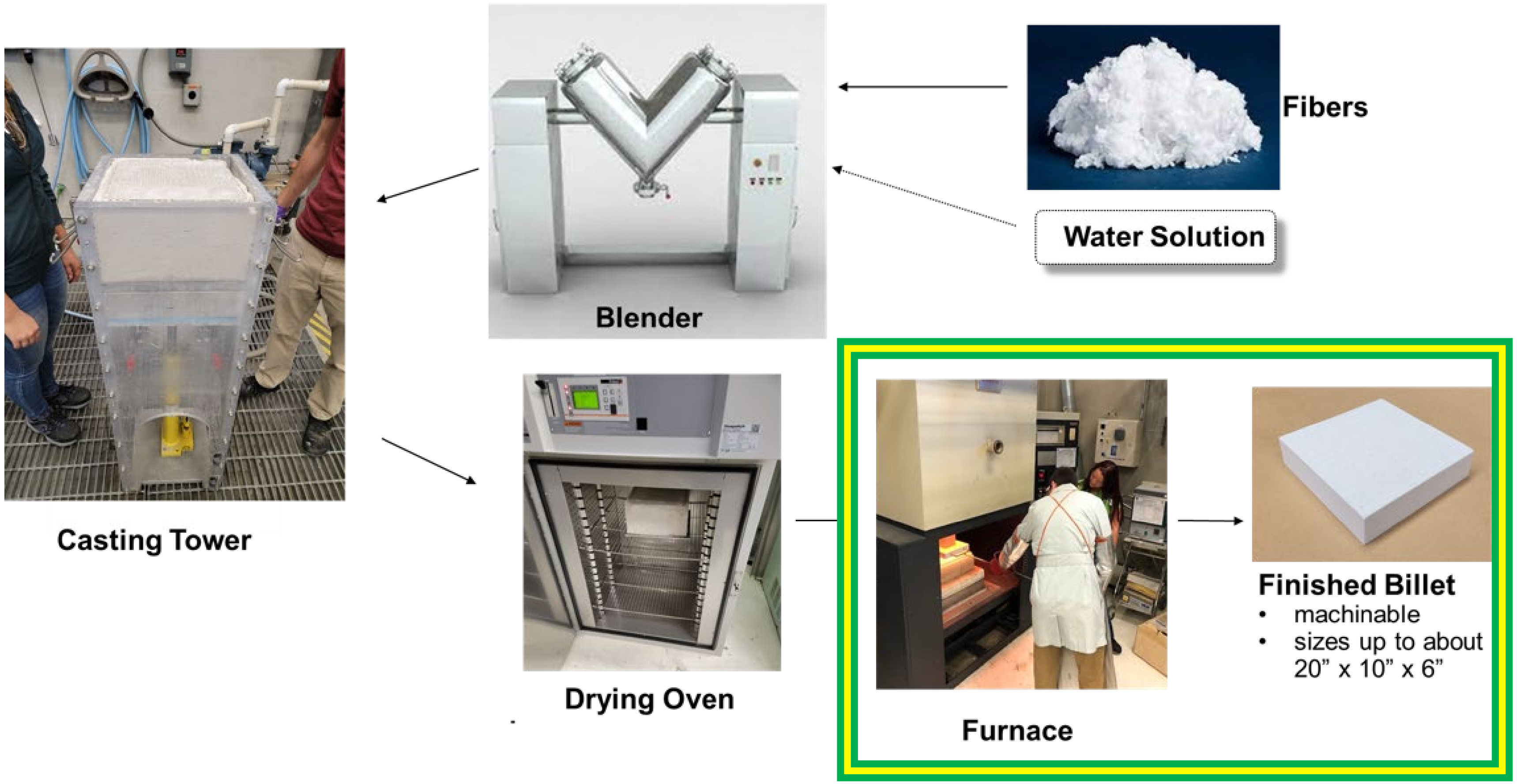
Casting Process Trends and Observations

Single Factor Observations	
Blend/Chop Time	Too short results in undermixed constituents and coarse structure. Too long results in cake height being too low (settled).
Blend/Chop Loading	Under or overloading the blender volume (slightly) is similar to running it for too short, or long, of a time.
Post-Blend/Chop “Mixing”	Upcoming study on different methods. If none is done, a few large voids and/or planes of weakness can end up in billet.
Surfactant	No surfactant leads to too low cake height. Further study ongoing.
pH Adjustment	Higher pH increased rate of chop: seen as lower cake height with higher pH (and vise versa) for constant chop time. Thus, chop time reduced for higher pH.
Casting Compression	Defines green billet size and related final density. With respect to cake height, controls the anisotropy (maybe).
