Evaluation of CVI SiC/SiC Composites for High Temperature Applications

D. Kiser, A. Almansour, and C. Smith, NASA Glenn Research Center, Cleveland, OH

D. Gorican and R. Phillips, Vantage Partners, LLC (at NASA GRC), Cleveland, OH

R. Bhatt, Ohio Aerospace Institute (at NASA GRC), Cleveland, OH

T. McCue, SAIC (at NASA GRC), Cleveland, OH

Presented at the 41st Annual Conference on Composites, Materials, and Structures
(U.S. Citizens Only / ITAR-Restricted Sessions)

January 23-26, 2017
Cocoa Beach / Cape Canaveral, Florida

Research Supported by the NASA Transformational Tools and Technology Project

Distribution A. Approved for public release. Distribution is unlimited.
Critical Aeronautics Technologies (CAT) Sub-Project

• High Temperature Engine Materials

• *Technical Challenge*: Develop high temperature materials for turbine engines that enable a 6% reduction in fuel burn for commercial aircraft, compared to current SOA materials
SiC/SiC Components for Gas Turbine Engines: Benefits

- Reduced component weight (1/3 density of superalloys)
- Higher temperature capability/increased thermal margin
- Reduced cooling requirements
- Improved fuel efficiency
- Reduced emissions ($\text{NO}_x$ and $\text{CO}_2$)

**Incentive to Increase Engine Operating Temperatures**

Further increase with 2700°F CMC components
Evaluation of CVI SiC/SiC Composites for High Temperature Applications

Objectives

- Establish stress-dependent and temperature-dependent parameters for modeling SiC/SiC composite creep behavior.

\[ \varepsilon' = B \sigma^n \]
\[ \ln(\varepsilon') = n \ln(\sigma) + \ln(B) \]
\[ B = A \times e^{\left(\frac{-Q}{RT}\right)} \]

Where \( \varepsilon' \) is creep strain rate, B and A are constants, \( \sigma \) is the applied stress, \( n \) is the stress exponent, \( Q \) is the apparent activation energy, \( R \) is the gas constant and \( T \) is the temperature in K.

- Determine damage mechanisms and failure modes under creep deformation from 2200°F (1200°C) to 2700°F (1482°C) in air.
Evaluation of CVI SiC/SiC Composites for High Temperature Applications

**Approach**

- Building on a previous GRC study\(^1\) of 2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic\(^{TM}\)-iBN SiC fabric (manufactured by Hyper-Therm*)

- Building on previous SiC fiber and SiC/SiC CMC and minicomposites creep modeling (DiCarlo\(^2\), Shinavski\(^3\), Bhatt\(^4\), and Almansour\(^5\))

- Conduct CMC creep study at 2200°F (1200°C) to 2700°F (1482°C) —with a limited number of specimens

- Examine samples following 2700°F (1482°C) creep testing (run-out condition) and characterize their residual properties / integrity

---

* Hyper-Therm HTC, Inc. became Rolls-Royce HTC
2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic™-iBN SiC fabric (manufactured by Hyper-Therm)

Machined EPM geometry samples were CVI SiC seal-coated to seal the coupons’ edges
EPM Tensile Geometry: 6” Dog-bone Sample

gage section: 20% reduction in width, with tapering from 0.5” (grip) to 0.4” (gage)
Representative RT FF Stress-Strain Curve

For Comparison
Sylramic™-iBN SiC Fiber-Reinforced CVI SiC Matrix CMC with BN Interphase — Fast Fracture Testing

- Room T FF (Ave. 3 tests)
  - σ ULT ave.: 47.1 ksi (325 MPa)
  - ε ULT ave.: 0.24%
  - PLS ave.: 18.6 ksi (128 MPa)
  - E ave.: 47.7 Msi (329 GPa)

- 2700°F FF (Ave. 2 tests)
  - σ ULT ave.: 31.9 ksi (220 MPa)
  - ε ULT ave.: 0.17%
  - PLS ave.: 32.9 Msi (227 GPa)

Creep Testing: Stay below RT PLS

Ref. 1
Sylramic™-iBN SiC Fiber-Reinforced CVI SiC Matrix
CMC with BN Interphase — 2700°F Tensile Creep, 10 ksi, Air

**Tensile Creep Test Elevated Temperature**

- SBN 04-15 Tested 2700 deg F @ 10 Ksi

**Representative Creep Curve**

- Three Samples Tested -- Observed
- Virtually Identical Creep Behavior
- Ave. Total Strain = 0.21%

**Residual Properties**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>σ ULT.</td>
<td>46.6 ksi</td>
</tr>
<tr>
<td>ε ULT</td>
<td>0.28%</td>
</tr>
<tr>
<td>PLS</td>
<td>18.8 ksi</td>
</tr>
<tr>
<td>E</td>
<td>47.3 Msi</td>
</tr>
</tbody>
</table>

