Advanced Ceramic Matrix Composites with Multifunctional and Hybrid Structures

M. Singh
QSS Group, Inc.
NASA Glenn Research Center
Cleveland, OH 44135

G. N. Morscher
Ohio Aerospace Institute
NASA Glenn Research Center
Cleveland, OH 44135

Abstract

Ceramic matrix composites are leading candidate materials for a number of applications in aeronautics, space, energy, and nuclear industries. Potential composite applications differ in their requirements for thickness. For example, many space applications such as "nozzle ramps" or "heat exchangers" require very thin (< 1 mm) structures whereas turbine blades would require very thick parts (≥ 1 cm). Little has been investigated as to the effect of thickness on stress-strain behavior or elevated temperature tensile properties controlled by oxidation diffusion. In this study, composites consisting of woven Hi-Nicalon™ fibers a carbon interphase and CVI SiC matrix were fabricated with different numbers of plies and thicknesses. The effect of thickness on matrix crack formation, matrix crack growth and diffusion kinetics will be discussed.

In another approach, hybrid fiber-lay up concepts have been utilized to "alloy" desirable properties of different fiber-types for mechanical properties, thermal stress management, and oxidation resistance. Such an approach has potential for the C-SiC and SiC-SiC composite systems. CVI SiC matrix composites with different stacking sequences of woven C fiber (T300) layers and woven SiC fiber (Hi-Nicalon™) layers were fabricated. The results will be compared to standard C fiber reinforced CVI SiC matrix and Hi-Nicalon reinforced CVI SiC matrix composites. In addition, shear properties of these composites at different temperatures will also be presented. Other design and implementation issues will be discussed along with advantages and benefits of using these materials for various components in high temperature applications.
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Outline

• Introduction and Background
• Needs for CMCs with Hybrid Structures
• Advantages
  • Experimental
    – Composite Lay-ups and CVI
    – Elastic Moduli Measurements
    – Stress Rupture Testing
    – Microstructural Characterization (Optical, SEM)
• Results and Discussion
  – Thermomechanical Behavior
  – Microstructural Analysis
• Summary and Conclusions
Ceramic Matrix Composites Components for Aerospace Systems

- Turbine Rotor
- Turbopump Stator
- Turbine Rear Frame Leading Edge
- Interstage Shroud
- Nozzle Flaps and Seals
- Combustor Liner

Need of Ceramic Composites with Varying Thickness and Hybrid Structures

- Advanced Composites for Radiators
- Composite Vane for Aeroengine
- Cooled Panels for Nozzle Ramps
- Composite Blisks

Composites with varying thickness and architecture are needed.
Approaches to Composite Fabrication

GE Power Systems
Composites, Newark DE

CVI BN or C Infiltration

2D lay-up fixed in tooling

Interphase deposition, then removal from tool

Dog Bone
Tensile Bars Machined

Final CVI SIC Infiltration

8, 30, & 36 ply Standard Panels

Cut into Rectangular Shapes

Epoxy Infiltrate

Tensile Bars Machined

8 ply Epoxy-Infiltrated

CVI SIC Infiltration

CVI BN or C Infiltration

CVI SIC infiltration, removal from tool and delamination

Straight-Sided Tensile Bars Machined

1, 2, and 3 ply Delaminated Panels

Composites with Hybrid Lay-up

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Potential Benefits of Hybrid Lay-Up in Ceramic Matrix Composites

- Vary plies (fiber-types) to manipulate residual stress and matrix cracking
- Create "oxidation fire-walls" to slow down oxidation of C-fibers
- Can manipulate ply sequence for thermal-degradation (e.g., > SiC fibers on cold side and > C fibers on hot side) or residual stress-management

Tested Panels of Hybrid C/SiC Fiber CVI SiC Composites

- 20 "EPM" dogbone specimens for each (12.5 mm in grip; 10 mm in gage)
- ½ the dogbone specimens seal-coated with SiC and the other ½ seal-coated with CBS coating
- RT tensile with acoustic emission and elevated temperature stress-rupture tests were performed in air

