Development and Characterization of SiC/SiC Composites for 3000°F Hypersonic Vehicle Applications

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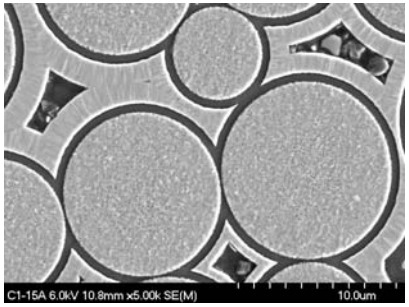
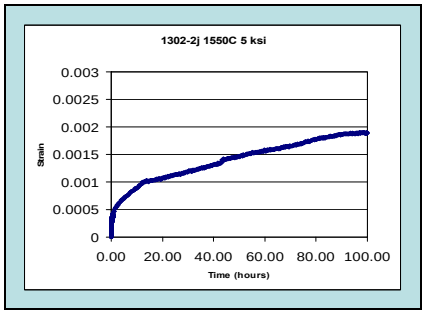
Research Supported by ARMD FA Hypersonic Project
Hypersonic Materials and Structures





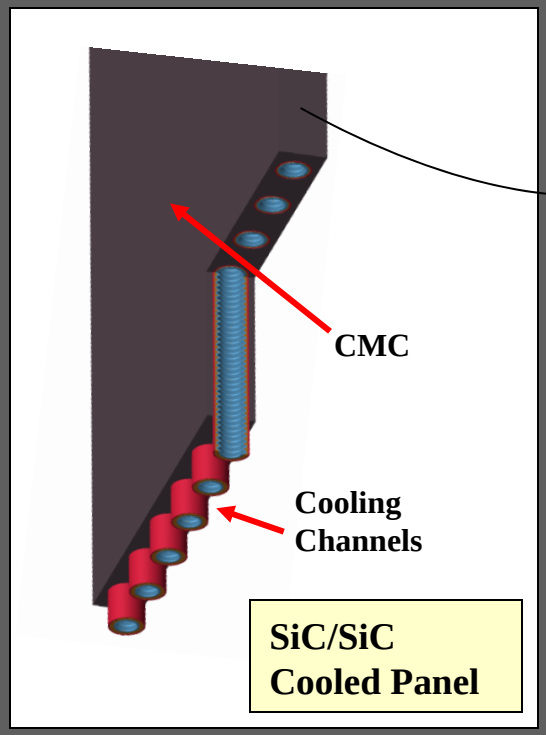
Outline

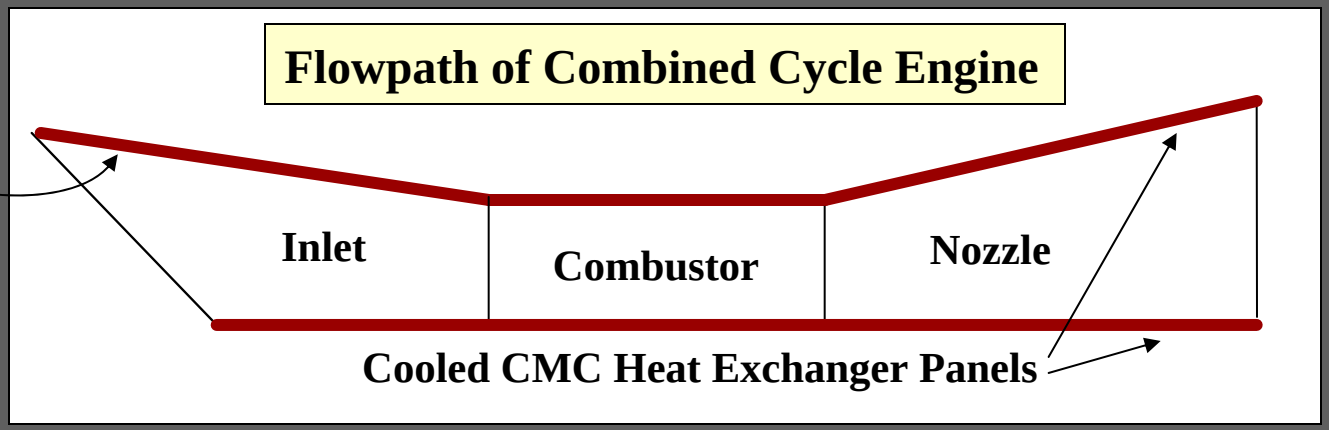
- **SiC/SiC Composites**
  - Applications and materials selection considerations
  - NASA development and characterization of CVI, PIP, and Hybrid SiC/SiC CMCs (ceramic matrix composites) for reusable high speed (hypersonic) vehicle applications
- **Coordination of SiC/SiC development efforts with AFRL (WPAFB)**
- **Summary**

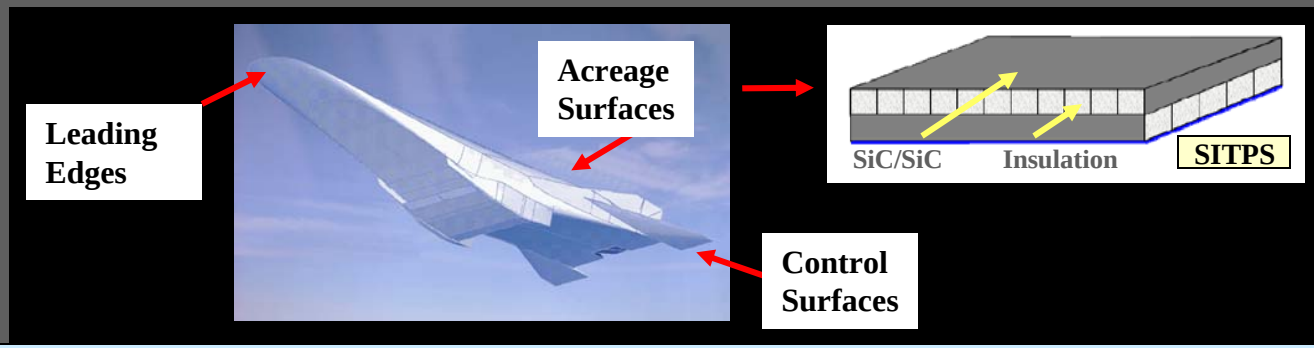
Potential Applications of CMC Components in Reusable Launch Systems (RLS)

PROPULSION

Actively-cooled Components (Cooled Heat Exchanger Panels and Combustion Chambers (Hot Gas Flow Path)), Uncooled Thin Wall Structures, and Turbomachinery Components (Inserted Blades)