Reproducibility	Can be +/-0.1pcf, pH within 0.1, and cake height +/-0.5” for a short run of billets.

Instructive trial billets

AETB Billet Process

Tile Process Schematic



Fired Billet Characterization

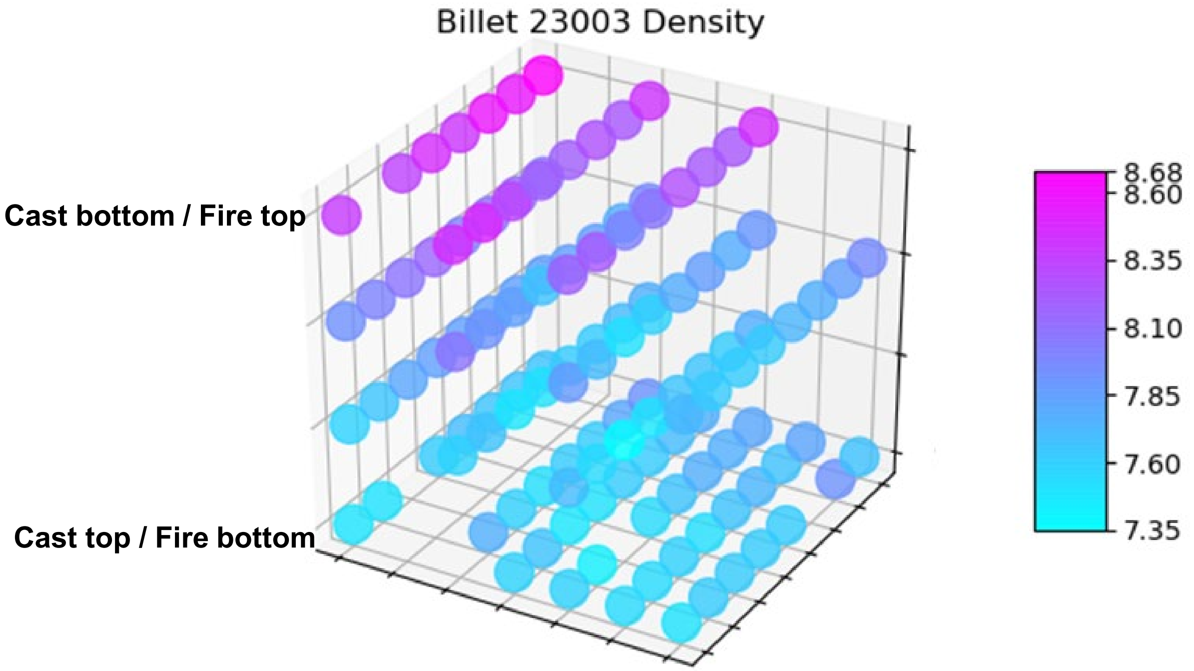
- Firing process
- Finished billet properties tie back to earlier steps

- Phase assemblage (cristobalite)
- Density and distribution of billet
- Tensile strength
- Void distribution and “texture”

AETB Density and Gradients

- Nominal Density
  - **Chop ↔ Cake thickness ↔ Compression ↔ Firing**
  - These are interrelated in processing to achieve the target density
  - Blend/chop to homogenize the fibers but still leave enough "bulk" for low density
  - Can be iterative process where shrinkage from firing feeds back into casting parameters

