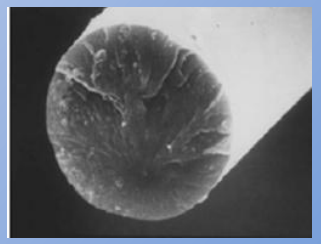
Ultra High Temperature (UHT) SiC Fiber



UHT Fiber Team and Expertise:

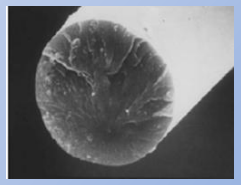
Dr. J. DiCarlo (PI) – Fiber Theory and Experimental Experience

Dr. N. Jacobson – High Temperature Chemistry

Dr. M. Lizcano – Material Science

Dr. R. Bhatt (OAI) – Ceramic Processing, Characterization

UHT Fiber: *Background*



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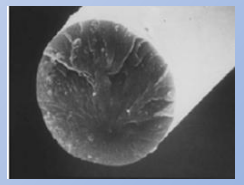
Ceramic Composites for Aeronautics

- The first generation of lightweight silicon carbide fiber-reinforced silicon carbide ceramic matrix composites (SiC/SiC CMC) with a temperature capability of 2200-2400^{o}F are on the verge of being introduced into the hot-section components of commercial and military gas turbine engines.

Prototype SiC/SiC airfoil

- In comparison to metallic components, these CMC components will not only reduce engine weight , but also reduce component cooling air requirements since metals can operate at best up to ~2100^{o}F. Reduction in cooling air would then have the additional engine benefits of reduced fuel burn and reduced harmful exhaust emissions.
- Although CMC with higher temperature CMC capability are highly desired by NASA, the AF, and the engine industry for further improving engine performance, the 2400^{o}F upper use temperature of current CMC is limited by the ~2500^{o}F temperature capability of today's best commercial SiC fiber, the NASA-developed Sylramic-iBN fiber.

UHT Fiber: *Objectives*



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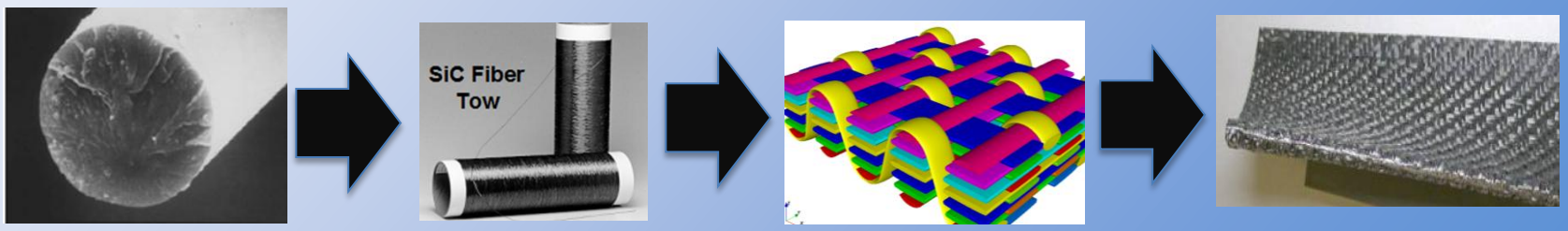
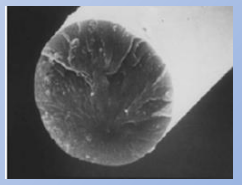
Starting with a commercial low-cost low-performance small-diameter (~10 µm) SiC-based fiber,

- Develop and demonstrate innovative thermo-chemical processes that convert this precursor fiber into a high-performance **Ultra-High Temperature (UHT) SiC fiber** with structural and thermal capability beyond that of the best commercial SiC fiber, thereby allowing SiC/SiC engine components to operate to 2700°F and beyond.
- Demonstrate that the UHT SiC fibers can not only be produced in single fiber form, but also within simple and complex preform structures of precursor fibers that are typically employed for SiC/SiC component fabrication