<table>
<thead>
<tr>
<th>Panel</th>
<th>Composition</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>8% HN, 30% T300, 38% Total V&lt;sub&gt;f&lt;/sub&gt;</td>
<td>~2.2 mm</td>
</tr>
<tr>
<td>B</td>
<td>14% HN, 21% T300, 35% Total V&lt;sub&gt;f&lt;/sub&gt;</td>
<td>~3.2 mm</td>
</tr>
<tr>
<td>C</td>
<td></td>
<td>~3.3 mm</td>
</tr>
</tbody>
</table>

C-fiber ply

SIC-fiber ply
Room Temperature Tensile Behavior of Hybrid C/SiC Fiber CVI SiC Composites

Some "stiffening" with higher HN fraction

- HN/C/HiSiC: 28% HN
- T300/C/HiSiC: 40% C

Strain, %

Stress, MPa

A: gage grip delam
A (CBS): gage
B: gage
B (CBS): radius
Matrix Cracking in Hybrid C/SiC Fiber CVI SiC Composites

Matrix cracks counted over 10 mm length on surface and along 0° bundles on interior

Matrix cracking increases with stress similarly whether in C plies or HN plies

High Temperature Stress-Rupture Behavior in Air for Hybrid C/SiC Fiber CVI SiC Composites

Temperature Dependence for 69 MPa Rupture in Air

CBS coating provides best benefit at low stresses – no discernable difference for different fiber contents

Some benefit with more HN fibers for specimens not coated with CBS
High Temperature Stress-Rupture Behavior in Air for Hybrid C/SiC Fiber CVI SiC Composites

815°C Stress-Rupture

Stress Dependence for 815°C Rupture in Air

CBS coating provides best benefit at low stresses – no discernable difference for different fiber contents

Some benefit with more HN fibers for specimens not coated with CBS

Increased loading of HN in C+HN/SiC due to oxidation of C fibers will be too great to significantly prolong rupture life in air

Load if total load shed to Hi-Nicalon

815°C stress-rupture of HN/C/CVI SiC

Initial load from load-sharing

50 45 40 35 30 25 20 15 10 5 0

Time, hr

50 100 150 200

Stress, MPa

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Typical Fiber Fracture Surfaces (HN) or Lack Thereof (T300)

(A) SiC Seal-coat; 815°C 3.6 hr Stress-Rupture

Hi-Nicalon
T300 Carbon

(B) CBS 815°C, 47 hr Stress-Rupture

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(003) CBS 815°C, 47 hr Stress-Rupture

Sharp demarcation between CBS coating and SiC matrix.

CBS coating appears to coat surface and matrix cracks like a "blanket".

No observation of oxidation product 'plugging' (flowing into) matrix cracks.

Composites with Hybrid Lay-up

Summary and Conclusions

- Composite plates with alternating C and HiNicalon fiber plies could be fabricated with some delamination – probably better suited for tube-shaped structures
- HN plies do increase stiffness; however, this is mostly due to higher modulus of HiNicalon
  - Matrix cracking occurred at low stresses for all of the C fiber-containing composites
- Minor intermediate temperature stress-rupture improvement observed for HiNicalon containing composites
- CBS coating significantly improves stress-rupture life at low stresses, regardless of C and HiNicalon content

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Effect of Composite Thickness on Thermomechanical Behavior