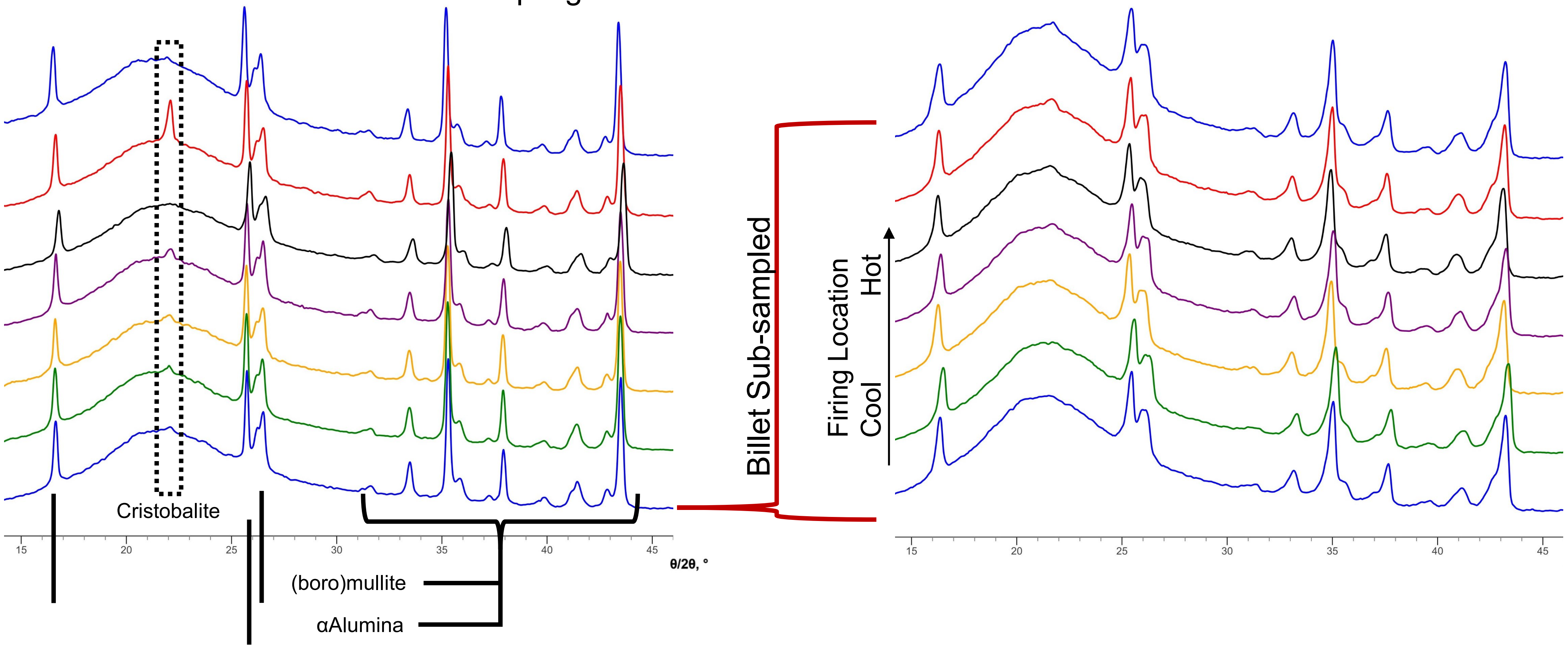
- Density gradient within billets
  - Casting: settling of slurry
    - Cast bottom denser
  - Casting: pressing direction
    - Moving platen side denser
  - Firing: creep
    - Densities fired bottom
  - Firing: gradients
    - Hotter and denser fired top