UHT Fiber: *Phase I Technical Approach*



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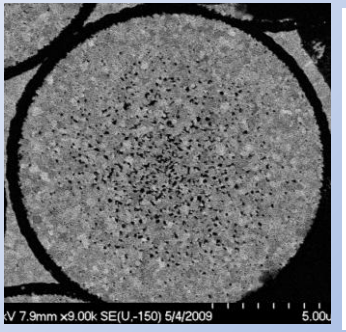
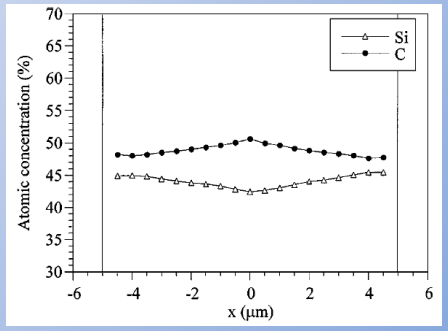
- Polycrystalline SiC fibers are thermally stable to well over 3000°F, but under stress will fracture with time at much lower temperatures due to creep and creation of flaws as grains slide over each other. Creep and fracture resistance can be improved by increasing grain size, grain size uniformity, and viscosity of grain boundary phases.
- Currently the state-of-the-art commercial SiC fiber is the NASA-developed "Sylramic-iBN", but is limited in temperature capability to ~2500°F due to a variety of microstructural issues, such as creep-resistant large grains only at the fiber surface, pores in the core region, and excess creep-prone carbon also in the core.





- Phase I approach will be to follow process steps similar to those of Sylramic-iBN fiber, but apply innovative thermo-chemical treatments that result in **a UHT fiber with larger grain sizes that are more uniformly distributed in the cross-section, with reduced pores, and with higher viscosity phases in the grain boundaries.**

UHT Fiber: *Innovativeness*

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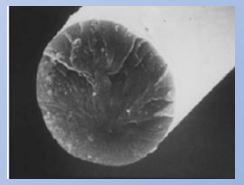
The UHT SiC fiber production approach is innovative in multiple ways in that

- ✓ It begins with a low-cost low-grade precursor fiber and coverts it by judiciously selected high-temperature chemical processes into a state-of-the-art high-performance SiC fiber with temperature and structural capability at least 300^{o}F higher than the best current SiC fiber
- ✓ It can be applied to precursor fibers within a variety of textile-formed architectures, which can range from continuous lengths of multi-fiber tows to the complex-shaped architectural preforms needed for reinforcement of multi-directionally stressed CMC components.
- ✓ It can be used for a wide range of commercial precursor fiber types with different additives that may provide extra beneficial properties to the final UHT fiber.
- ✓ It can be stream-lined with less process steps than currently employed for commercial state-of-the art SiC fibers, and thus be more cost-effective.
- ✓ It can produce high performance fibers with important properties other than greater temperature capability, such as, high thermal conductivity, and with surface coatings that are not only environmentally protective, but also compliant enough to provide the weak matrix bonding needed for tough CMC.

UHT Fiber: *Impact*



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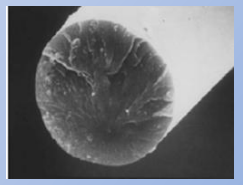
Besides addressing the **challenge of higher temperature SiC fibers** for higher temperature CMC components, this UHT fiber task will address **three other fiber-related challenges** for improved SiC/SiC hot-section engine components:

Challenge: High modulus and surface roughness of high-performance SiC fibers do not allow continuous-length tows to be formed into complex fiber architectures without fiber degradation and fracture.
Approach: Demo UHT fiber processes on highly deformable precursor tows after preforming them into complex shapes

Challenge: Acquisition costs for component preforms of high-performance SiC fibers can be more $10000 per pound due in large part to the multiple steps from continuous tow production to component preforming and shaping.
Approach: Demo cost-effective UHT fiber using (1) low-cost precursor fibers, (2) stream-lined processes, and (3) shaped preforms of final SiC/SiC components.

Challenge: Current production issues at the commercial vendor for producing high-quality Sylramic-iBN SiC fibers.
Approach: Develop a deeper understanding of high-performance SiC fiber processes for possible implementation at the vendor.

