Evaluation of CVI SiC/SiC Composites for High Temperature Applications

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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

Further increase with 2700°F CMC components

- Reduced emissions ($$\text{NO}_x$$ and $$\text{CO}_2$$)

Incentive to Increase Engine Operating Temperatures
Objectives

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

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

\[ \ln(\varepsilon') = n \ln(\sigma) + \ln(B) \]

\[ B = A^* e^{\left(-\frac{Q}{RT}\right)} \]

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™-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*
Previous Study

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

Representative RT FF Stress-Strain Curve

<table>
<thead>
<tr>
<th></th>
<th>Room T FF (Ave. 3 tests)</th>
<th>2700°F FF (Ave. 2 tests)</th>
</tr>
</thead>
<tbody>
<tr>
<td>σ ULT ave.</td>
<td>47.1 ksi (325 MPa)</td>
<td>31.9 ksi (220 MPa)</td>
</tr>
<tr>
<td>ε ULT ave.</td>
<td>0.24%</td>
<td>0.17%</td>
</tr>
<tr>
<td>PLS ave.</td>
<td>18.6 ksi (128 MPa)</td>
<td></td>
</tr>
<tr>
<td>E ave.</td>
<td>47.7 Msi (329 GPa)</td>
<td>32.9 Msi (227 GPa)</td>
</tr>
</tbody>
</table>

Creep Testing: Stay below RT PLS

For Comparison

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

1482°C, 69 MPa

Tensile Creep Test Elevated Temperature

Representative Creep Curve

Strain (%) vs Time (hours)

Three Samples Tested -- Observed

Virtually Identical Creep Behavior

Ave. Total Strain = 0.21%

Residual Properties Very Similar to Those of the As-Received CMC

Run Discontinued at 100 hrs

Residual RT FF Properties

<table>
<thead>
<tr>
<th></th>
<th>Ave. 3 tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma_{ULT}$</td>
<td>46.6 ksi</td>
</tr>
<tr>
<td>$\epsilon_{ULT}$</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>

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 (Temperature: °F, Stress: ksi, Time: hrs)</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
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 fractography) 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

Examine cracking in gage section

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
- 2700°F, 2.5 ksi for 300 hrs
- Material shows less creep than Study 1 CMC
- Strain rate measured at end of each test

- 2700°F, 10 ksi for 100 hrs
- SPLCF, R = 0.5, 5 / 10 ksi

Creep Strain, %
Exposure Time, Hours
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

Sample S2-3

Creep Strain, %

Exposure Time, Hours

Strains:
- 69 MPa (10 Ksi)
- 86.2 MPa (12.5 Ksi)
- 103.4 MPa (15 Ksi)
2D CVI SiC/SiC Reinforced with Sylramic™-iBN: Creep in Air at 2700°F (1482°C)

Determination of CMC Creep Stress Dependence

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

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

\[ y = 9.72 \times 10^{-15} x^{3.48 \times 10^{00}} \]

\[ R^2 = 9.77 \times 10^{-01} \]
Determination of CMC Stress Dependence Creep at 2700 °F

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

Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures

Sample S2-5

Heating / expansion

0 50 100 150 200 250 300 350

Exposure Time, Hours

0 0.05 0.1 0.15 0.2 0.25 0.3 0.35 0.4

Creep Strain, %

1200°C 2200 °F
1350°C 2460 °F
1482°C 2700 °F

Sylramic™-iBN/iBN/BN/CVI-SiC Reinforced with Sylramic™-iBN:

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

- 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

Heating / expansion

0 50 100 150 200 250 300 350

Exposure Time, Hours

0 0.05 0.1 0.15 0.2 0.25 0.3 0.35 0.4

Creep Strain, %

1200°C 2200 °F
1350°C 2460 °F
1482°C 2700 °F

Sylramic™-iBN/iBN/BN/CVI-SiC Reinforced with Sylramic™-iBN:
Determination of CMC Stress Dependence Creep at 2700 °F

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

Slope = \(-Q/R\)

\( R = \text{Gas Constant} = 8.314 \text{ J/K.mol} \)
\( Q = \text{Activation Energy} = 107 \text{ KJ/mol} \)

at 100 hr, 86.2 MPa

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

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:
Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures
CMC Creep Dependence on Mechanical and Thermal Loading Histories

Example of How Measured Strain Rate Depends on Loading History

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Loading History</th>
</tr>
</thead>
<tbody>
<tr>
<td>S2-5</td>
<td>After: 12.5 ksi: 100 hrs at 2200°F, 100 hrs at 2460°F, 100 hrs at 2700°F</td>
</tr>
<tr>
<td>S2-3</td>
<td>After: 100 hrs at 2700°F (10 ksi), 100 hrs at 2700°F (12.5 ksi)</td>
</tr>
<tr>
<td>S2-6</td>
<td>After: 12.5 ksi: 300 hrs at 2700°F</td>
</tr>
</tbody>
</table>

Creep Strain Rate, S

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 al.), 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.
References


