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"Processing-Related Issues for the Design and Lifing of SiC/SiC Hot-Section Components", J. DiCarlo and R. Bhatt, NASA Glenn Research Center; G. Morscher, Ohio Aerospace Institute; and H.M.Yun, Matech/GSM, Inc.

For successful SiC/SiC engine components, numerous process steps related to the fiber, fiber architecture, interphase coating, and matrix need to be optimized. Under recent NASA-sponsored programs, it was determined that many of these steps in their initial approach were inadequate, resulting in less than optimum thermostructural and life properties for the as-fabricated components. This presentation will briefly review many of these process issues, the key composite properties they degrade, their underlying mechanisms, and current process remedies developed by NASA and others.

Processing-Related Issues for the Design and Lifing of SiC/SiC Hot-Section Components

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Objective

- Briefly discuss a variety of processing-related issues and their underlying mechanisms that have been observed by NASA to negatively impact key design and lifing properties of SiC/SiC composites and prototype hot-section components
- Indicate best current remedies from NASA and others for minimizing these process issues (Lessons Learned).

Outline

- Typical SiC/SiC component processing routes
- Key properties for component design and lifing
- Lists of issues, properties affected, current remedies for some current processes for:
  - SiC Fiber
  - SiC Fiber Preforming (architectural effects)
  - BN Interphase Coating
  - SiC-based Matrix Formation
- Select examples of best current remedies

Typical Processing Routes for SiC/SiC Components

- Select SiC **fiber** type in commercial tow form:

- Form 3D Component Shape by Preforming:
  - A. <u>2D Textile Route</u> : Weave or braid tows into 2D fabric or cylindrical plies, and lay up plies into 3D architectures

  - B. <u>2D Prepreg Route</u>: Form unidirectional plies from straight tows, and lay up plies into 3D architectures

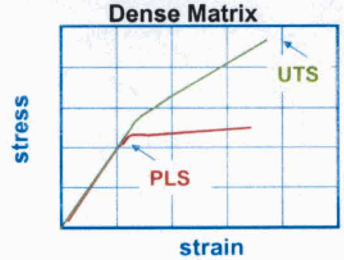
  - C. <u>3D Textile Route</u>: Weave or braid tows into 3D architectures with fibers thru-thickness for improved interlaminar properties

- Coat fibers in tow with thin BN-based **interphase** material using ***CVI*** (chemical vapor infiltration).
- Infiltrate 3D preform with various precursor **matrix** materials that convert to SiC-based ceramics using gases ***(CVI)*** <u>and/or</u> liquids such as polymers ***(PIP),*** slurries, and molten metals ***(MI)***

Key Design Properties for Thin-Walled SiC/SiC Components

Green = X or 0° Fiber Axis direction, Red = XY or 45/45 off-axis direction

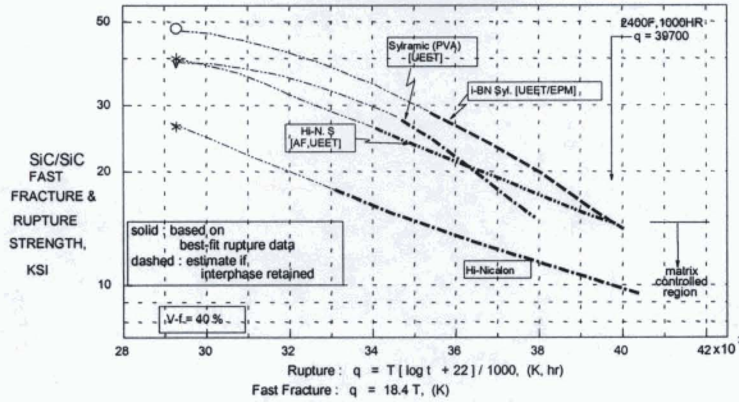
Dense Matrix



PLS ~upper design limit stress to prevent oxygen ingress

<u>Key Properties (3 directions)</u>	<u>Goals</u>
• **S** = *Component Shape*	- *<u>Near-Net</u> Shape*
• **Mv** = *Modulus Variability (tensile, shear)*	- *<u>Low</u> as Possible*
• **P** = *Proportional Limit Strength*	- *<u>High</u> as Possible*
• **U** = *Ultimate Tensile Strength*	- *<u>High</u> as Possible*
• **TC** = *Thermal Conductivity*	- *<u>High</u> as Possible*