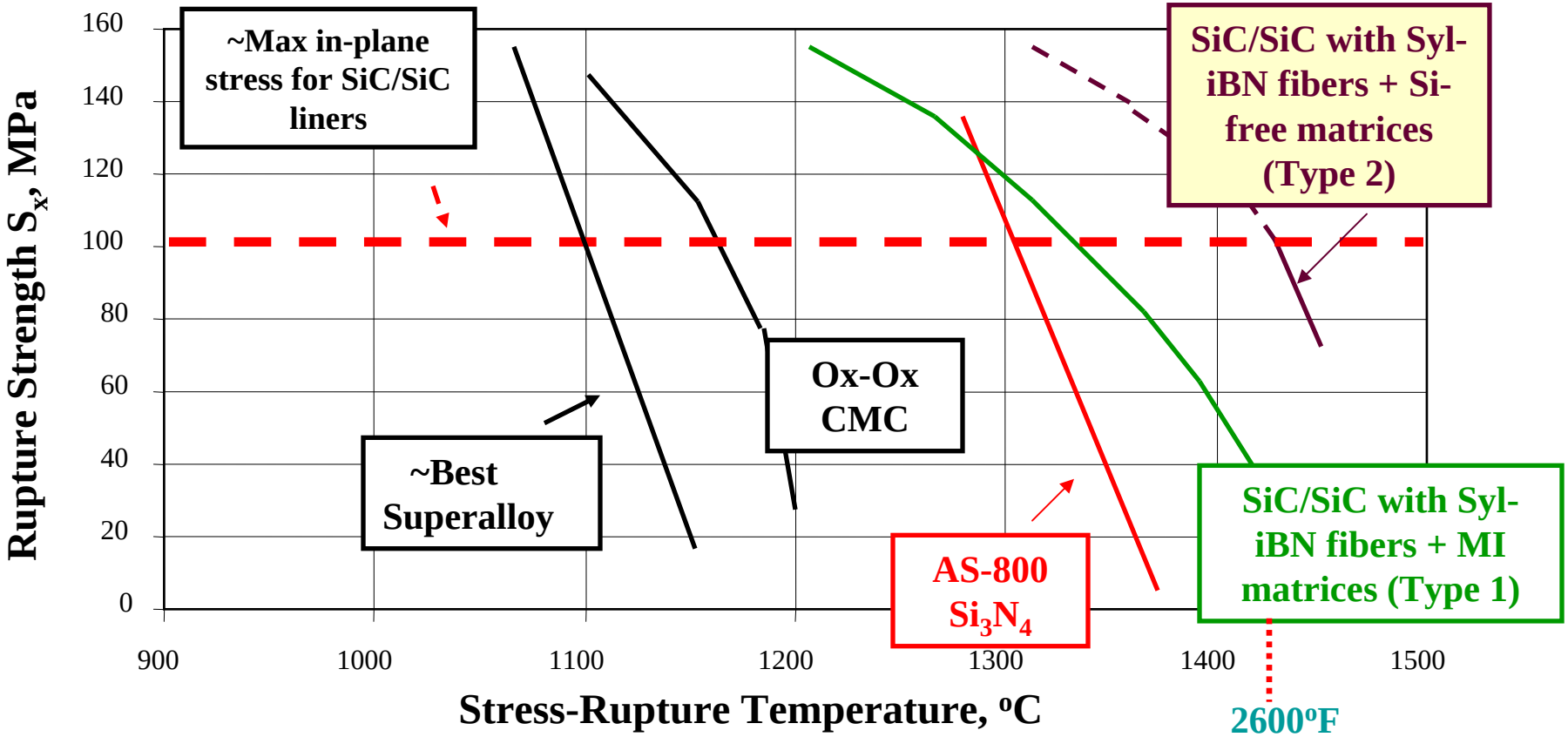


AIRFRAME

Leading Edges, Hot Structures, Acreage Surfaces, and Seals

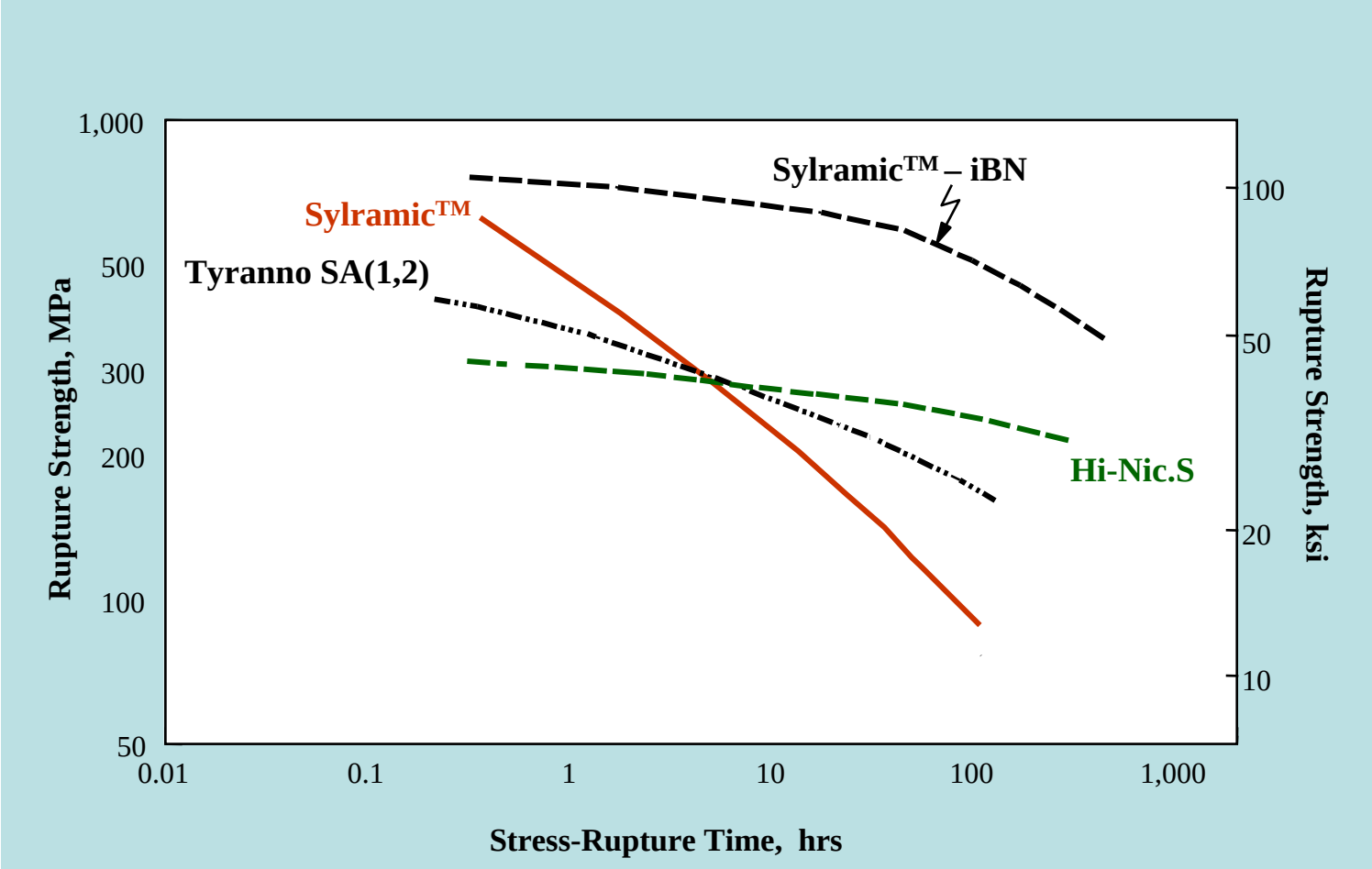
Materials Selection Considerations

500-Hour Rupture Strength in Air for <u>Reusable</u> High-Temperature Structural Materials



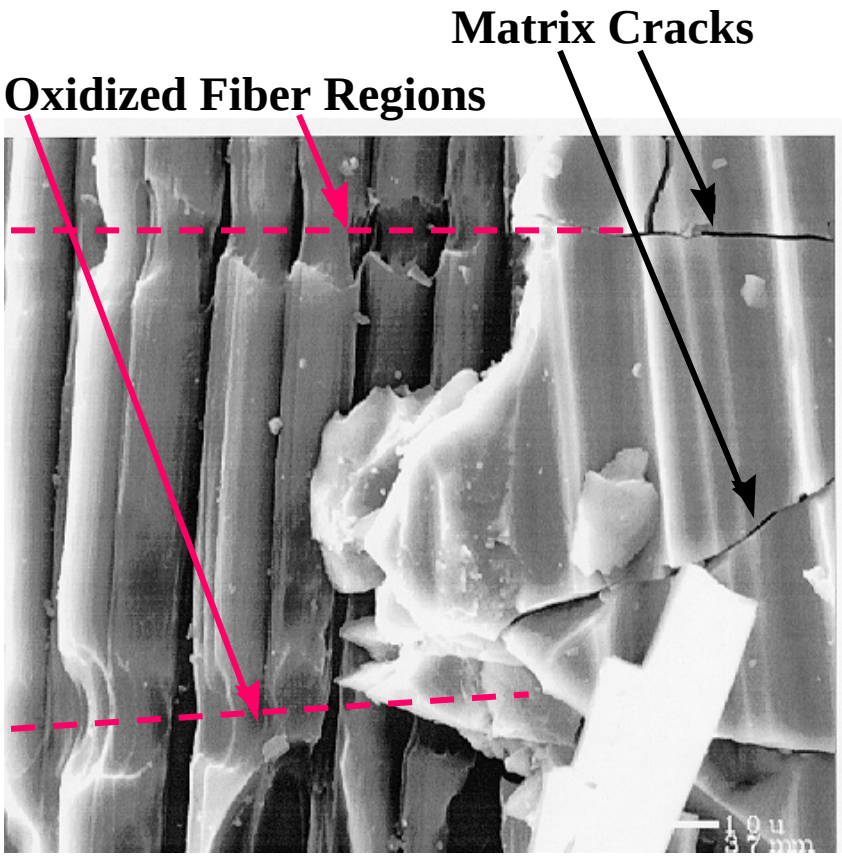
Thermostructural capability for Type 2 SiC/SiC system is state-of-the art

Stress-Rupture Behavior of Different High-Performance SiC Fibers at 1400°C (2552°F) in Air

