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 (NOₓ and CO₂)

Incentive to Increase Engine Operating Temperatures
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.

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

Where \( \dot{\varepsilon} \) 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
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

For Comparison

**Creep Testing:**
Stay below RT PLS

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<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>

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

Three Samples Tested -- Observed

Virtually Identical Creep Behavior

Ave. Total Strain = 0.21%

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

Residual RT FF Properties

<table>
<thead>
<tr>
<th></th>
<th>Ave. 3 tests</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>

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 Syrlamic™-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><strong>1520-S2-4</strong></td>
<td><strong>RT FF Tensile Test</strong></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 fractography) to determine which ones provide the most useful post-test information.

[ In Progress ]

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

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

Creep Strain, %

Exposure Time, Hours

Creep in Air—Results of 5 different testing conditions

- 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, 12.5 ksi for 300 hrs
- 2700°F, 10 ksi for 100 hrs
- SPLCF, R = 0.5, 5 / 10 ksi
- Material shows less creep than Study 1 CMC
- 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

Sample S2-3

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, %

0 0.05 0.1 0.15 0.2 0.25 0.3

Exposure Time, Hours

0 50 100 150 200 250 300 350

69 MPa 86.2 MPa 103.4 MPa

10 Ksi 12.5 Ksi 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 = $9.72 \times 10^{-15}$
- n is the stress exponent $\sim 3.5$

Sample S2-3

<table>
<thead>
<tr>
<th>Stress, MPa</th>
<th>Creep Strain Rate at 100 hrs, S^-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>69</td>
<td>2.66E-08</td>
</tr>
<tr>
<td>86.2</td>
<td>4.51E-08</td>
</tr>
<tr>
<td>103.4</td>
<td>1.64E-07</td>
</tr>
</tbody>
</table>

Equation:

\[ y = 9.72E-15 \times 3.48E+00 \]

R² = 9.77E-01
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 Syrlamic™-iBN: Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures

Determination of CMC Stress Dependence Creep at 2700 °F

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

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

S2-5

2200 °F

1200 °C

1482 °C

1350 °C

2700 °F

2460 °F

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

Temperature Dependence 2200 – 2700 °F under 86 MPa in Air

Slope = \((-Q)/R\)

R = Gas Constant = 8.314 J/K.mol

Q = Activation Energy = 107 KJ/mol

Activation Energy is dependent on when strain rate is measured (hrs of creep completed)

Contribution from Primary Creep leads to low Activation Energy

Sample S2-5
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.
References


