ADVANCES IN SiC/SiC COMPOSITES FOR AEROSPACE APPLICATIONS

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In recent years, supported by a variety of materials development programs, NASA Glenn Research Center has significantly increased the thermostructural capability of SiC/SiC composite materials for high-temperature aerospace applications. These state-of-the-art advances have occurred in every key constituent of the composite: fiber, fiber coating, matrix, and environmental barrier coating, as well as processes for forming the fiber architectures needed for complex-shaped components such as turbine vanes for gas turbine engines. This presentation will briefly elaborate on the nature of these advances in terms of performance data and underlying mechanisms. Based on a list of first-order property goals for typical high-temperature applications, key data from a variety of laboratory tests are presented which demonstrate that the NASA-developed constituent materials and processes do indeed result in SiC/SiC systems with the desired thermal and structural capabilities. Remaining process and microstructural issues for further property enhancement are discussed, as well as on-going approaches at NASA to solve these issues. NASA efforts to develop physics-based property models that can be used not only for component design and life modeling, but also for constituent material and process improvement will also be discussed.
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Advanced Lightweight High-Temperature Structural Materials are Needed for Multiple Aerospace Applications

- Turbine Shrouds
- Turbine Vanes and Blades
- Nozzle Flaps and Seals
- Combustors
- Cooled Combustor Panels, Thrusters, Rocket Nozzles
- Control Surfaces
- Nose Caps, Leading Edges

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Typical Function of a Structural Material In a Hot Aerospace Application

Key Material Property Requirements:
- Low Density, Low Permeability, High Emissivity
- Microstructural Stability at Maximum Temperature
- High Tensile Stress Capability In-Plane and Thru-Thickness
- High Thermal Conductivity Thru-Thickness to reduce thermal gradients and stresses

Advantages of SiC Fiber / SiC Matrix (SiC/SiC) Ceramic Matrix Composites (CMC)

versus Superalloys:
- Lower density (~30% metal density)
- Higher temperature capability (>1100°C)
- Lower thermal expansion

versus Monolithic Ceramics:
- Non-catastrophic failure
- Higher toughness, better damage tolerance
- Capability for larger and more complex shapes

versus Carbon Fiber Composites (C/SiC, C/C):
- Higher oxidative durability, more predictable life
- Lower permeability

versus Oxide/Oxide Ceramic Composites:
- Higher strength, temperature capability, creep-rupture resistance, thermal conductivity, emissivity
- Lower permeability
Objective / Outline

- Present brief review of recent NASA efforts aimed at developing *Advanced Constituent Materials and Processes* for SiC/SiC composites to better meet the thermal, structural, and conductivity requirements for aerospace components.

  - NASA developments to 2002:
    - *Sylramic*-iBN SiC Fibers
    - SiC Matrices based on Silicon melt infiltration

  - More recent developments:
    - Silicon free Matrices
    - Special Post-production Treatment
    - Advanced Fiber Architectures

Acknowledgements

**SiC/SiC Development Team at NASA Glenn**

- **Fibers/Architectures:**
  - *Hee Mann Yun*
  (now at MATECH/GSM)
- **Matrices:** *Ram Bhatt*
- **Modeling:** *Greg Morscher*
  *Jerry Lang*

**NASA Funding Programs**

- *Ultra Efficient Engine Technologies (UEET)*
- Internal Research and Development
NASA 2002 Fabrication Route for SiC/SiC Components

- Sylramic Fiber
- Weaving, Braiding into 2D or 3D preforms
- Low Temp. CVI Si-BN Interphase Infiltration
- Reactor
- Slurry Cast SiC Matrix
- CVI Preform
- CVI-SiC Matrix Infiltration
- Reactor
- Sylramic-iBN fibers
- NASA Treatment to form Sylramic-iBN fibers
- Furnace
- Silicon Melt Infiltration
- CVI-MI SiC/SiC

NASA Advanced Process for Sylramic SiC Fibers

- In ~2000, NASA developed a high-temperature process for commercial Sylramic SiC fibers to produce the stoichiometric Sylramic-iBN fiber with a thermally stable in-situ grown BN (iBN) coating and state-of-the-art tensile strength and creep-rupture resistance.

[Graphs showing creep strain and rupture strength]
**Durability Benefit of In-Situ BN Coating**

**Without NASA treatment,** almost every Sylramic SiC fiber in each tow contacts another fiber, allowing rapid and detrimental fiber bonding upon oxygen ingress.

**With NASA treatment, in-situ BN coating prevents** SiC-SiC contact between Sylramic-iBN fibers, delaying oxide bonding and increasing SiC/SiC durability and reproducibility.

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**CVI-MI SiC/SiC Panels with Sylramic-iBN Fibers Show Improved In-Plane UTS and Burner Rig Durability**

- **As-fabricated**
  - Syrlamic-iBN Fiber: UTS ~ 450 MPa
  - Syrlamic or Hi-Nicalon-S Fiber: UTS ~ 400 MPa
- **After burner rig at 800°C, 100 hours**
  - Syrlamic-iBN Fiber: UTS ~ 450 MPa
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In-Plane 500-Hour Rupture Strength in Air for NASA CVI-MI SiC/SiC versus Competing Materials (~Yr 2002)

With upper use temperatures near ~1315°C, NASA 2002 SiC/SiC systems display higher thermostructural capability than competing materials.