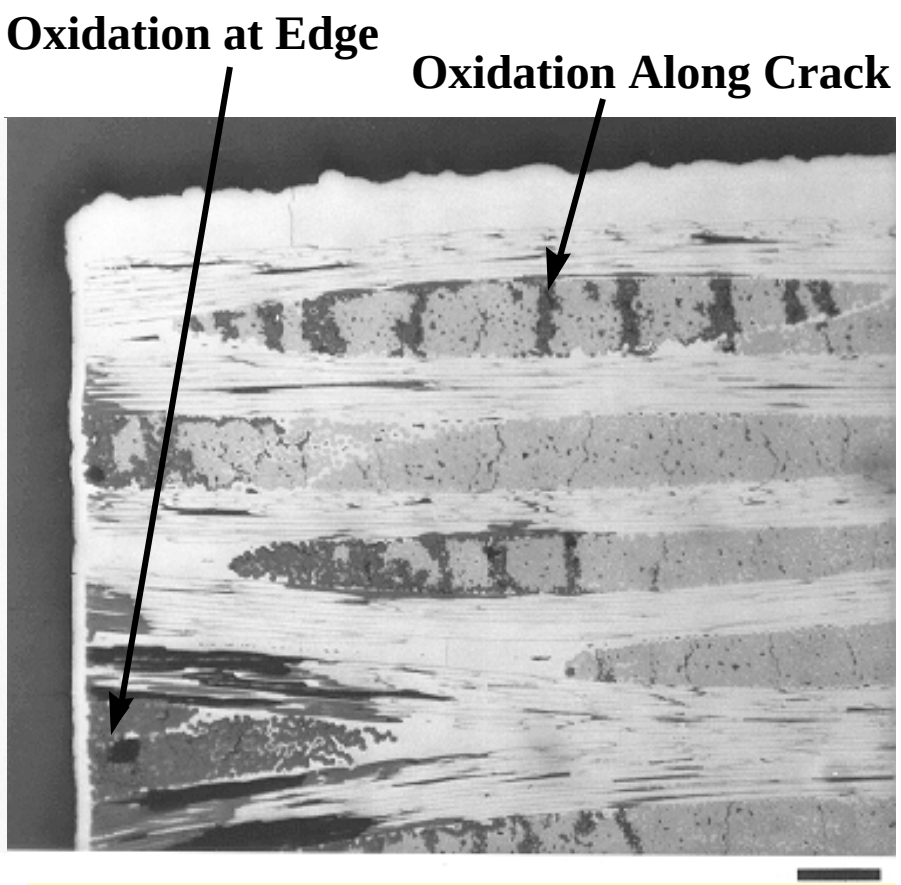


Justification for use of Sylramic-iBN SiC Fibers in High-T SiC/SiC CMCs: Reactive heat treatment forms a thin protective in-situ BN surface coating and provides the best high-temperature stress-rupture resistance of any SiC fiber (reported in 2001, H. M. Yun).

Cracks Act as Oxygen Diffusion Paths in C/SiC

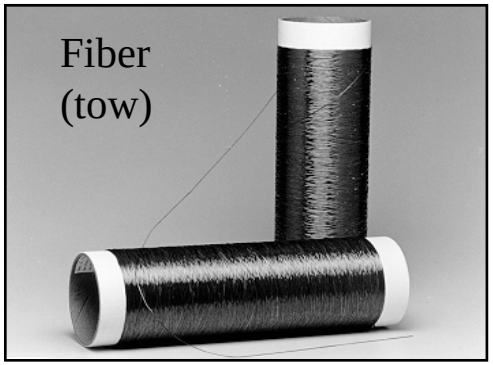


Fracture surface showing oxidation of crack bridging fibers at an elevated temperature in air.

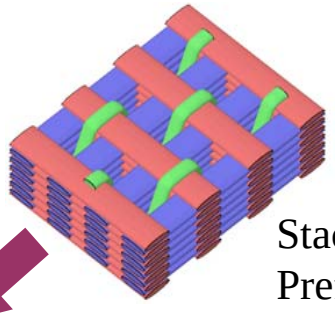


Polished cross-section showing oxidation of carbon fibers occurring preferentially along microcracks. 1000°C/25 ksi/air. (scale bar equals 0.1 mm) (Source: M. C. Halbig)

Chemical Vapor Infiltration (CVI) CMC Manufacturing Process

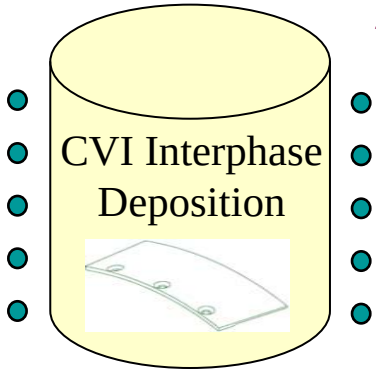


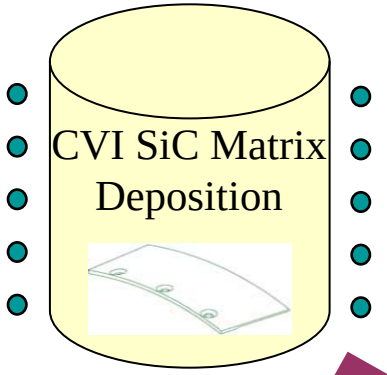
Weaving/Braiding



Stacked Fabric/
Preform

Tooling





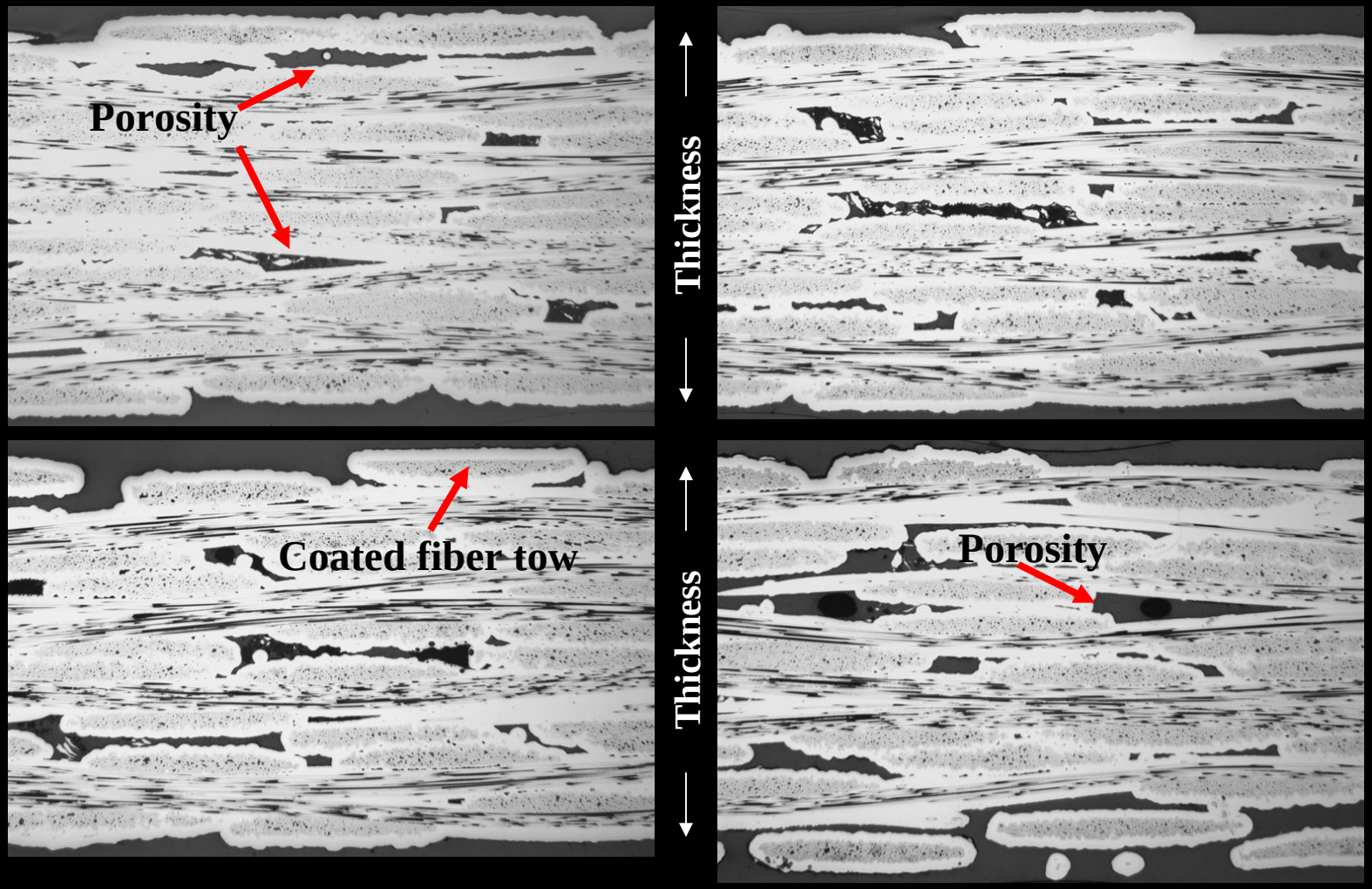
CVI C/SiC or SiC/SiC Composite

CMC properties result from the selection of constituents and the processing approach.