Microstructure of Hi Nicalon SiC-CVI SiC Composites

1 Ply Longitudinal Section

3 Ply Longitudinal Section

8 Ply (BN1) Cross-Section

E8Ply-8HS(BN) Longitudinal Section

30 Ply Longitudinal Section
Physical Properties of Composite Specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Weave</th>
<th>Specimen shape</th>
<th>$t_2$ (mm)</th>
<th>$t_1$ (mm)</th>
<th>$l_1$ (mm)</th>
<th>$l_2$ (mm)</th>
<th>$f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>8 Ply (C)</td>
<td>S-68</td>
<td>dog-A</td>
<td>3.62</td>
<td>0.78</td>
<td>0.13</td>
<td>0.45</td>
<td>0.18</td>
</tr>
<tr>
<td>8 Ply (BN1)</td>
<td>S-68</td>
<td>dog-A</td>
<td>2.30</td>
<td>0.30</td>
<td>0.05</td>
<td>0.29</td>
<td>0.31</td>
</tr>
<tr>
<td>9 Ply (BN2)</td>
<td>S-68</td>
<td>dog-B</td>
<td>2.50</td>
<td>0.35</td>
<td>0.06</td>
<td>0.40</td>
<td>0.13</td>
</tr>
<tr>
<td>9 Ply (BN3)</td>
<td>S-68</td>
<td>dog-B</td>
<td>2.38</td>
<td>0.33</td>
<td>0.05</td>
<td>0.47</td>
<td>0.15</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Standard Thick Panels</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 Ply (C)</td>
</tr>
<tr>
<td>10 Ply (D)</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Determined Thick Panels</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Ply (C)</td>
</tr>
<tr>
<td>2 Ply (C)</td>
</tr>
<tr>
<td>2 Ply (D)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Epoxy Infiltrated Panels</th>
</tr>
</thead>
<tbody>
<tr>
<td>EP3y-0H6 (BN1)</td>
</tr>
<tr>
<td>EP3y-0H10 (BN2)</td>
</tr>
<tr>
<td>EP3y-0H10 (BN3)</td>
</tr>
</tbody>
</table>

a Dogbone tensile specimens 285 mm in length, approximately 15.5 mm in width at grip section and 10.5 mm in width at gauge section.
b Dogbone tensile specimens 152 mm in length, approximately 12.6 mm in width at grip section and 10.3 mm in width at gauge section.
c Straight-sided tensile specimen 152 mm in length and approximately 12.6 mm in width throughout.

Tensile Stress-Strain Behavior of Different HiNicalon-CVI SiC Composites

8 Ply (BN1); E = 256 GPa, $f_v = 0.17$; 2.5 mm thick
36 Ply; E = 217 GPa, $f_v = 0.17$; 10.5 mm thick
3 Ply; E = 114 GPa, $t_f = 0.16$; 0.92 mm thick
2 Ply; E = 102 GPa, $f_v = 0.14$; 0.76 mm thick
8 Ply (BN2); E = 244 GPa, $f_v = 0.16$; 2.45 mm thick
2 Ply; E = 102 GPa, $f_v = 0.14$; 0.76 mm thick
8 Ply (BN1); E = 203 GPa, $f_v = 0.18$; 2.35 mm thick
4 Ply (C); E = 177 GPa, $f_v = 0.14$; 2.26 mm thick

$\rho$ refers to the volume fraction of fiber in the loading direction.

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Stress on Fibers at Failure for Different CVI SiC Composite Specimens

\[ \sigma_f = 2342 \pm 329 \text{ MPa} \]

Effect of Various Physical Properties on Elastic Modulus of Composites

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Polished Longitudinal Section Showing Matrix Cracks in 8ply BN3

Estimated Crack Density versus Strain
Effect of Composite Thickness

Summary and Conclusions

- The effect of constituent content on elastic modulus and matrix cracking behavior not only depends on the relative amounts of constituents, but also on the effectiveness of the structure, i.e., 90° minicomposites, to carry load.
- Lower density composites have very little load-carrying contribution from 90° minicomposites when loaded in the 0° direction.
- Higher density composites were affected by 90° minicomposites as low-stress flaw sources, whereas the matrix cracking behavior of low density 2D woven composites were not and behave very much like single tow minicomposites as opposed to high density 2D woven composites.
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