UHT Fiber: *Research Status*



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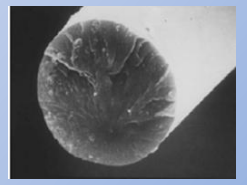
- **Current Progress towards <u>Phase I Technical Milestones</u>**
  1. Down-select UHT fiber process approach
  2. Purchase and characterize precursor SiC fiber
  3. Up-grade GRC fiber process and test facilities for UHT fiber
  4. Demo feasibility for UHT fibers
- **Summary Phase I Accomplishments**
- **Next Steps**

UHT Fiber: *Phase I Progress, Milestone 1*



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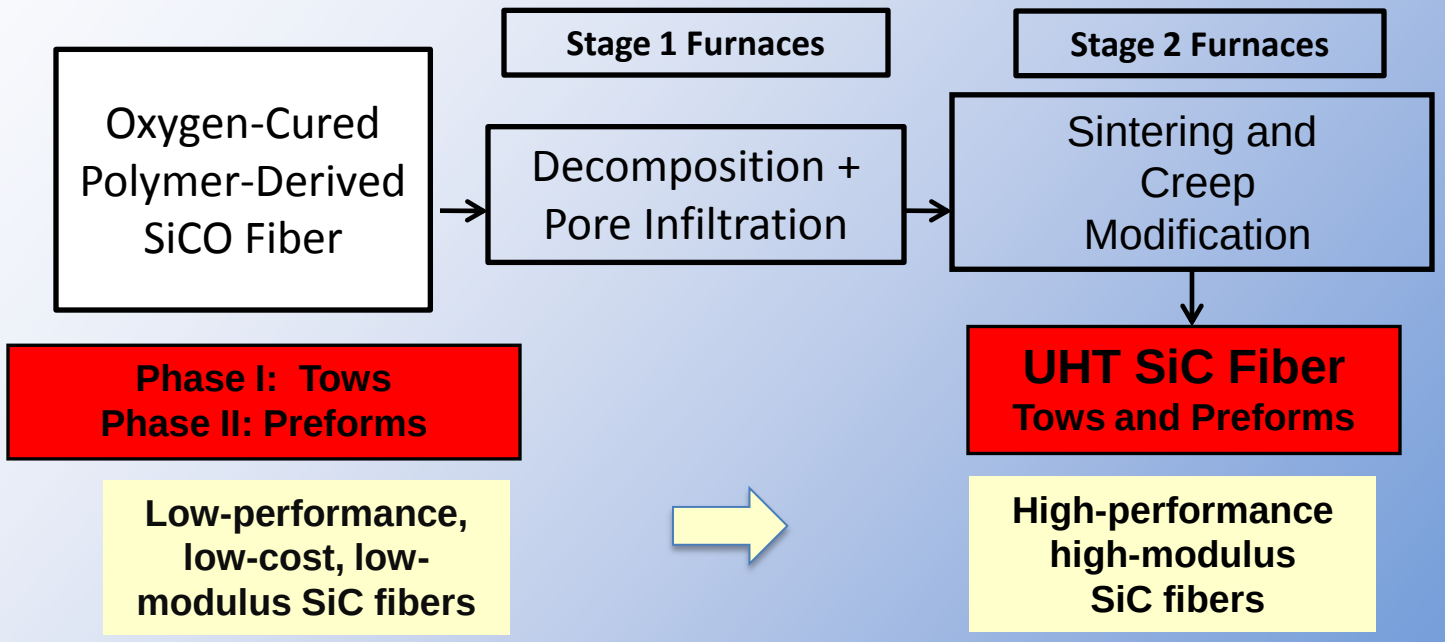
Milestone 1. Down-select UHT fiber process approach

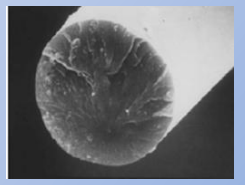


Reduced UHT costs due to starting materials, stream-lined processes, and final component fiber architectures

Phase I: boron-containing gases as pore infiltrants to set-up and verify GRC furnace facilities for producing a high-performance SiC fiber.

Phase II: alternate gas compositions to achieve UHT fiber microstructure.