CMC Processing Variables Include:

- Fiber
- Fiber architecture
- Fiber loading (vol %)
- Fiber heat treatment
- Preform and CMC annealing
- Interphase (fiber coating)
- SiC matrix infiltration approach (CVI, melt infiltration (MI), polymer impregnation pyrolysis (PIP) or a hybrid approach)

Sylramic-iBN SiC Fiber-Reinforced CVI SiC CMC

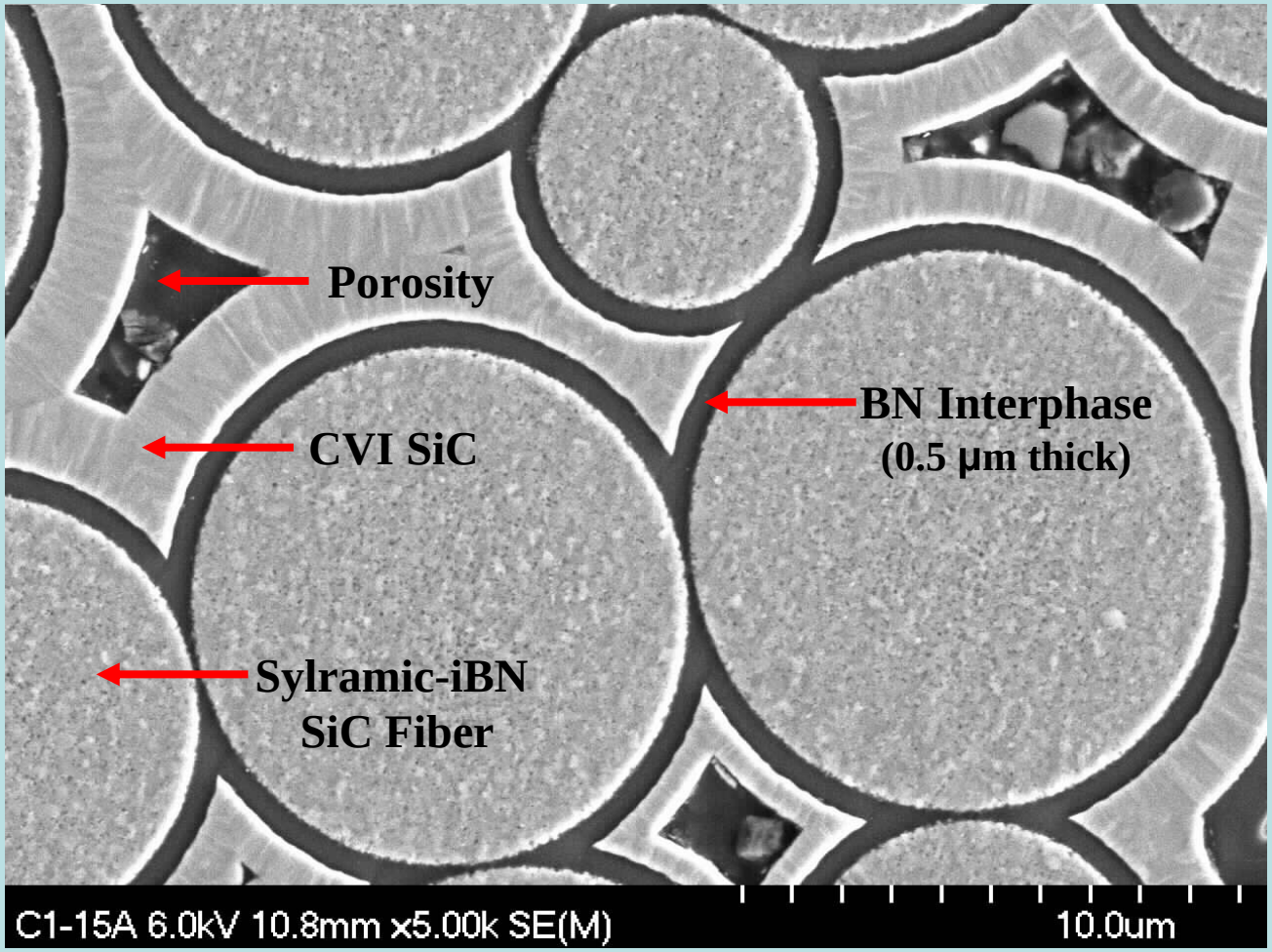


Average specimen thickness: 2.1 mm

Average specimen density ~ 2.6 g/cc

Variable distribution of porosity throughout specimens, which is typical for CVI CMCs

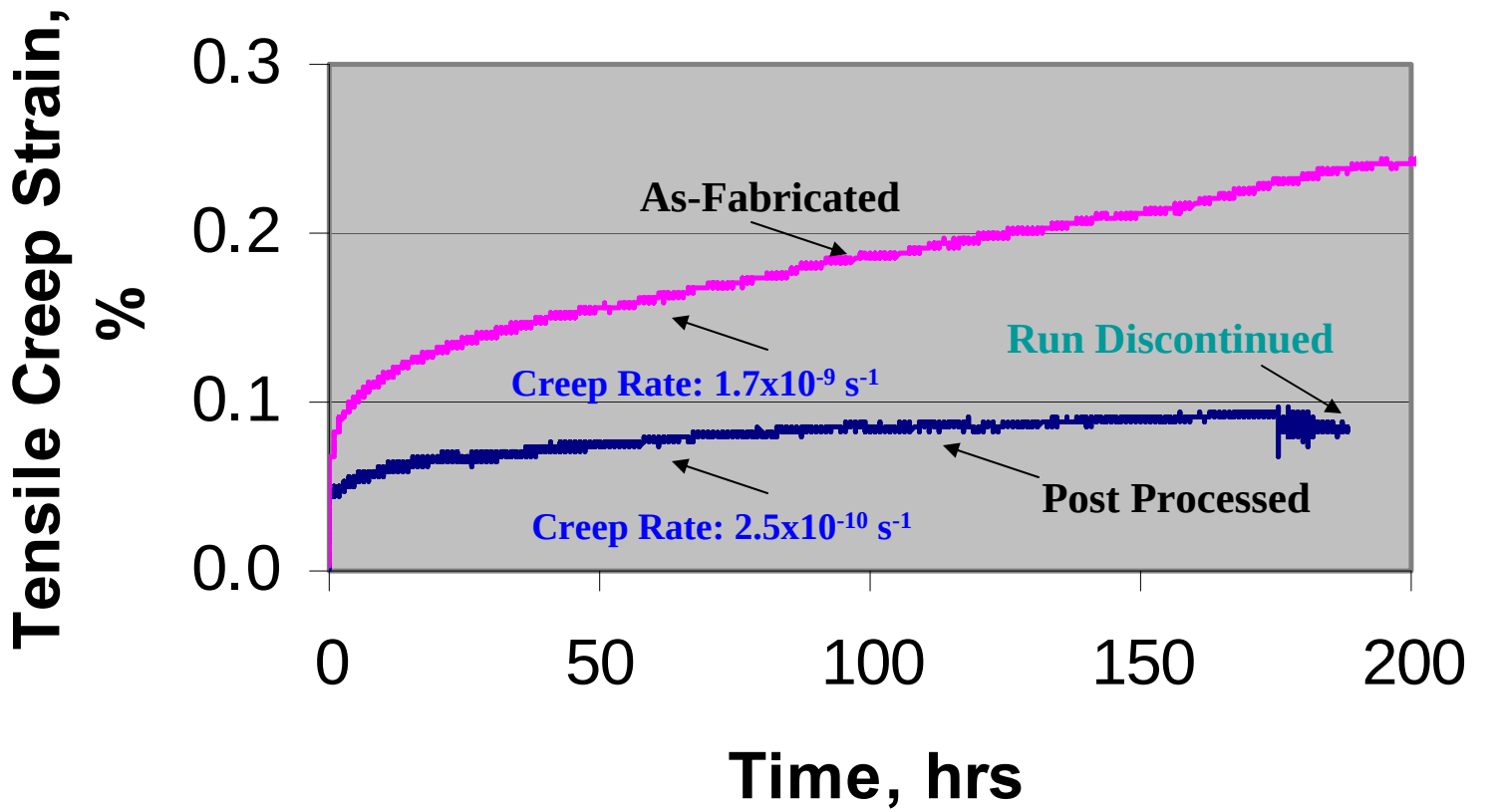
Sylramic-iBN SiC Fiber-Reinforced CVI SiC CMC



Center of specimen, center of fiber tow

Uniform BN interphase observed in center of specimen

Creep-Rupture of As-Received and Heat Treated CVI SiC/SiC in Air at 1450°C (2642°F), 69 MPa (10 ksi)



(Source: R. T. Bhatt)

Post processing method developed for improving creep resistance of CVI SiC/SiC CMCs. Residual strength at 1450°C of post processed sample was 243 MPa (35 ksi).

