High Temperature Lightweight Self-Healing Ceramic Composites for Aircraft Engine Applications

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Introduction

- Advanced aircraft engines require the use of reliable, lightweight, creep-resistant and environmentally durable materials.

- Silicon carbide-based ceramic matrix composite (CMC) technology is being developed to replace nickel-based superalloy blades and vanes.
  - Near term 1589 K (2400 °F) (cooled).
  - Medium term 1755 K (2700 °F) (cooled).
  - Long term 1922 K (3000 °F) (uncooled).
Rule of Mixtures (ROM) Composite Theory

\[ P = P_{fiber}V_{fiber} + P_{matrix}V_{matrix} \]

\[ V_{fiber} + V_{matrix} = 1 \]

\[ P_i = \text{Property of the } i^{th} \text{ component (e.g. strength).} \]

\[ V_i = \text{Volume fraction of the } i^{th} \text{ component}. \]

- Properties of the composite are determined by the properties of the fiber and the matrix and their relative volume fractions.
Objectives

- Develop a new class of ceramic composites - **Engineered Matrix Ceramics (EMCs).**
  - Design different engineered matrices.
  - Demonstrate thermal strain compatibility with SiC.
  - Evaluate oxidation and mechanical properties.
- Fabricate engineered matrix composites.
  - Evaluate self-healing properties.
Current SiC/SiC CMC Fabrication Processes

(courtesy R. Bhatt)

SiC Fiber

0/90 Fabric Weaving

Preform

CVI SiC Matrix Infiltration

CVI BN Interface Infiltration

Reactor

Preform Compression

Slurry Infiltration

Si Melt Infiltration

Full CVI CMC

CVI-MI CMC

PIP or CVI-PIP CMC

CVI SiC Preform

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Current Generation of CMCs: Matrix Microstructure

- Silicon carbide (SiC).
- Unreacted or free carbon and silicon.
- Porosity:
  - \(~10\text{-}25\text{ vol.\%}\) for chemical vapor infiltration (CVI).
  - \(~10\text{-}25\text{ vol.\%}\) for polymer infiltration and pyrolysis (PIP).
  - \(~3\text{-}10\text{ vol.\%}\) for Melt Infiltration (MI).
Typical Microstructure of As-Processed BN-Coated Hi-Nicalon MI SiC Composites

Density ~ 96-97 %

(Courtesy M. Singh)
Current SiC/SiC CMC Matrix Capabilities

- Brittle at all temperatures.
- No crack tip blunting - fast crack propagation.
- No self-healing.
- Oxygen ingress to fibers shortens fiber life.
- Free Si in the matrix limits temperature usage to below 1588 K (2400 °F).

- Low matrix cracking strength (proportional limit - 69 MPa/10 ksi)
- Matrix fills space and provides a thermally conductive path.
- Fracture toughness due to crack bridging and interface debonding.
Recession of BN and Formation of Glassy Phase in BN-Coated Hi-Nicalon MI SiC Composites

(T = 973 K; \( \sigma = 250 \) MPa; 1000 h in air)

\[
2\text{BN (s)} + \frac{3}{2} \text{O}_2 (g) = \text{B}_2\text{O}_3 (l) + \text{N}_2 (g)
\]

\( \text{B}_2\text{O}_3 - \text{SiO}_2: \) Low eutectic temperature of 372 \(^\circ\)C

(Courtesy M. Singh)
Important Question

Can the matrix properties be suitably engineered to ensure certain desirable characteristics?
Present Concept

![Graph showing the relationship between ΔL/L₀ (%), temperature (T, K), and material types (SiC, Silicide, Nitride, Engineered matrix (ROM)).]
Crack Tip Blunting and Self-Healing

SiC fibers

Crack blunting due to matrix plasticity slows crack growth

SiC fibers

Self-healing of fine cracks minimizes oxygen ingress to fibers

Increased reliability and load carrying capacity
Innovation: Desired Characteristics of the Engineered Matrix (EM)

- Thermal strain compatibility of the matrix with the SiC fibers.

- Plastically compliant matrix to blunt cracks.

- Self-healing crack capabilities to minimize ingress of oxygen.

- Minimize the volume fraction of unreacted silicon to prevent corrosive attack of fibers and incipient melting.

- Dense matrix to increase thermal conductivity.
Expected Impact of Innovation

- **Matrix plasticity** - increased reliability, compliant matrix.

- **Self-healing matrix** - prevents or minimizes oxygen ingress.

- **Low free Si** - reduces fiber attack, reduces incipient melting, increased high temperature capability.