Key Lifing Properties for Thin-Walled SiC/SiC Components



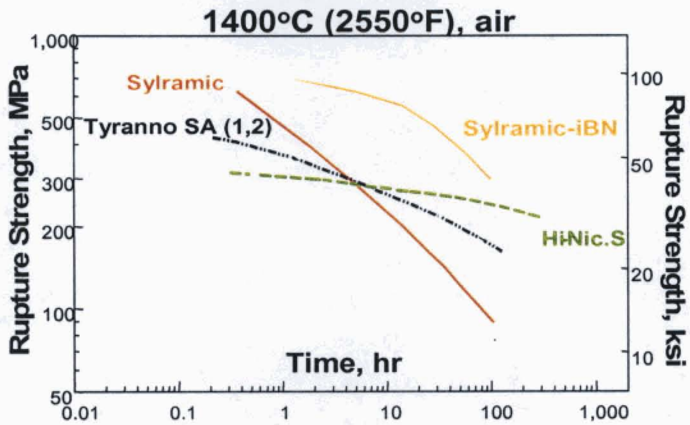
Key Properties (3 directions)	**Goals**
• **IS =** *Intrinsic Stability Temperature*	*- High as Possible*
• **LC =** *Life due to Creep*	*- Long as Possible*
• **LE =** *Life due to Environment*	*- Long as Possible*

SiC Fiber Process Issues for Key SiC/SiC Design and Lifing Properties

PROCESS ISSUE (SiC fiber example)	REDUCED CMC PROPERTIES U	TC	IS	LC	LE	CURRENT REMEDY (example)
Metallic impurities (Nippon Carbon fibers)			X			Non-metallic polymer lines (UBE fibers)
Oxide impurities (Nicalon, LOX M)			X	X		Remove oxides during process (Sylramic, Tyr SA)
Sintering aids-B, Al (Sylramic, Tyr SA)				X		Remove aid at high temp (Sylramic-iBN)
Grain size < 200 nm (Nicalon, LOX M)		X	X	X		Control process conditions for ~200 nm grains (Sylramic, Sylramic-iBN)
Grain size > 200 nm (Tyr SA)	X					
Carbon-rich surface (Nippon Carbon fibers)*					X	Oxidize carbon, but strength loss (Nippon Carbon, NASA)
Boron-rich surface (Sylramic)					X	Remove boron at high temp (Sylramic-iBN)

Sylramic-iBN SiC fiber solves most fiber issues:
No impurities or sintering aids, optimum grain size for tensile strength, creep resistance, conductivity, and improved durability surface

Rupture Resistance Advantage of NASA-Developed Sylramic-iBN SiC Fiber



As with monolithics, SiC-based fiber with the best rupture resistance also display highest creep resistance and thermal structural capability

SiC Fiber Preforming Issues for Key SiC/SiC Design and Lifing Properties

PROCESS ISSUE (preform example)	REDUCED CMC PROPERTIES S	Mv	P	Ux	Uz	LE	CURRENT REMEDY (examples from NASA studies with 2D-3D textile preforms and Sylramic-iBN fibers)
Excess fiber-fiber contact (2D-3D textile)		X	X			X	Tow spreading, Low epi, In-situ grown fiber coatings
Non-nesting of tows (2D textile)					X		Avoid non-nesting 2D-textile lay-ups, Use high compaction
Non-uniform tow distribution thru thickness (2D textile)		X	X		X		3D textile
Large tows 90° to stress (2D-3D textile)			X		X		Angle 2D woven to stress or use 2D, 3D braiding
Excess bend stresses (2D-3D textile)				X	X		Relieve stresses by annealing with no fiber strength loss
Low total fiber fraction (2D prepreg)			X	X		X	?
Ply ends thru thickness (2D prepreg, 2D woven)		X	X	X			Assure ply ends in lowest stress areas
Lack of sharp radii (2D prepreg, 2D-3D textile,)	X			X			Creep form 2D-3D textile preforms with no strength loss

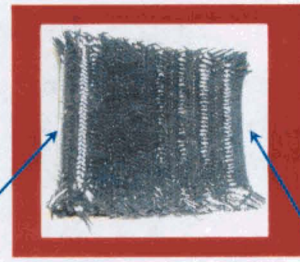
3D textile with creep forming and stress relaxation may be best