ARMD FA Program: Hypersonics Project: M&S
Durable Material Systems

Objectives

- ***Advance fundamental understanding*** **of key hypersonic materials.**
- ***Improve*** **the high temperature performance of** ***enabling*** **hypersonic** ***materials*****.**
- ***Develop*** **validated physics-based response** ***models*** **for reliable design.**

ARMD FA Program: Hypersonics Project: M&S
3000°F Composites*

Task Objective:
Develop, demonstrate, characterize, and model composite material systems capable of operation at temperatures of at least 3000°F under a tensile load of at least 10 ksi, for 100 hrs in an oxidizing environment**.

***** initial tensile creep testing environment will be air***

*** Activity POC: Doug Kiser (GRC, 216-433-3247)**

ARMD FA Program: Hypersonics Project: M&S
3000°F Composites*

Technical Challenge:
Extend performance capability >2700ºF through material processing modifications, varying constituents, and heat treatment cycles for enhanced creep resistance and thermal conductivity.

Collect Data (Establish Baseline Properties)
— Analysis and Understanding
— Model Behavior
— Improve Material and/or Structure (as needed)
— Document Results Throughout Process

*** Activity POC: Doug Kiser (GRC, 216-433-3247)**

SiC/SiC Composites Currently Being Evaluated (3000°F Composites)

Mat'l	Description	Fiber	Architecture	Interface	Matrix	Vendor	Notes
1	CVI SiC/SiC	Sylramic-iBN	2D, 20 epi 8 plies	BN (Si doped)	CVI SiC	GE	Sealed edges
2	Annealed CVI SiC/SiC	Sylramic-iBN	2D, 20 epi 8 plies	BN (Si doped)	CVI SiC	GE	Sealed edges, Annealed
3	PIP SiC/SiC	Sylramic-iBN	2D, 20 epi 10 plies	Mod1	PIP SiNC/SiC*	COIC	Sealed edges
4	PIP SiC/SiC	Sylramic-iBN	2D, 10 epi DBLT** 10 plies	Mod1	PIP SiNC/SiC*	COIC	Sealed edges

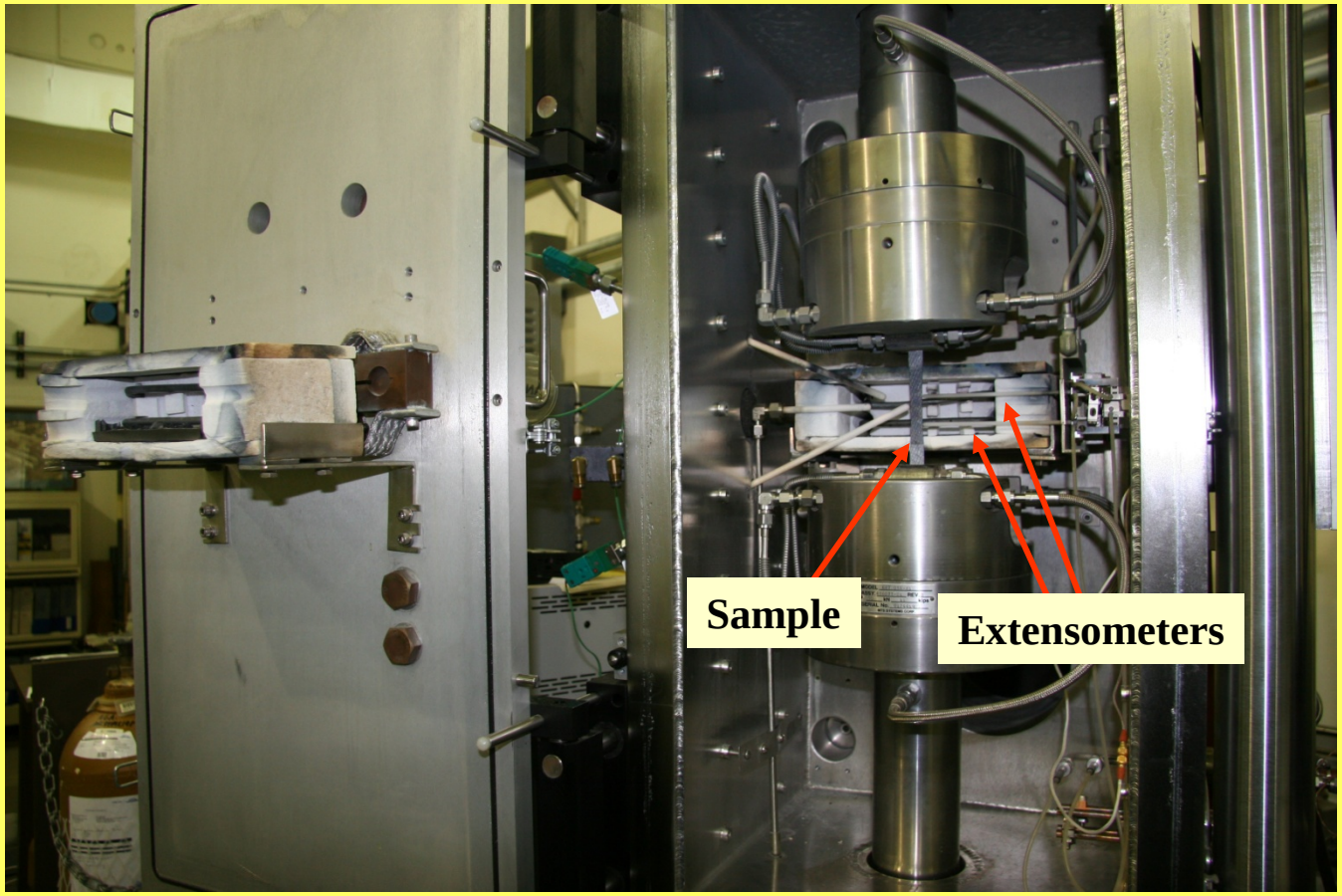
** Double Tow * with particulate filler

Characterization of Composite Materials

- Density, microstructure (before and after testing)
- NDE (with LaRC)
- RT fast fracture (FF) tensile testing with AE (acoustic emission)
- High temperature fast fracture tensile testing
- From FF: Modulus, proportional limit, ultimate tensile stress, ultimate strain
- Stress-rupture (creep) at high temperature (2640 - 3000°F) under various loads
- ILT, thermal conductivity, ILS

SiC/SiC Composites: Tensile Testing (3000°F Composites)

Tensile Testing Set-up Utilized for FF and Creep Rupture Testing



- **Furnace is open to show how extensometers (for monitoring strain) touch sample.**
- **SiC extensometer probe rods for high temperature creep rupture tests.**

SiC/SiC Composites: Fast Fracture Tensile Testing (3000°F Composites)

Fast Fracture Tensile Test Results: Ultimate Tensile Strength (UTS)

	CVI SiC/SiC*	PIP SiC/SiC
Temperature	Ave. UTS (ksi)	Ave. UTS (ksi)
Room Temperature	58.7	60.2
2642°F (1450°C)	41.4	42.5
2822°F (1550°C)	33.7	35.4

* Annealed

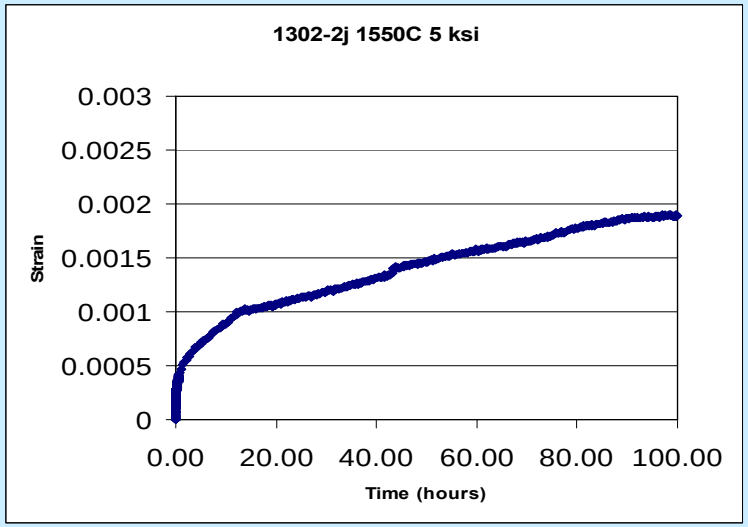
At each temperature:
UTS of CVI and PIP SiC/SiC are roughly equivalent (within 5%)