UHT Fiber: *Key Performance Metric*



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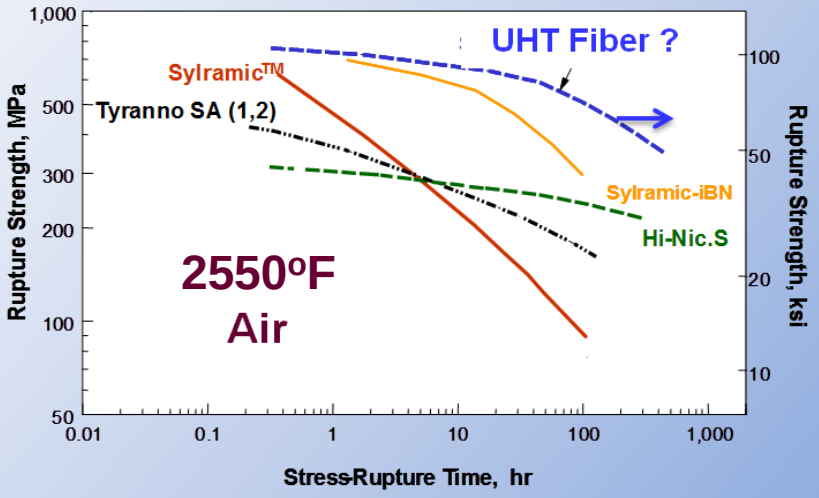
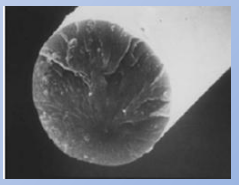
- NASA data concerning the time-dependent strength and strength retention of various high-performance SiC fibers at 2550°F in air is shown in the figure.



- **Key metric for the UHT SiC fiber will be to demonstrate that it can retain it's structural strength for longer times at 2550°F than current SOA Sylramic-iBN fibers.**
- **Actual upper use temperature would depend on stresses within a UHT-reinforced CMC component**

UHT Fiber: *Phase I Progress, Milestone 2*



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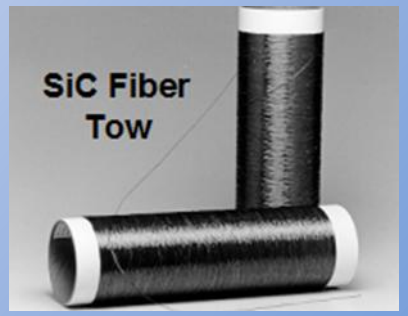
Milestone 2. Purchase and characterize precursor SiC fiber

One type of low-cost precursor SiC fibers have been acquired from two sources: (1) recently fabricated fibers in the form of spools of continuous multi-fiber tow and pieces of 2D woven fabric, and (2) long lengths of older tows of same which may possess slightly different quantities of chemical impurities that arise during production of these fiber types.



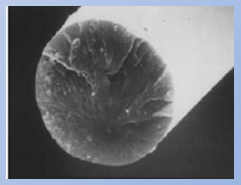
Chemical analysis of Source 2 Precursor Fibers

Element	Tow 1	Tow 2	Tow 3	Tow 4	Tow 5
Si, wt%	53.211	52.736	52.692	53.593	52.720
C, wt%	33.82	33.45	34.145	33.82	33.42
O, wt%	10.885	11.660	11.105	10.505	11.715
Ca,ppm	ND	44	ND	ND	ND
Fe, ppm	110	260	70	80	50
N, wt%	0.115	0.125	0.129	0.132	0.146
Na,ppm	ND	10	5	ND	2

ND-Not Detected

- **Starting C/Si ratio of precursor fiber tows is ~ 1.3, but needs to be decreased to ~1.0 during processing for a high performance UHT SiC fiber.**
- **Precursor tows should have low metallic content to avoid exaggerated grain growth during processing that will cause fiber strength degradation.**

UHT Fiber: *Phase I Progress, Milestone 3*



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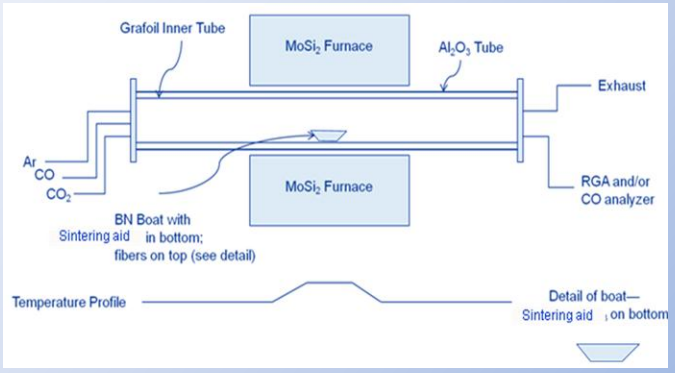
Milestone 3. Up-grade GRC fiber process and test facilities for UHT fiber

Stage 1 Facilities for Decomposition and Pore Infiltration



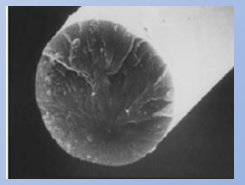
Graphite tube inside alumina tube with BN spacers

Small Research Furnace

Gases ~1 atm

Small Production Furnace

UHT Fiber: *Phase I Progress, Milestone 3*



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Stage 2 Facilities for Sintering and Creep Modification

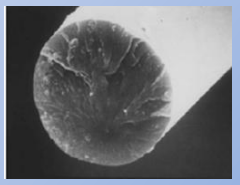
Small, 1 atm.