Advantages of Performing Sylramic-iBN Treatment On Complex-shaped Sylramic Preforms

2 in. diameter
2D-braided 3-ply
Sylramic tube

shape and
heat treat

Sylramic-iBN
vane pre-form



Leading Edge:
R = 300 mil

Trailing Edge:
R = 70 mil

For complex architectures, Sylramic-iBN treatment simultaneously offers net-shape creep-forming, stress relaxation, and Sylramic fiber conversion

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BN Interphase Process Issues for Key SiC/SiC Design and Lifing Properties

PROCESS ISSUE (example)	REDUCED CMC PROPERTIES					CURRENT REMEDY (example)
	Mv	P	U	TC	LE	
Carbon char from fiber sizing (PEO sizing, high wt. % PVA)	X				X	Low wt. % PVA sizing (Sylramic, Sylramic-iBN)
Oxygen impurities in BN (standard CVI BN)			X			Avoid carbon, boron on fiber (Sylramic-iBN fiber)
Low-temp deposition causes BN contraction at hi temp (standard CVI BN)	X	X		X	X	Process BN-coated preform, Hi-temp BN deposition (NASA, Synterials)
BN attacked by moisture (standard CVI BN)					X	Dope with silicon, Overcoat with Si_3N_4 (GE prepreg, Hypertherm, Synterials)

Silicon-doped BN currently appears to be best composition, but additional processing may be needed to stabilize for higher temperature applications

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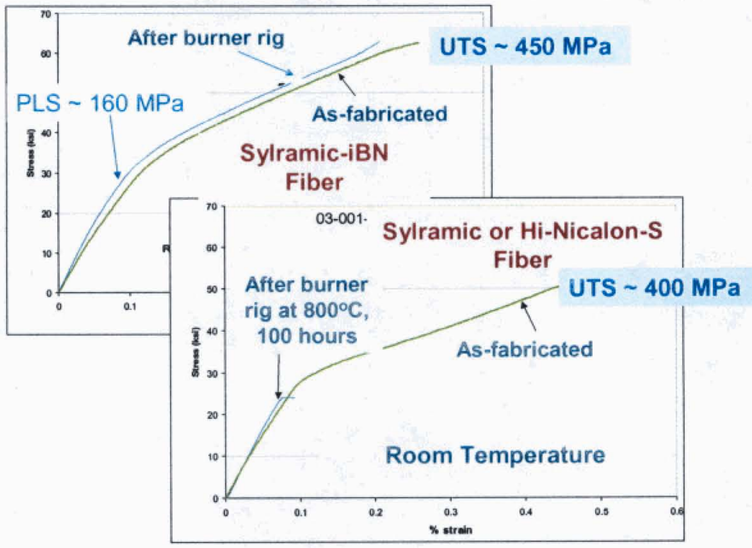
(CVI-MI) 2D SIC/SIC Panels with Sylramic-iBN Fibers Show Improved In-Plane UTS and Burner Rig Durability



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SiC Matrix Process Issues for Key SiC/SiC Design and Lifing Properties

PROCESS ISSUE (example)	REDUCED CMC PROPERTIES S	MV	P	U	TC	IS	LC	CURRENT REMEDY (source)
Closed porosity > 10 vol. % (CVI, PIP)		X	X		X			Melt Infiltration (GE, Goodrich)
Low-temp formation (CVI, PIP)					X	X		Anneal matrix-filled preform (NASA, COIC)
Free silicon in matrix and interphase/fiber attack during process/service (CVI, MI)				X	X	X	X	•Anneal CVI matrix-filled preform (NASA) •Si-resistant interphase over-coatings (GE prepreg)
One-sided matrix infiltration thru part thickness (CVI. MI, PIP)		X	X		X	X		Thin parts, open 3D preforms (NASA)
O_2 internal generation and interphase/fiber attack during process (PIP)				X				Employ getters in matrix (COIC)
Non-smooth surface finish (CVI, slurry cast MI)	X							Polymer precursors and smooth surface tooling (COIC, GE prepreg)

CVI + MI matrices for high temperature applications, but CVI + PIP with reduced porosity for highest temperature applications