Creep Rupture of SiC/SiC Composites (3000°F Composites)

Creep (Stress-Oxidation) Rupture Testing Approach

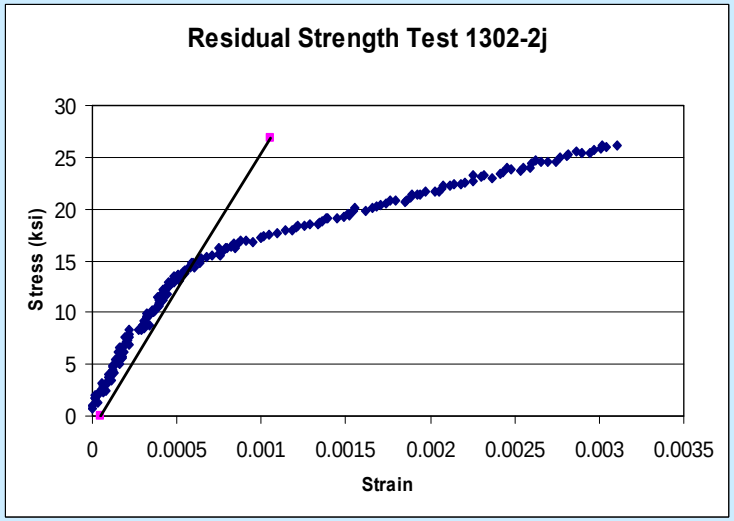
1) Heat to test temperature in air
2) 10 second ramp to hold stress
3) Hold for 100 hr run-out
4) Release load, but maintain hold temperature
5) Perform residual strength testing at elevated temperature

CVI SiC/SiC



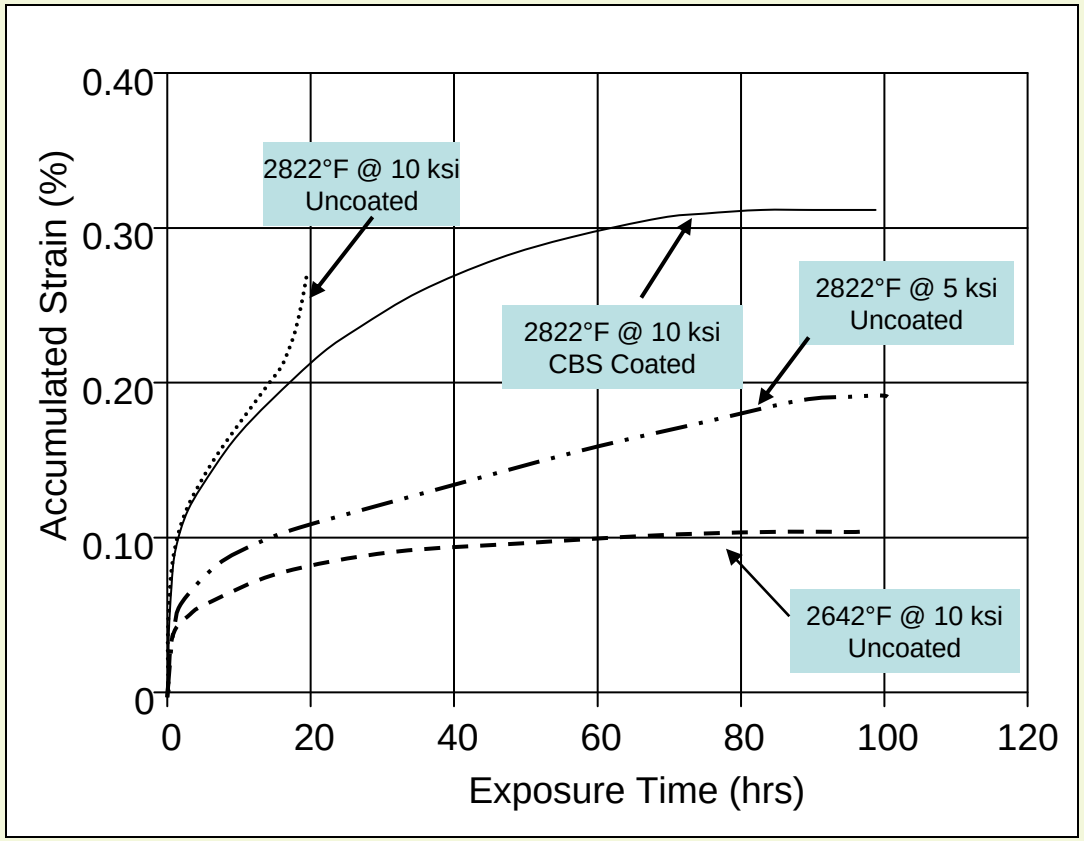
Creep curve showing deformation occurring during test at 2822°F

Following Run-out



Fast fracture (FF) stress-strain curve showing residual strength at 2822°F

Creep Rupture of CVI SiC/SiC Composites (3000°F Composites)



Results

Uncoated Samples

Three repeat samples survived 2642°F/10 ksi for 100 hr run-out (94% strength retention)

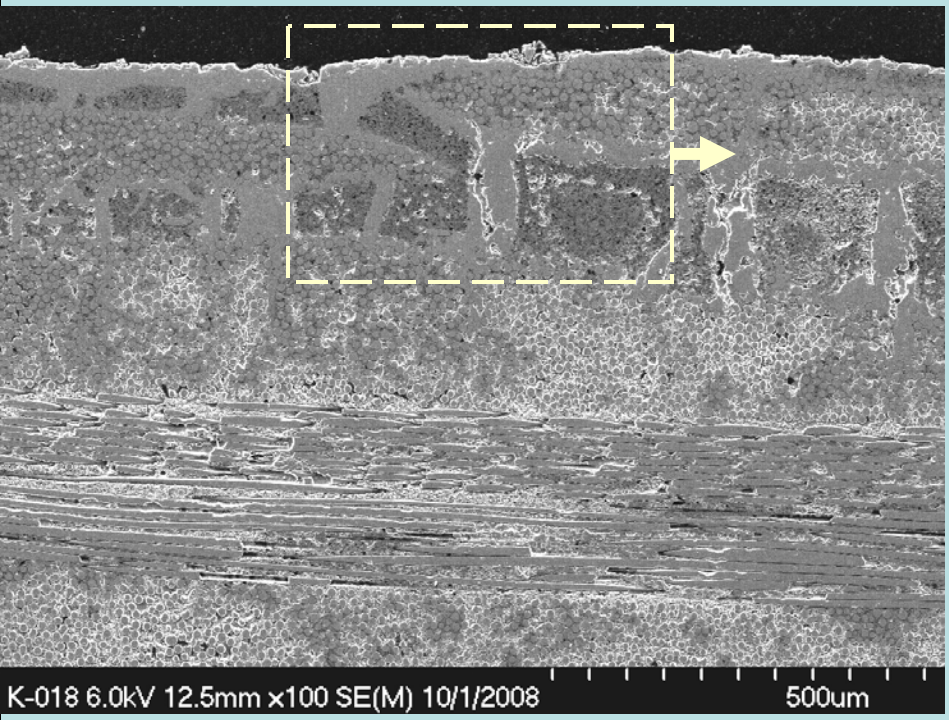
Three repeat samples survived 2822°F/5 ksi for 100 hr run-out (69% strength retention)