Composition – Phase and Chemistry

- Finished AETB composition by chemistry (ICP) and phase (XRD)
- Chemistry: no clear trend in B content; billets from new fibers are lower in Na, with similar Ca and Fe, compared to legacy
- Phase assemblage – sampled either as arbitrary specimens crushed to powder or systematically selected bulk specimens
  - Major phases similar across billets and location (amorphous, αAlumina, (boro)mullite) but cristobalite peak varies
  - Cristobalite content reduces strength and usable TPS life
  - Samples from hot and "cool" locations for firing show cristobalite content trends with temperature (as expected for devitrification)

AETB Billets – "Random" Sampling



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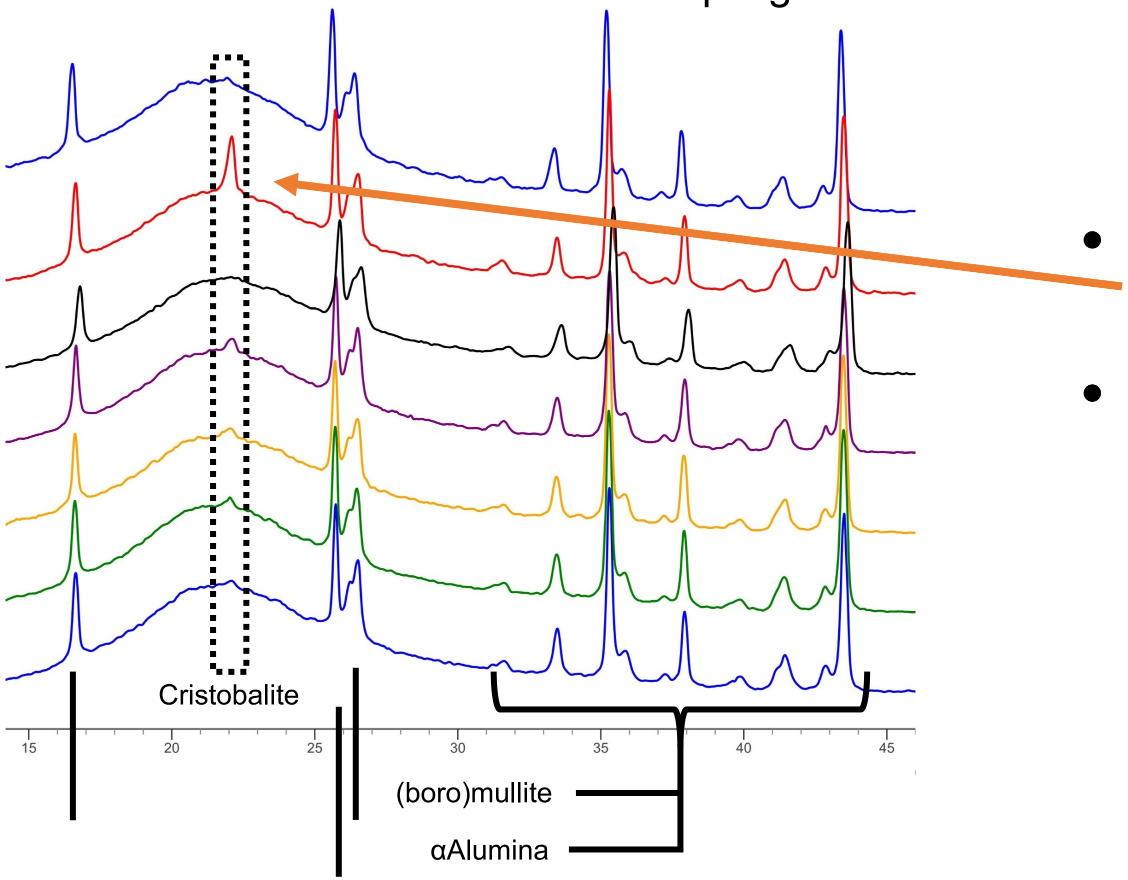
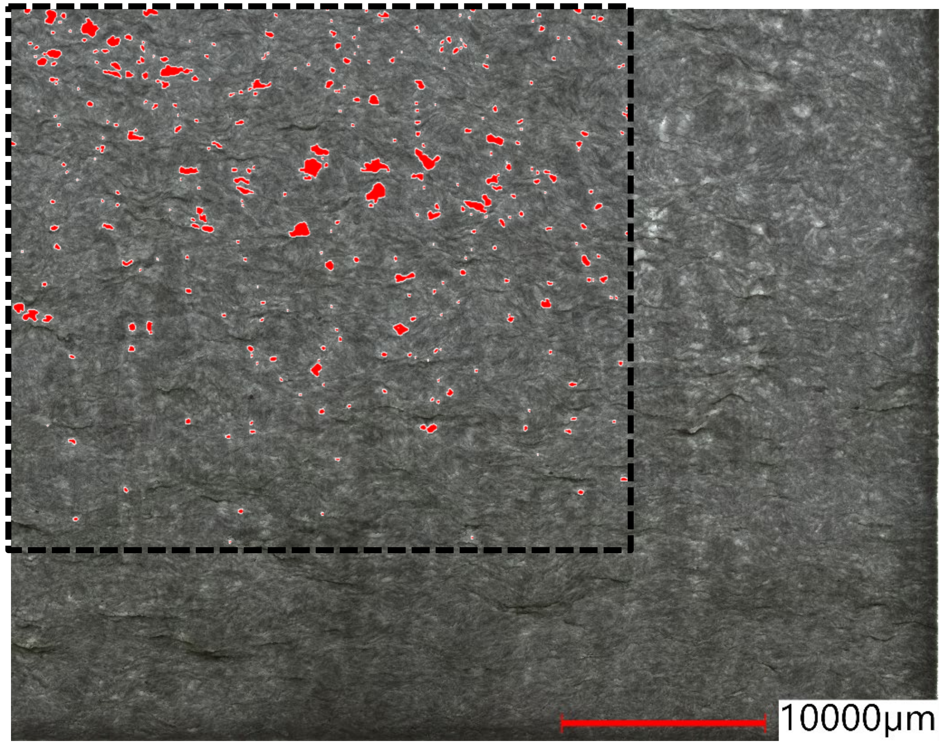
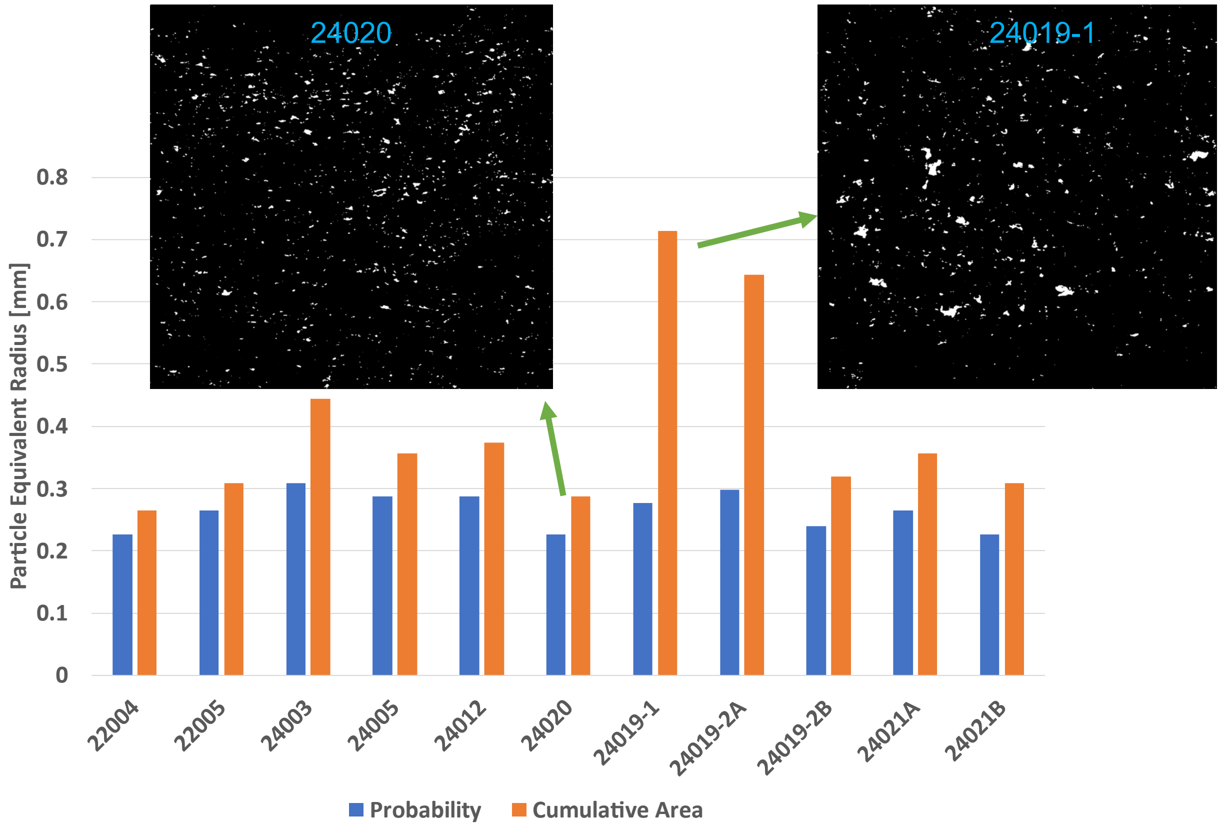