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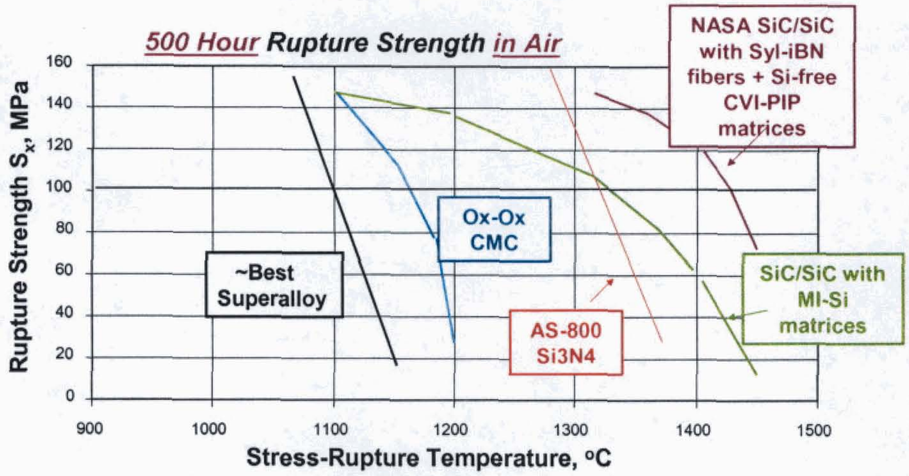
In-Plane Rupture Strength S_x of Current 2D and 3D CMC Systems



Time-Temperature Structural Capability for Si-free CVI-PIP SiC/SiC systems are currently the highest of any CMC

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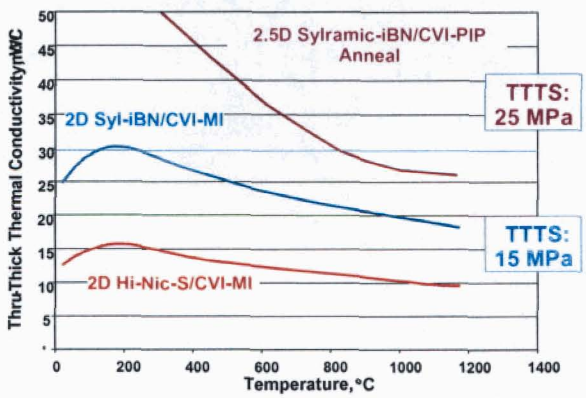
NASA is Continuing to Develop Improved SiC/SiC Using Alternate Fiber Architectures and Sylramic-iBN Fibers



2.5D angle-interlock architecture with low content of Sylramic-iBN fibers in z-direction (~3 vol. %) and NASA special anneal yields stoichiometric CVI-PIP SiC/SiC system with significantly improved thru-thickness conductivity and thru-thickness tensile strength (TTTS) at 20°C

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Summary

- By an understanding of process-structure-property relationships for the constituents, NASA and others has been able to develop advanced constituent and composite processes for high-temperature SiC/SiC systems with improved structural properties, thermal conductivity, and life under oxidative conditions.
- Key NASA approaches have been to
  - Select commercial materials and processes for the SiC fiber and matrix that result in minimal flaw sizes during processing and high-temperature service
  - Develop thermal treatments that stabilize and purify the constituent microstructures by removing unstable and creep-prone second phase elements such as boron, silicon, and oxygen.
  - Transfer advanced technologies to industry

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Conclusions and Future Needs

- **NASA's performance goals have been aimed primarily at achieving SiC/SiC composites and hot-section components with the highest temperature and structural capability in order to insure longest life and best durability at lower temperatures.**
- **Currently the SiC/SiC constituent materials and processes that provide the best design and lifing properties are:**
  - ***Stoichiometric Sylramic-iBN SiC fiber***
  - ***CVI BN-based fiber coating after NASA special treatment***
  - ***Stoichiometric SiC matrix with partial CVI plus PIP SiC, again after NASA special treatment.***
  - ***3D Fiber Architectures with non-orthogonal high-strength high-conductivity fibers, such as Sylramic-iBN.***
- **Key future processing goals at NASA are to**
  - **Increase creep-rupture resistance of Sylramic-iBN fiber**
  - **Reduce porosity in the CVI + PIP matrix**
  - **Develop 3D preforming approaches that meet both the complex shape and structural requirements of airfoils.**

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