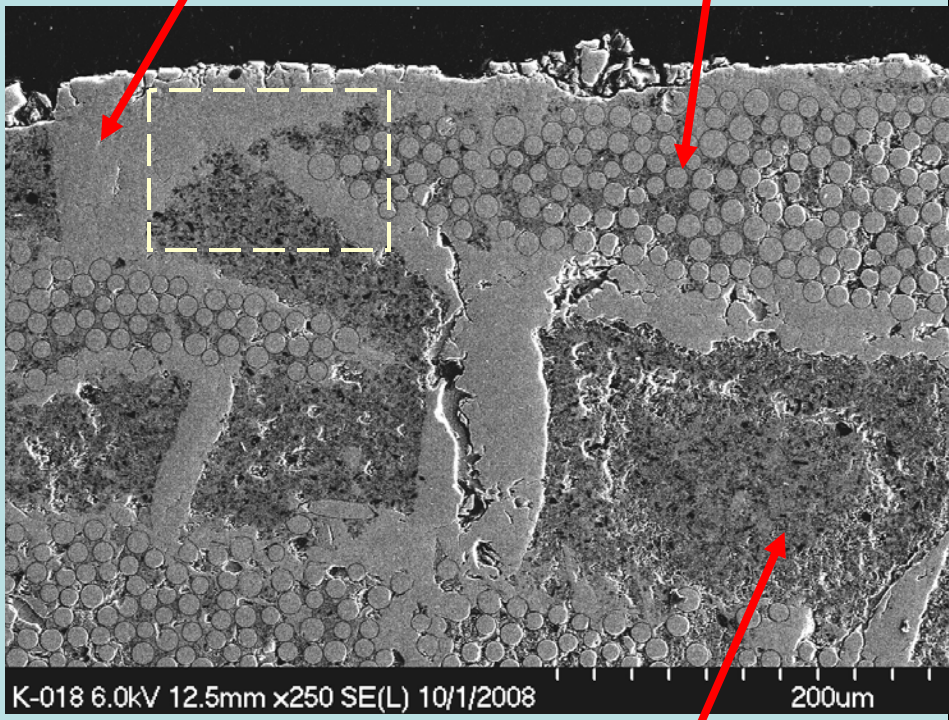
Three repeat samples failed at 2822°F/10 ksi before 100 hr run-out (ave. life: 15.5 hr)

CBS Coated Samples

Single specimen survived 2822°F/10 ksi for 100 hr runout (atypical). Two others failed at ave. life: 10 hrs.

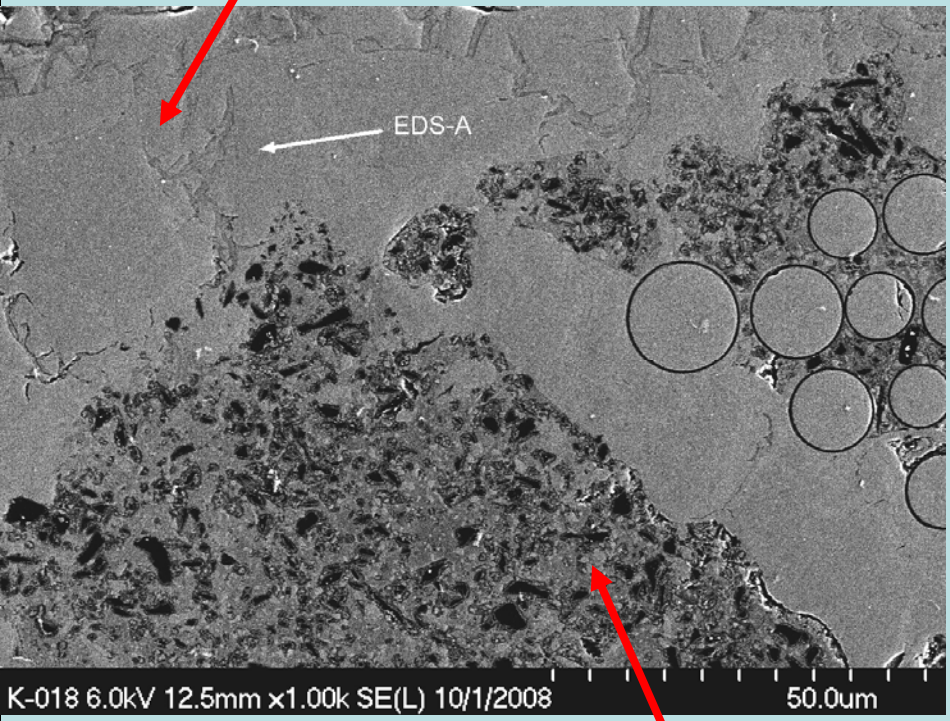
Sylramic-iBN SiC Fiber-Reinforced PIP SiC CMC





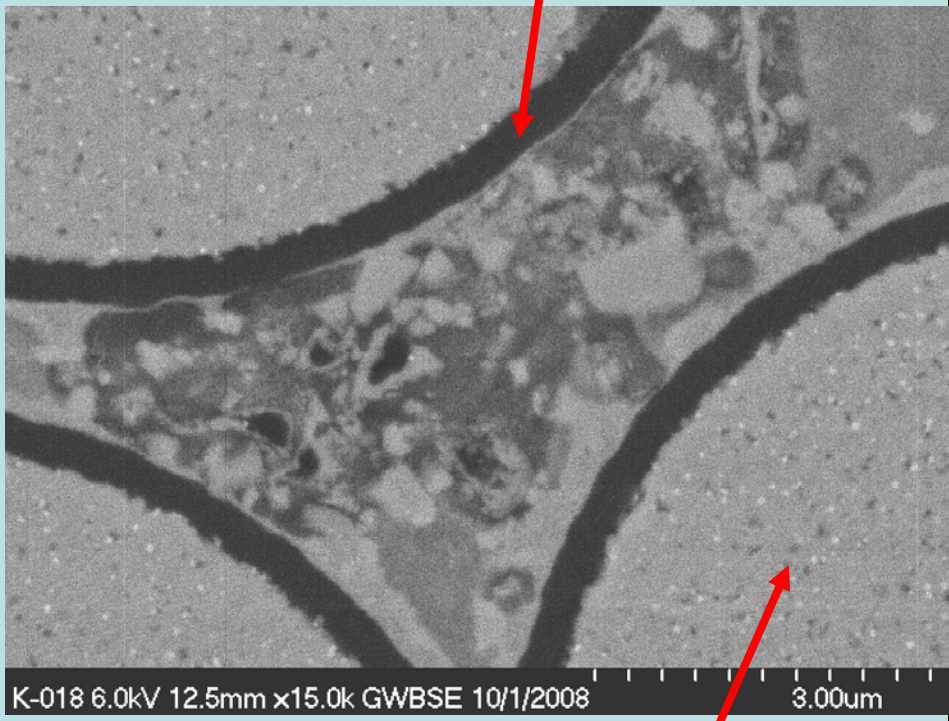
Sylramic-iBN SiC Fiber-Reinforced PIP SiC CMC

Matrix Infiltration Y



Matrix Infiltration X

BN Interphase (0.4 µm thick)



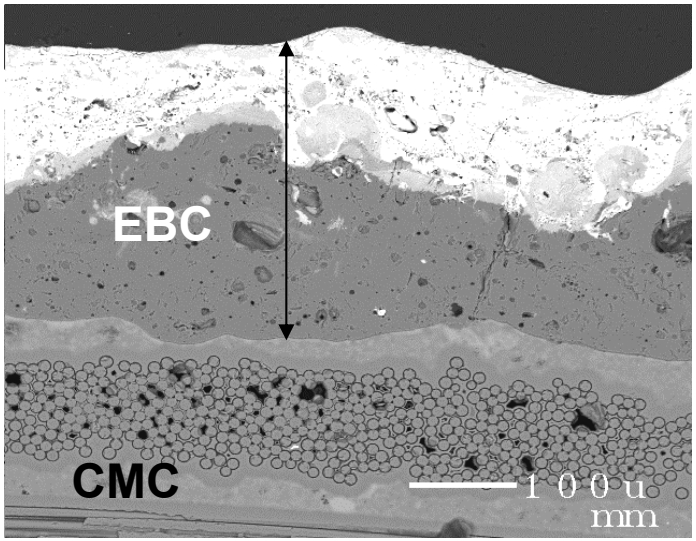
Sylramic-iBN SiC Fiber

2822°F /10 ksi: Average life of the PIP samples was 48.5 hrs

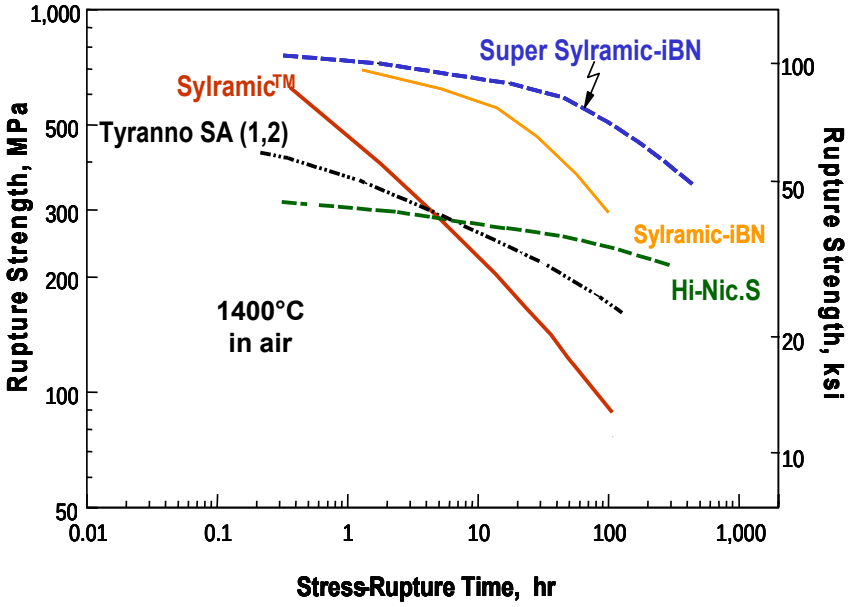
ARMD FA Program: Hypersonics Project: M&S (3000°F Composites)