Medium, 1 atm.

Large, high atm.

Large, 1 atm.

UHT Fiber: *Phase I Progress, Milestone 3*



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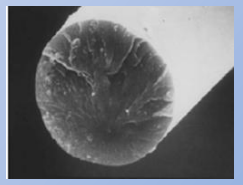
- Initially short tow lengths are being processed for microstructural characterization and process optimization.
- Larger lengths will then be processed for mechanical testing

Specimen Holders

Stage 1 Production Furnace Holder

Small Stage 2 Furnace Holders

UHT Fiber: *Phase I Progress, Milestone 3*



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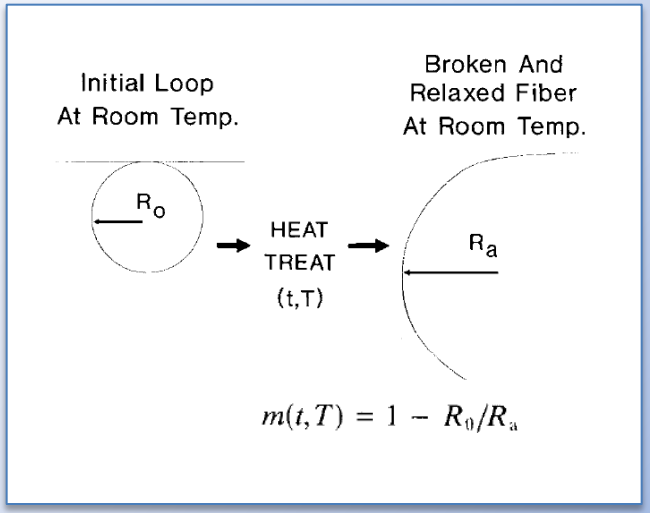
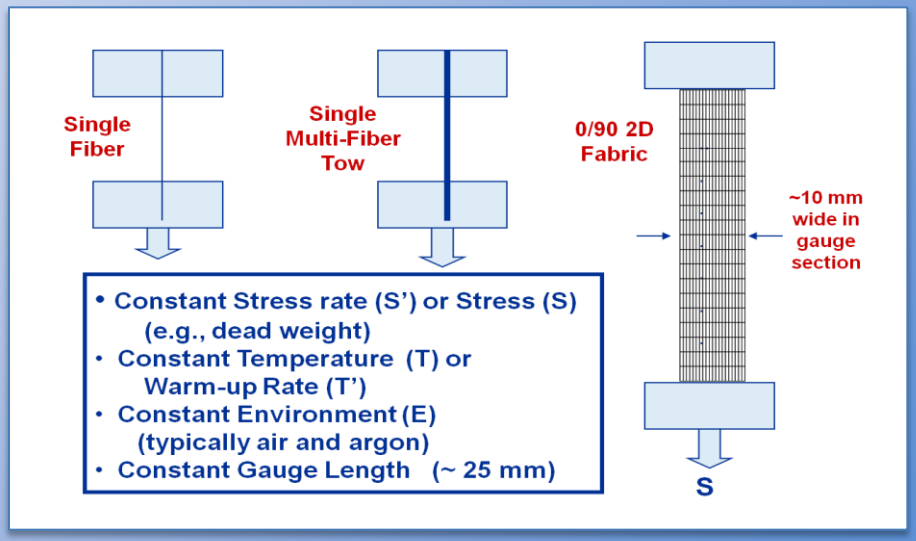
Fiber Characterization and Test Facilities

GRC: SEM, TEM, chemical analysis, TGA, RGA, microprobe
Case Western University: Auger Surface Analysis (draw contract)
GRC: Mechanical :
- Bend Strength and Bend Stress-Relaxation for short single fibers up to 1600°C in argon
- Tensile Strength, Creep, Rupture for longer single, multi-fiber tows, and fabric up to 1400°C in air