- **Dense matrix** - High thermal conductivity.
Fabrication and Testing of Engineered Matrix Composites (EMC)

2D coated preform impregnation with engineered matrix

Engineered matrix preparation
- Powder composition
- Attrition milling

Preform impregnation
- Slurry preparation
- Slurry casting
- Iteration and optimization

Engineered matrix preparation
- Powder composition
- Ball milling

Monolithic matrix specimens
- Optimization of hot-pressing conditions
- Hot-pressing & machining of specimens

Composite preparation and testing

Melt infiltration of impregnated preforms
- Process optimization

Matrix characterization
- CTE measurements
- Oxidation ("pest"; thermal stability)
- Microstructures
- Bend tests

Composite characterization
- Microstructures
- Thermal cycling
- Bend & tensile tests

Interim and final reports
Hot-Pressed Plate and Optical Micrograph

CrMoSi-EM

50 x 50 x 4 mm

Optical micrograph
Proof-of-Concept: Thermal Strains

\[ \Delta L / L_0 (\%) \]

\[ T (K) \]

- CrSi2 (Run 582)
- CrSi2 (Run 619)
- CrSi2 (Run 620)
- (Cr,Mo)3Si (Run 621)
- Si3N4
- SiC
- TiSi2 (Run 641)
- WSi2 (Run 622)
- CrSi2-EM (Run 594)
- CrMoSi-EM (Run 623)
- TiSi2-EM (Run 639)
- WSi2-EM (Run 640)
Optical Macrographs of MoSi$_2$
Engineered Matrix

Thermally cycled between room temperature and 1500 K (2240 ºF) three times.

• Thermal cycling resulted in cracking.
• No longer considered in the program.
Isothermal Oxidation Behavior

![Graph showing the isothermal oxidation behavior of different materials at 1600 K.](image)

- **WSi₂-EM**
- **CrMoSi-EM**
- **CrSi₂-EM (spalled)**
- **SiC & Si₃N₄ (T = 1573 K) (Ogbuji & Opila (1995))**

**Y-axis:** $\Delta W/A$ (mg/cm²)

**X-axis:** t (h)

**Temperature:** T = 1600 K
Bend Stress-Strain Curves for CrMoSi-EM

![Graph showing stress-strain curves for CrMoSi-EM at different temperatures and conditions.](image-url)
Bend Stress-Strain Curves for CrSi$_2$-EM
Catastrophic oxidation occurred during heat-up to 1473 K.
Engineered Matrix Composites Fabrication

(courtesy R. Bhatt)

SiC Fiber

0/90 Fabric Weaving

CVI SiC Matrix Infiltration

CVI SiC Preform

Engineered Matrix Infiltration

CVI BN Interface Infiltration

Fiber coating

Melt Infiltration

EMC

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Microstructures of Particulate-Infiltrated SiC Fiber Preform

- Coated Preform
- Fibers and tows
- Voids
- Particulates
CT Scans of Particulate Infiltrated Preform

As-received Preform

- Area fraction of porosity ~ 21-23%

Particulate Infiltrated

- Area fraction of porosity ~ 0.9%

The red regions are voids
Summary and Conclusions

- Bend, CTE, isothermal oxidation and thermal cycling tests were conducted on several engineered silicide/SiC/Si$_3$N$_4$ matrices.

- Two promising engineered matrix compositions were down-selected for further development.

- Trials to infiltrate one of these engineered matrices into SiC-coated fiber preforms have been completed. Microstructural analysis and CT scans demonstrated almost complete infiltration of the preform.

- Efforts are underway to produce Engineered Matrix Composites (EMCs) specimens to determine self-healing capabilities.
Summary of Phase I Accomplishments: TRL 2

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<thead>
<tr>
<th>Milestone</th>
<th>Status</th>
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<tr>
<td>Demonstrate thermal strains for engineered matrices match those of SiC.</td>
<td>Completed.</td>
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<td>Demonstrate high temperature matrix plasticity.</td>
<td>Completed.</td>
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<td>Develop processing techniques for fabricating EMCs.</td>
<td>Particulate infiltration trials completed; melt infiltration trials to be completed.</td>
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<tr>
<td>Evaluate mechanical properties of EMCs and demonstrate self-healing capabilities.</td>
<td>To be completed.</td>
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Next Steps

• Complete tensile tests of monolithic specimens.

• Particulate and melt infiltrate bend specimens.

• Mechanical testing of EMC bend specimens.

• Evaluate self-healing properties in EMCs.

• Write and submit final report.

• Submit Phase II proposal.
Distribution and Dissemination

• Submitted NF 1679 to GRC patent attorney (LEW-18964-1).

• Submit papers to journals/NASA TMs after receiving approvals.

• Present papers at conferences after receiving approvals depending on the availability of travel funds.