- Residual Properties Very Similar to Those of the As-Received CMC
- Run Discontinued at 100 hrs

**Conclude that the matrix did not crack and that the fibers were not degraded during creep**
Material

- Similar to CMC material from previous GRC study

- 2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic™-iBN SiC fabric

- Machined tensile samples were CVI SiC seal-coated to seal the coupons’ edges

- Made by HTC (via NASA LaRC-funded SBIR Phase II Contract NNX11CB63C). Bequeathed by D. Brewer

- Relevant material system, especially for 2700°F applications
Creep of CVI SiC/SiC CMC

- When CMCs are loaded below the matrix cracking stress (PLS), fibers are not exposed to oxidation damage and they carry a fraction of the applied load.

- If the matrix is more creep resistant, the fiber unloads over time and matrix load increases, which increases the possibility of matrix damage.
# 2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air—5 Different Conditions, and RT FF of As-Received

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Test Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1520-S2-1</td>
<td>2700°F, 10 ksi for 100 hrs</td>
</tr>
<tr>
<td>1520-S2-2</td>
<td>2700°F, SPLCF*, R=0.5, 5 / 10 ksi for 100 hrs</td>
</tr>
<tr>
<td>1520-S2-3</td>
<td>2700°F, 10 ksi for 100 hrs, 12.5 ksi for 100 hrs, 15 ksi for 100 hrs</td>
</tr>
<tr>
<td>1520-S2-4</td>
<td>RT FF Tensile Test</td>
</tr>
<tr>
<td>1520-S2-5</td>
<td>2200°F, 12.5 ksi for 100 hrs, 2460°F, 12.5 ksi for 100 hrs, 2700°F, 12.5 ksi for 100 hrs</td>
</tr>
<tr>
<td>1520-S2-6</td>
<td>2700°F, 12.5 ksi for 300 hrs</td>
</tr>
</tbody>
</table>

* Sustained Peak Low Cycle Fatigue

Creep Testing: Stay below RT PLS
2D CVI SiC/SiC Reinforced with Sylramic™-iBN:
Testing at Room Temp.: As-Rec. and Following Creep in Air

Used various characterization approaches (AE, resistivity, hysteresis testing, and fractographic) to determine which ones provide the most useful post-test information.

[S2-6 (Post-creep) Unique Acoustic Emission (AE) Set-up]

[S2-4 (As-Fabricated Sample) Prepped for Resistivity Measurement]

[In Progress]
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Testing at Room Temp.: As-Rec. and Following Creep in Air

Unique Acoustic Emission (AE) Set-up for Characterizing Cracking
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Testing at Room Temp.: As-Rec. and Following Creep in Air

Also Collecting Resistivity Data for Characterizing Cracking
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air—Results of 5 different testing conditions

- One test per condition

- No failures occurred due to creep
- All tests were run-outs: discontinued as-planned
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air at 2700°F (1482°C)

- Held at T without loading following test to observe elastic creep recovery
- Material shows less creep than Study 1 CMC
- Strain rate measured at end of each test

- S2-6
- S2-1
- S2-2
- SPLCF, R = 0.5, 5 / 10 ksi
- 2700°F, 10 ksi for 100 hrs
- 2700°F, 12.5 ksi for 300 hrs
- Strain rate measured at end of each test
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air at 2700°F (1482°C)—Exposed to 3 stresses

Determination of CMC Creep Stress-Dependence

- Very similar to S2-1 creep curve
- Strain rate measured at end of each 100 hr segment

Creep Strain, %

<table>
<thead>
<tr>
<th>Exposure Time, Hours</th>
<th>Creep Strain, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>50</td>
<td>0.05</td>
</tr>
<tr>
<td>100</td>
<td>0.15</td>
</tr>
<tr>
<td>150</td>
<td>0.20</td>
</tr>
<tr>
<td>200</td>
<td>0.24</td>
</tr>
<tr>
<td>250</td>
<td>0.25</td>
</tr>
<tr>
<td>300</td>
<td>0.30</td>
</tr>
</tbody>
</table>

- 69 MPa 86.2 MPa 103.4 MPa
- 10 Ksi 12.5 Ksi 15 Ksi

Sample S2-3
Determination of CMC Creep Stress Dependence

\[ \varepsilon' = B \sigma^n \]

- \( B \) is a constant \( = 9.72 \times 10^{-15} \)
- \( n \) is the stress exponent \( \sim 3.5 \)

\[ y = 9.72E-15x^{3.48E+00} \]
\[ R^2 = 9.77E-01 \]