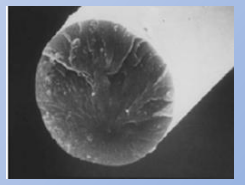


Fiber Bend Tests



Fiber Tensile Tests

UHT Fiber: *Phase I Progress, Milestone 4*



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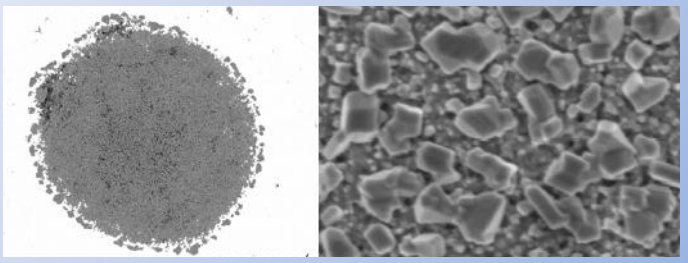
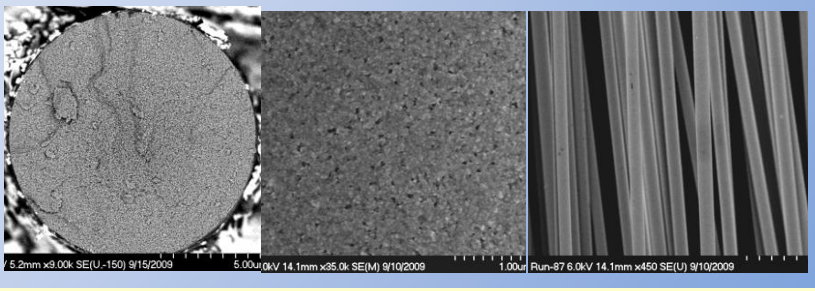
Milestone 4: Demo feasibility for UHT fibers (Four key steps)

Milestone 4A: Down-select temperature, time, and gas conditions in **Stage 1 furnace** to decompose precursor fiber tows, leaving fine size pores and grains uniformly distributed across each fiber cross-section**.**

Stage 1 Research Furnace

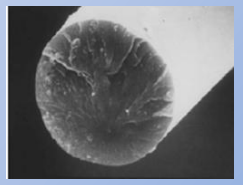


Stage 1 Production Furnace



- **Stage 1 Production Furnace allows better gas composition and flow control resulting in desired output of decomposed precursor fibers with fine and uniform grains in cross-section and on surface, plus well-separated and handle-able fibers within tow**

UHT Fiber: *Phase I Progress, Milestone 4*



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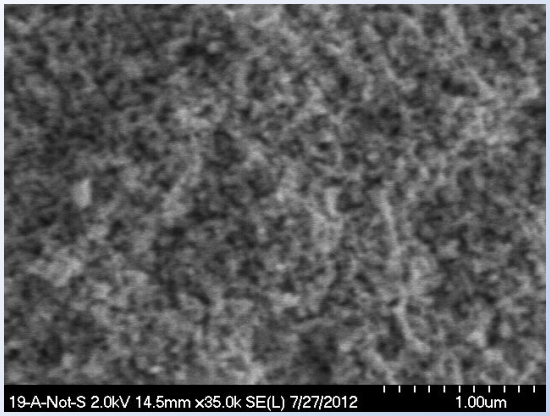
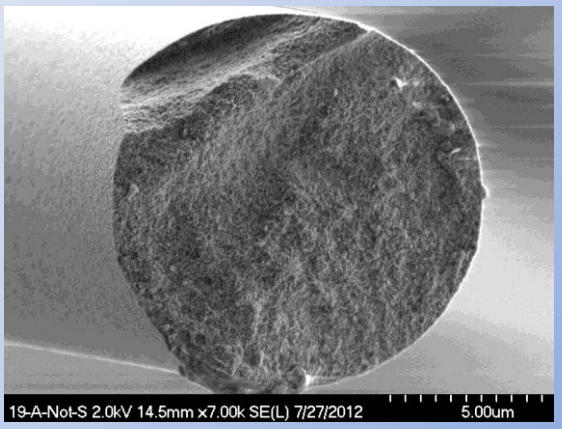
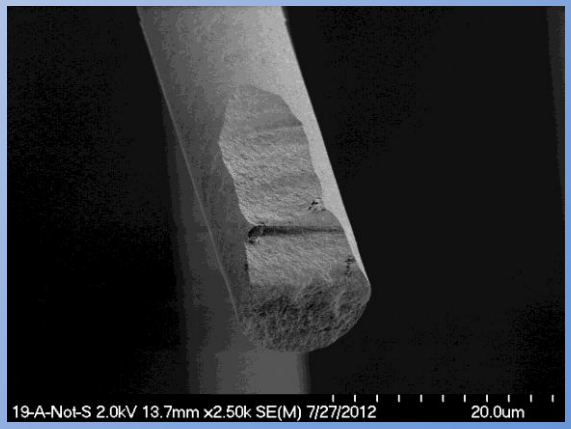
Milestone 4B: Down-select temperature, time, and gas conditions in **Stage 1 furnace** to infiltrate boron into the fine pores of the precursor fiber tows, leaving a boron-containing sintering aid uniformly distributed across fiber cross-section with no carbon-rich core.