Next Steps

- Check cristobalite distribution within this billet
- Correlations to measured strengths

Void Distribution and "Texture"

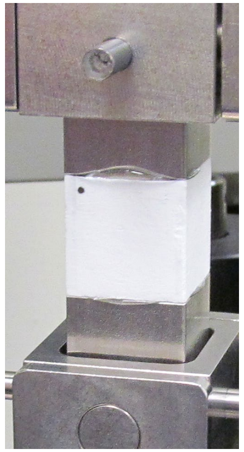




- Prompted by machined surface texture challenges for coatings
  - RCG (Reaction Cured Glass) is sensitive to voids around 0.1 to 1mm – not an easy size for inspection
- Developing technique to quantify the void/porosity distribution
  - Thin AETB slices imaged in transmitted light microscopy and threshold by brightness/opacity
  - Voids as 'particles' extracted and measured
- Size threshold for good porosity (insulating) vs large voids – fixed fraction or scaled to density
- Probability distribution (number by size) may minimize the effect of few, large voids. Cumulative void area distribution magnifies the effect of large voids.

Tensile Strength

- Specification AETB tensile testing is 1” cross section sample, bonded to pull blocks, in double-articulating fixture (ASTM D1623 type B)
  - Orientated in-plane (IP) or through-thickness (TT)



- Two sampling plans used
  - 1x1x1” samples taken throughout billet
  - 1x1x2” samples taken from one side similar to lot acceptance testing (LAT) plan
- Variability in strength is high. Compared to historical, strength is low but IP/TT ratio similar.
  - Density & phase distributions within billets
  - Size distribution of voids
  - Surface finish of machining

Billet ID	Density (lb/ft^3)	UTS		Sample		Cake Compression
		Rel. #23003	+/- (%)	Count	Length (in)	
23003 IP	7.8 ± 0.1	1	9.8	48	1	1.52
23003 TT		1	8.9	76	1	
24004 IP	8 ± 0.5	1.22	15.6	4	2	1.17
24004 TT		1.07	8.0	4	2	
24005 IP	7.8 ± 0.1	1.02	24.6	10	1	1.56
24005 TT		1.14	17.5	8	1	
24006 IP	8.1 ± 0.3	1.41	22.9	4	2	1.65
24006 TT		1.16	15.3	4	2	
24007 IP	7.8 ± 0.2	1.20	3.9	4	2	1.56
24007 TT		1.13	9.0	4	2	
24008 IP	7.6 ± 0.1	1.27	13.4	4	2	1.5
24008 TT		1.22	11.8	4	2	
24009 IP	8.2 ± 0.1	1.40	12.7	4	2	1.55
24009 TT		1.33	4.7	4	2	