Creep Strain Rate at 100 hrs, \( S^{-1} \):

- 2700 °F
- 15 Ksi: 103.4 MPa
- 12.5 Ksi: 86.2 MPa
- 10 Ksi: 69 MPa

Sample S2-3

2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air at 2700°F (1482°C)
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures

Examination of CMC Creep Temperature-Dependence at 2200-2700 °F

- Strain rate measured at end of each 100 hr segment

- Held at 2700 °F to assess creep recovery; broke during cool-down

Sample S2-5
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures

Examination of CMC Creep Temperature-Dependence at 2200-2700 °F

\[ y = -12849x - 8.8175 \]
\[ R^2 = 0.9681 \]

Sample S2-5

- Activation Energy is dependent on when strain rate is measured (hrs of creep completed)
- Contribution from Primary Creep leads to low Activation Energy

Slope = \( \frac{-Q}{R} \)

\( R = \) Gas Constant = 8.314 J/K.mol
\( Q = \) Activation Energy = 107 KJ/mol at 100 hr, 86.2 MPa
CMC Creep Dependence on Mechanical and Thermal Loading Histories

Example of How Measured Strain Rate Depends on Loading History

Specimen Number
S2-5
After: 12.5 ksi: 100 hrs at 2200°F, 100 hrs at 2460°F, 100 hrs at 2700°F

S2-3
After: 100 hrs at 2700°F (10 ksi), 100 hrs at 2700°F (12.5 ksi)

S2-6
After: 12.5 ksi: 300 hrs at 2700°F

Creep Strain Rate, S^{-1}
0  2E-08  4E-08  6E-08  8E-08  0.0000001  1.2E-07
# 2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air—5 Different Conditions, and RT FF of As-Received

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Test Condition (Temperature: °F, Stress: ksi, Time: hrs)</th>
<th>RT Fast Fracture Residual UTS (ksi, MPa)</th>
<th>RT Fast Fracture Ultimate Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1520-S2-1</td>
<td>2700°F, 10 ksi for 100 hrs</td>
<td>49.5, 341</td>
<td>0.26</td>
</tr>
<tr>
<td>1520-S2-2</td>
<td>2700°F, SPLCF, R=0.5, 5 / 10 ksi for 100 hrs</td>
<td>51.6, 356</td>
<td>Ext. Moved</td>
</tr>
<tr>
<td>1520-S2-3</td>
<td>2700°F, 10 ksi for 100 hrs, 12.5 ksi for 100 hrs, 15 ksi for 100 hrs</td>
<td>45.1, 311</td>
<td>0.31</td>
</tr>
<tr>
<td>1520-S2-4</td>
<td>RT Tensile test</td>
<td>49.5, 341</td>
<td>0.33</td>
</tr>
<tr>
<td>1520-S2-5</td>
<td>2200°F, 12.5 ksi for 100 hrs, 2460°F, 12.5 ksi for 100 hrs, 2700°F, 12.5 ksi for 100 hrs</td>
<td>Broke upon cooling</td>
<td>Not tested at RT</td>
</tr>
<tr>
<td>1520-S2-6</td>
<td>2700°F, 12.5 ksi for 300 hrs</td>
<td>47.1, 325</td>
<td>0.31</td>
</tr>
</tbody>
</table>
Work Remaining / Future Work

- Fractography and microstructural characterization.
- Analyze AE (acoustic emission) and resistivity data.
- Analyze hysteresis testing data.
- Compare fiber loadings: Study 1 (previous) and Study 2 (current) materials.
- Review SiC/SiC activation energy data in open literature.
- Examine crack spacing in gage section of tested samples.
- Examine data collected when specimens were held at T following the creep testing to see how much strain recovery occurred.
- **Prepare updated presentation (A. Almansour presenting at Pac Rim Conf. in 2017).**
- Consider obtaining another panel of CVI SiC/SiC and conduct testing at 2700°F / 3 stresses and 12.5 ksi / 3 temperatures. Test minimum 2 samples per condition. Use selected post-test analysis techniques.
Summary and Conclusions

• CVI SiC/SiC CMCs incorporating Sylramic™-iBN SiC fiber are being evaluated via tensile creep testing to determine creep parameters for modeling.

• A stress exponent was determined at 2700°F, and an activation energy was calculated.

• As reported previously (Shinavski et al³), the activation energy measured depends on the time/strain at which strain rates are measured, and on loading history.

• All creep specimens achieved a run-out condition. Fractography conducted on those samples following RT FF residual strength measurement will help determine whether or not any samples cracked during creep testing.

• We are investigating various approaches to analyzing specimens following creep testing such as AE and resistivity to help us understand CMC damage mechanisms.