Stage 1 Production Furnace

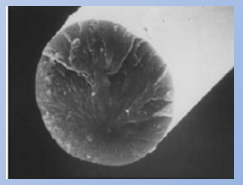






- **Decomposition plus boron infiltration in Stage 1 Production Furnace has resulted in excellent microstructures with uniform grain size and boron distribution.**
- **Results have provided new insight to the UHT Team on the proper conditions not only for precursor decomposition, but also for boron infiltration.**

UHT Fiber: *Phase I Progress, Milestone 4*



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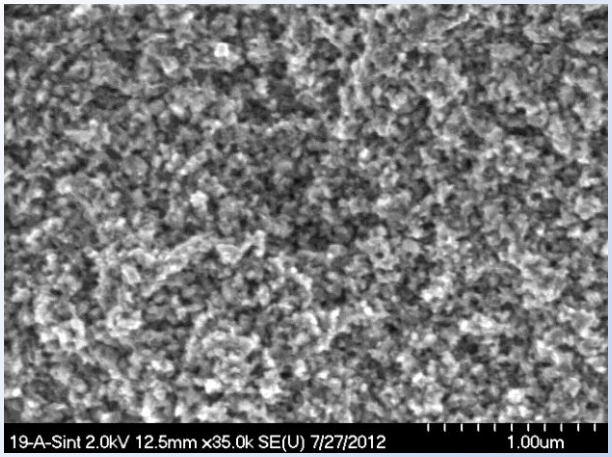
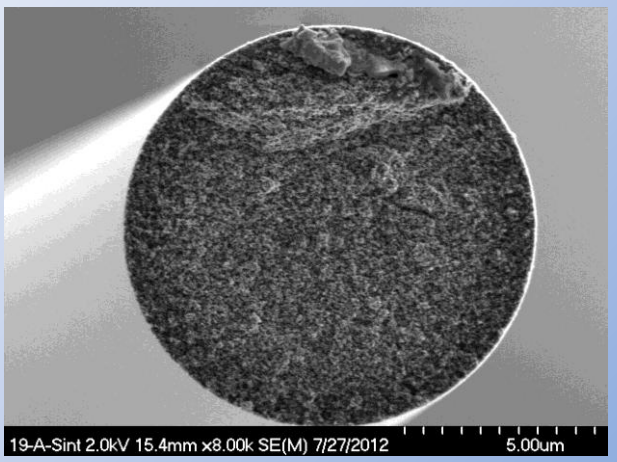
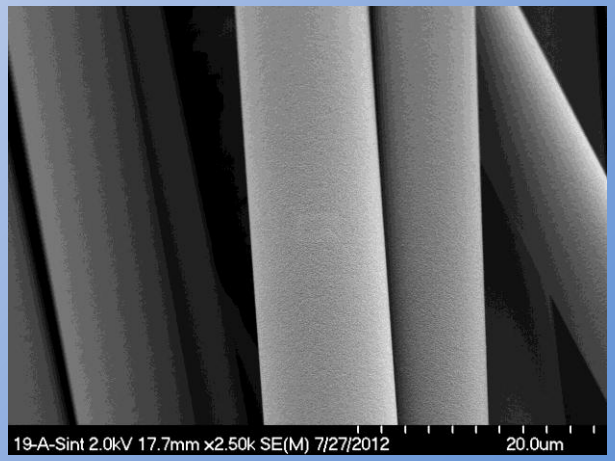
Milestone 4C: Down-select temperature, time, and gas conditions in **Stage 2 furnace** to allow boron-sintering aids to remove all pores and grow grains into a uniform distribution across each precursor fiber cross-section with as large a size as possible.