New vs Legacy Fibers

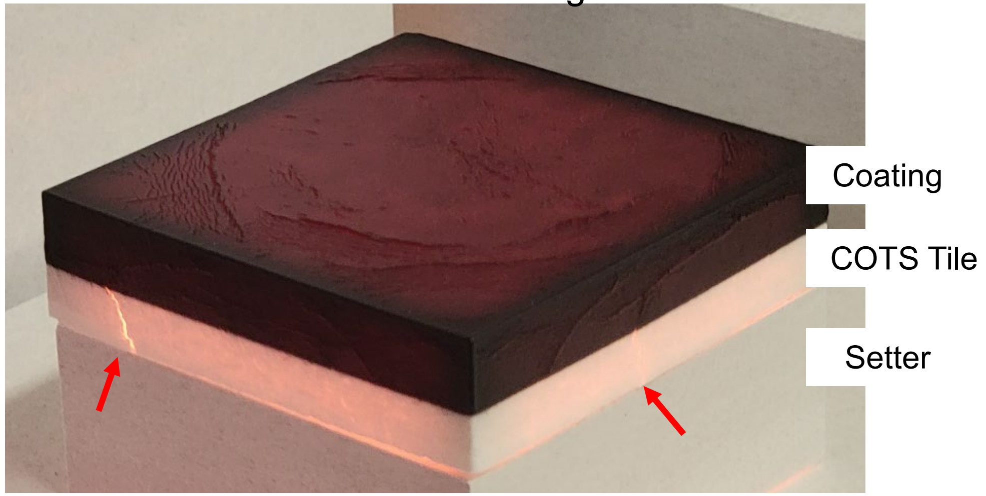
- Limited changes to the fibers, per manufactures. However, experience of AETB processing suggest significance.
- Casting change from one set/lot of new fibers to legacy did yield a significant difference
  - Legacy fibers vs new: higher pH and lower cake height (equivalent chop time)
  - Can't separate lot-to-lot variation given the small sample size
- No difference in fired XRD patterns
- Tensile strengths are statistically equivalent

Billet ID	Density (lb/ft^3)	UTS Rel. #23003	UTS +/- (%)	Sample Count
23003 IP	7.8 ± 0.1	1	9.8	48
23003 TT		1	8.9	76
24005 IP	7.8 ± 0.1	1.02	24.6	10
24005 TT		1.14	17.5	8

Developmental Exploration of Reusable, Insulating TPS

- Commercial, off the shelf (COTS) products promise reduced cost, variability, and greater availability
- Rigid fiberboards as drop-in replacement for tile
  - Thermal shock is an issue for most >1300C rated materials
  - Temperature rating needs to be understood further as time at temperature is very different for a kiln vs aerothermal entry
  - Thermal expansion matching is needed for tile coatings
- Alternatives to rigid ceramic billets/boards
  - AFRSI (Advanced Flexible Reusable Surface Insulation)
    - Larger, flexible "blankets" used on leeward side of Shuttle
    - Extensive stitching which added cost and was sensitive to fatigue failure
  - Looking at multiple avenues to bridge the gap between industrial batting and TPS application

Hot Unload from Coating Sinter



Summary

- Establishing variability and process sensitives for Alumina Enhanced Thermal Barrier (AETB) supports NASA missions and the commercial space sector
- Material characterization to understand and troubleshoot AETB processing
  - Composition of raw materials
  - Blending, casting, and firing parameters
- AETB properties and microstructural characterization techniques developed
  - Support for reusable TPS partnerships
- Tools and know-how extended from AETB to next generation reusable TPS development

Questions

National Aeronautics and Space Administration

Ames Research Center
Entry Systems and Technology Division