Material/Test Specimen Modifications (FY'09):

- Investigate effect of SOA coatings on PIP and CVI SiC/SiC durability.
- Examine effect of utilizing Super Sylramic-iBN SiC fibers and an alternate interphase in PIP and CVI SiC/SiC CMCs.
- Test tensile specimens with broader gage widths to determine if this affects observed durability (in creep rupture tests).
- Evaluate Hybrid SiC/SiC (CVI followed by PIP) CMCs.



Example of environmental barrier coating



Joint NASA/AFRL Roadmap for SiC/SiC Composites

- NASA Management visited Air Force Research Laboratory (AFRL) to discuss collaboration opportunities.
- Several technology areas were identified for collaboration. One key area was development of CMC materials/components with temperature capability of 2700°F (or greater).
- Both organizations agreed to develop a joint roadmap in this technology area and to identify potential interdependencies.
- The initial NASA/AFRL focus is on SiC/SiC CMCs.

Summary

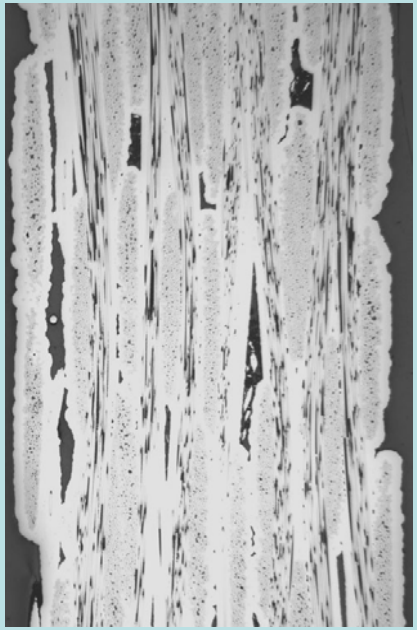
- Improving the durability of SiC/SiC CMCs at T>1400°C via alternate processing (fabrication and selection of constituents) approaches is being pursued in the NASA ARMD FA Program Hypersonics Project (M&S: 3000°F Composites Task).
- Thus far, "Full" CVI SiC/SiC samples have achieved 100 hrs life (run-out) in stress-rupture testing under the following conditions: 1450°C/69 MPa, and 1550°C/34 MPa. Good strength retention was observed.
- The influence of using tensile specimens with broader gage widths and specimens having various protective surface coatings (to prevent or reduce oxidation effects) and/or will be evaluated.
- A significant amount of high temperature SiC/SiC data will be generated during the next year. Material performance will be correlated with microstructure/constituents.
- NASA is developing a joint SiC/SiC Roadmap with AFRL.

Conclusion

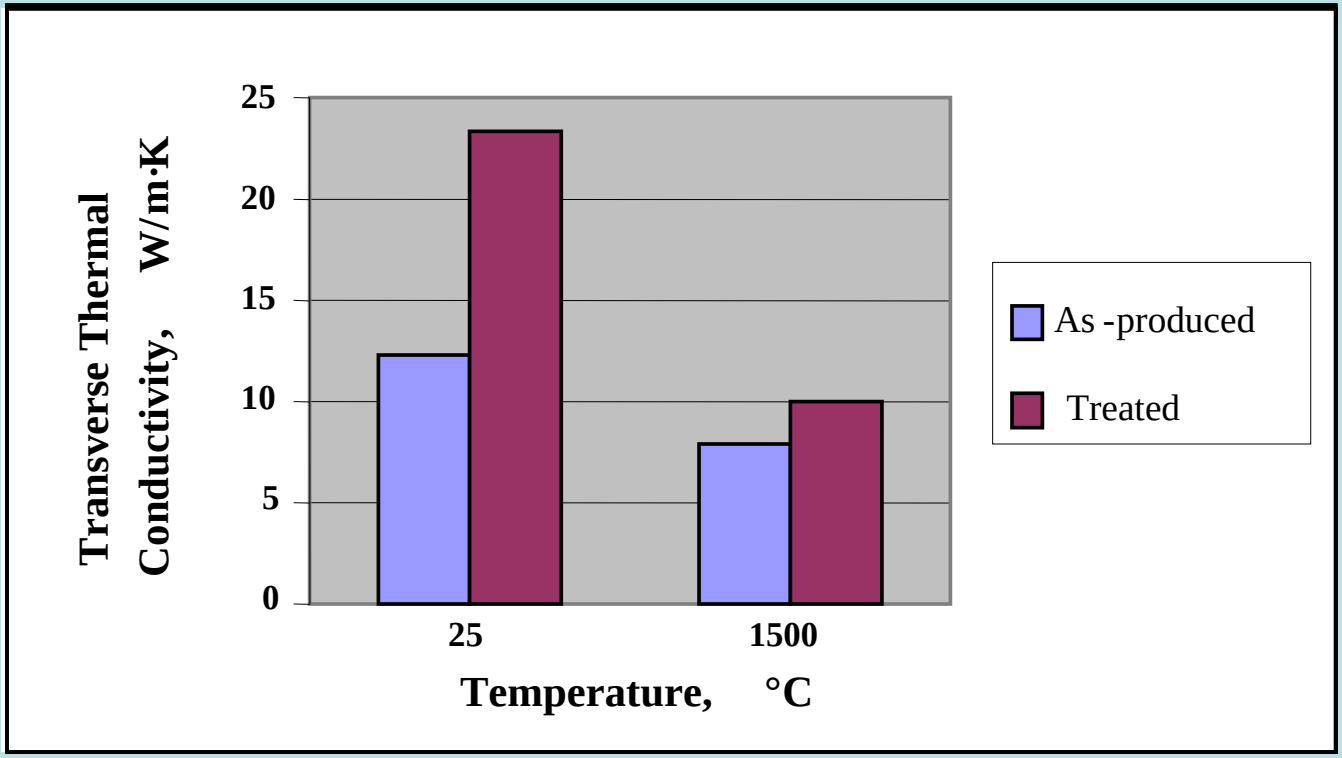
"……..for the dream of yesterday
is the hope of today
and the reality of tomorrow."
Dr. Robert H. Goddard
US physicist & pioneer rocket engineer (1882 - 1945)

- With continued development by NASA, DoD, and Industry, SiC/SiC CMC aerospace components can become a reality.

Thermal Conductivity of CVI SiC/SiC Composites



← Thickness →



Heat treatment of CVI SiC/SiC composites improves the thermal conductivity; ~ 90% and 25% improvement in 25 and 1500°C transverse (through-thickness) thermal conductivity, respectively.

(Source: R. T. Bhatt)

CMC Materials Selection Considerations

SiC/SiC Advantages wrt C/SiC:

- In contrast to C/SiC, matrix cracks and cracks within the fiber tows are not present in as-fabricated CVI and MI SiC/SiC CMCs.
- Near-stoichiometric SiC fibers are more isotropic in properties and significantly more oxidation resistant.
- BN-based interphases (fiber coatings) for SiC/SiC composites are more oxidatively stable than carbon interphases for C/SiC composites, but they can still be degraded by oxygen and water vapor.

CMC Materials Selection Considerations

SiC/SiC Disadvantages wrt C/SiC:

- Density of SiC/SiC is higher than that of C/SiC (2.6 g/cc vs. 2.1 g/cc).
- SiC fibers are more susceptible to creep (type of SiC fiber will determine the creep resistance). However, the use-temperature-limit of SiC fibers should depend on the stress state in the component (e.g., hot surface in compression, etc.).
- Fiber cost and availability.
- Ability to handle and weave stoichiometric SiC fiber imposes restrictions on fiber architecture.