Stage 2 Small Sintering Furnace

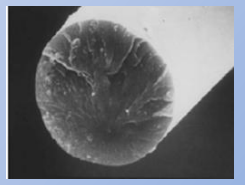






- **When initially sintered, precursor fibers after decomposition and boron infiltration in small production furnace showed good fiber densification with a uniform distribution of small grains, but perhaps with excess boron in the outer rim.**
- **Sintering studies are continuing to grow these grains further for improved creep resistance and higher temperature capability.**

UHT Fiber: *Phase I Progress, Milestone 4*



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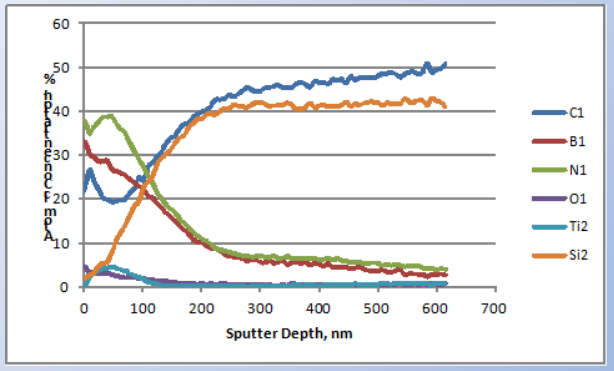
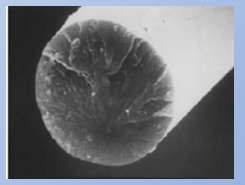
Milestone 4D: Down-select temperature, time, and gas conditions in **Stage 2 furnace** to allow a nitrogen atmosphere to remove boron from the precursor fiber tows, infusing creep-resistant silicon-nitride into grain boundaries of each fiber, and forming a thin protective BN coating on each fiber surface.



- **Nitrogen treatment has yet to be performed in Phase I, but NASA has prior experience (US Patent 7687016-B1) that this process is indeed feasible and will significantly enhance the performance of the final UHT fiber and its composites.**
- **Figure shows an Auger depth analysis of a boron-doped Sylramic fiber after nitrogen treatment, indicating formation of thin BN layer and infusion of nitrogen.**
- **Compliant in-situ grown BN layer not only improves fiber strength by filling in fiber surface flaws, but also provides environmental protection.**

UHT Fiber: *Current Phase I Accomplishments*



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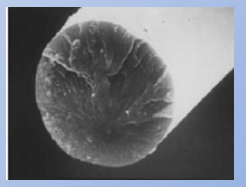
- All the equipment and safety permits required for the initial UHT fiber processes have been assembled, set up, and up-graded, and are now in place in two GRC buildings.
- Low-cost precursor fiber acquired in tow and fabric forms, and chemically characterized for major and impurity elements.
- Fiber microstructural characterization methods established and up-graded in terms of turn-around time and analysis across fiber cross-section.
- Stage 1 process conditions determined for achieving optimum precursor microstructures after decomposition.
- Feasibility demonstrated for Stage 1 boron infiltration and subsequent Stage 2 fiber densification, **but both processes have yet to be optimized**.
- Innovation has moved from basic principles (TRL1) to formulated concept (TRL 2)

UHT Fiber: *Next Steps*



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- **Finalize time-temperature-gas conditions in Stage 1 Production Furnace for pore infiltration and optimum cross-sectional microstructures.**
- **Optimize Stage 2 Furnace conditions for fully densifying fiber and increasing its grain size and creep resistance without debiting fiber strength (~3 GPa).**
- **Demonstrate enhanced UHT fiber thermal and mechanical properties in comparison to current SOA Sylramic-iBN SiC fibers.**
- **Demonstrate optimized process conditions that can be practiced on tightly contacting fibers in simple and complex-shaped preforms for CMC components**
- **Determine feasibility of enhancing all processes in terms of increased fiber performance, streamlined process steps, and reduced process costs.**
- **Report all successful results to the NASA ARMD, Air Force, and other interested government agencies to determine the best path forward**
- **Work with outside ceramic processors to determine feasibility of technology transfer for eventual commercialization of the UHT fiber and processes.**