CVI-MI SiC/SiC Properties Needed Further Improvement

- On scientific side, temperature capability, thermal conductivity, and design strength were less than the best SiC monolithic ceramics.
- On technical side, lifing risks still exist for SiC/SiC component failure due to unexpected hot spots and/or loss of the thermal barrier coatings.

NASA Activities:
- Understand, mechanistically model, and minimize sources for property limitations.
- Key Concerns: Improving SiC/SiC
  - Microstructural Stability at higher temperatures
  - Structural Capability at higher temperatures
  - Thru-Thickness thermal conductivity
  - Thru-Thickness strength (ILTS or interlaminar tensile strength for 2D composites)
National Aeronautics and Space Administration

Stability Issues for CVI-MI SiC/SiC

Key Issues:
- For long times above 1300°C, free Si in matrix diffuses thru grain boundaries of initial CVI SiC matrix coating, attacks BN fiber coating and SiC fiber, and reduces UTS.
- Free Si in matrix also degrades composite creep-rupture resistance, reducing CVI-MI SiC/SiC structural capability at high temperatures.
- CVI BN fiber coating, which is deposited below 1000°C, densifies and shrinks at high temperatures, reducing fiber-matrix load transfer, thru-thickness conductivity and tensile strength.

NASA Approaches to Microstructural Stability Issues

- Retain Sylramic-iBN SiC fiber because of its high strength, high creep-rupture resistance, high thermal conductivity, and microstructural stability to over 1650°C.
- Develop materials and processes for two advanced SiC/SiC systems with Si-free matrices:
  - Full CVI SiC
  - Partial CVI SiC + SiC derived from polymer infiltration and pyrolysis (hybrid CVI-PIP).
- Develop special annealing treatment that
  - Stabilizes BN coating, minimizing gap formation between fiber and matrix, and also
  - Removes process-related defects in the SiC matrix to improve CMC creep-rupture resistance and thermal conductivity.
### Annealing Effects on Partial CVI SiC Panels with Different Fibers

**Graph:**
- CVI SiC ~ 20 vol.%
- Sylramic-IBN
- Hi-Nicalon
- Hi-Nicalon Type S

**Legend:**
- 100-hr Exposure Temperature, °C
- Ultimate Tensile Strength, MPa

**Intrinsic stability and in-situ grown BN coating for Sylramic-IBN fiber allows annealing of Si-free CVI SiC matrices to >1500°C with NO loss in in-plane UTS**

### Creep-Rupture Behavior for NASA 2D SiC/SiC Systems

**Graph:**
- 1450°C / 10 ksi / AIR
- CVI-MI matrix
  - (with silicon)
- Annealed Full CVI or CVI-PIP matrix
  - (without silicon)

**Legend:**
- Si-free matrices with NASA special annealing treatment significantly improves creep rupture resistance of SiC/SIC
Effects of NASA Special Annealing Treatment on Thru-Thickness Properties for 2D Sylramic-iBN/SiC Systems

Unannealed  Annealed Special

Closed Porosity:
~15%  ~14%  ~5%

Improves
SiC matrix

Improves
BN fiber coating

In-Plane 500-Hour Rupture Strength in Air for NASA Advanced SiC/SiC versus Competing Materials (~Yr 2005)

~Max in-plane stress for SiC/SiC combustors and vanes
~Max SiC/SiC Design Strength
~Best Superalloy

Advanced SiC/SiC with Syl-iBN fibers + Si-free matrices
NASA SiC/SiC with Mi-Si matrices

Thermostructural capability for advanced SiC/SiC system is state-of-the-art with upper use temperatures of ~1450°C (2640°F)
NASA Continues 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 thru-thickness conductivity and 20°C thru-thickness tensile strength significantly improved over original CVI-MI system.

Summary and Future Needs

- NASA SiC/SiC systems with high performance Sylramic-iBN fibers are capable of outperforming the best superalloys, Ox/Ox systems, and monolithic ceramics in in-plane strength capability at high temperatures (to 1450°C), while providing low density, high emissivity, high thermal conductivity, and high thru-thickness strength.

- Currently the SiC/SiC constituent materials and processes that provide the best combination of thermal and structural performance 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. (Future improvements are needed to reduce porosity in this matrix).

- Although 2D SiC/SiC systems offer simplicity in fabrication of aerospace components, 3D systems reinforced by non-orthogonal high-strength high-conductivity fibers, such as Sylramic-iBN, offer even higher thru-thickness conductivity and strength. (Future improvements are needed to increase Z-direction fiber content.)
Current SiC/SiC Commercialization Issues

Life-cycle cost-benefit analyses need to be conducted for each SiC/SiC component to determine economic viability, BUT

- High costs still exist for high-performance SiC fibers, for quality controls at every process step, and for generation of accurate design data bases
- Constituent vendors are often different and single organizations, complicating production time and resulting in multi-tiers of profit taking,
- Little or no experience exists demonstrating reliable performance under actual component service conditions
- SiC/SiC property and lifing models are complex, and approaches for converting models into Finite Element codes for component design are lacking.

Even at design stresses below matrix cracking, lifing analyses of SiC/SiC components at high temperatures can be very complex:
- Creep effects, residual stress development
- Environmental effects with and without barrier coatings.