Mechanical properties and real-time damage evaluations of environmental barrier coated SiC/SiC CMCs subjected to tensile loading under thermal gradients

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SiC/SiC ceramic matrix composites (CMCs) require new state-of-the-art environmental barrier coatings (EBCs) to withstand increased temperature requirements and high velocity combustion corrosive combustion gasses.

The present work compares the response of coated and uncoated SiC/SiC CMC substrates subjected to simulated engine environments followed by high temperature mechanical testing to assess retained properties and damage mechanisms.

Our focus is to explore the capabilities of electrical resistance (ER) measurements as an NDE technique for testing of retained properties under combined high heat-flux and mechanical loading conditions.

Furthermore, Acoustic Emission (AE) measurements and Digital Image Correlation (DIC) were performed to determine material damage onset and accumulation.
SiC/SiC CMC material (Hyper-Therm HTC; currently Rolls Royce HTC)
- 8 plies, balanced 5 harness satin 2D woven 0°/90°, SiC/BN/SiC
- Hi-Nicalon Type-S fiber reinforced
- Produced by CVI + SiC/Si slurry melt infiltration (SMI)
- Machined into 6 in. tensile bars

Environmental Barrier Coating – EBC
- Deposited via EB-PVD
- NASA HfO$_2$-Si bond coat
- NASA HfO$_2$-doped ytterbium-gadolinium di-silicate (Yb,Gd)$_2$Si$_2$O$_7$ EBC system

<table>
<thead>
<tr>
<th>Specimen</th>
<th>width (mm)</th>
<th>thickness (mm)</th>
<th>$f_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>coated</td>
<td>12.80</td>
<td>2.25</td>
<td>0.143</td>
</tr>
<tr>
<td>uncoated</td>
<td>12.72</td>
<td>2.21</td>
<td>0.145</td>
</tr>
</tbody>
</table>
Experimental Technique: combustion environment exposure

- **NASA High Pressure Burner Rig (HPBR)**
  - Closely simulates aero-turbine engine combustion environments for specimen and component testing
  - Burns jet-fuel and air at user controlled ratios
  - Used to quantify high temperature material oxidation and T/EBC performance over a range of temperatures, pressures and velocities

- Specimens subjected to HPBR exposure at **1316°C for 30 hours** at gas pressures of **10 atm** and combustion gas velocities of and **200 m/s**

**HPBR CAPABILITIES**
- Jet fuel & air combustion with mass air flow 1.5-2.0 lb/s
- Gas temperature up to 3000°F (1650°C)
- Adjustable testing pressures from 4 to 16 atmospheres
- Gas velocity up to 850 m/s combustion gas velocity in the testing section
- Incorporated advanced air preheater for 800-1200°F cooling air for high temperature film cooling
Experimental Technique: high temperature tensile testing

- Specimens are loaded in uni-axial tension rig
- Digital Image Correlation (DIC) is used to determine localized strain fields
- Nominal strain measurements are taken using a 25.4 mm extensometer with a ±0.5 mm travel

Laser high heat-flux testing:
- Face of specimen gage-section heated by a 3.5kW CO$_2$ high heat-flux laser
- Asymmetrical heating by laser generates thermal gradients (thru thickness and longitudinal)
- Thermal gradients can be increased by the addition of active back side air-cooling
- Front and back temperatures of the heated region are monitored by optical pyrometers
NDE Measurement

- Electrical Resistance (ER) measured by four-point probe method
- In order to avoid high temperature exposure during laser heating, ER leads for in-situ measurement are attached within the gripped areas
- Acoustic Emission (AE) sensors are attached ±40 mm from center
Modal Acoustic Emission Monitoring

- Fracture energy of solids released as elastic waves which are detected by the use of wide-band sensors in order to quantify stress-dependent cracking initiation and accumulation.

- Location of AE events estimated by the difference in arrival times of AE signals

\[ x = \frac{v}{2}(t_{bottom} - t_{top}) \]

Electrical Resistance Measurement

- Damage in the form of matrix cracks and associated fiber debonding/sliding increase the overall electrical resistance of the composite specimen

- Matrix cracking of MI SiC/SiC is especially sensitive due to the highly conductive matrix formed from excess silicon deposits left from processing
Post-HPBR: Retained HT Tensile Properties

- Mechanical behavior of the uncoated specimen indicates severe degradation of composite properties
- Oxidation of CMC in HPBR
  - Recession of SiC (matrix, fibers), Oxidation of BN interphase

### Table: Specimen Properties

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Surface Temp. (°C)</th>
<th>Back Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>coated</td>
<td>1230</td>
<td>1070</td>
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<tr>
<td>uncoated</td>
<td>1200</td>
<td>1010</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Specimen</th>
<th>E (GPa)</th>
<th>Extensometer</th>
<th>DIC</th>
<th>σ_{UTS} (MPa)</th>
<th>ε_{fail} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>coated</td>
<td>241</td>
<td>266</td>
<td>238</td>
<td>0.371</td>
<td></td>
</tr>
<tr>
<td>uncoated</td>
<td>146</td>
<td>221</td>
<td>166</td>
<td>0.134</td>
<td></td>
</tr>
</tbody>
</table>
Accumulation of AE energy indicative of stress-dependent cracking behavior
- Bridged vs. unbridged matrix cracking

Small ER increase prior to AE onset (approx. 115 MPa and 150 MPa respectively)

Drastically different ER behavior in increased stress region
HT Tensile Test: ER change with plastic deformation

- Little change in with elastic strain, followed by increased rate with plastic strain
- High strain sensitivity
  - ~200% ER change to failure
- ER response indicative of nature of damage accumulation
  - Coated v. uncoated

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Electrical Resistance (ohm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Room temp.</td>
</tr>
<tr>
<td>coated</td>
<td>0.61985</td>
</tr>
<tr>
<td>uncoated</td>
<td>0.73418</td>
</tr>
</tbody>
</table>

Post HPBR exposure
Energy distribution of AE events recorded in specimen gage section with corresponding DIC strain mapping at failure stress.

**COATED**
- Nominal thermomechanical strain: 0.96%
- Failed below area of DIC interest

**UNCOATED**
- Nominal thermomechanical strain: 0.71%
AE waveform analysis: frequency content

- Frequency centroid was calculated for each waveform captured by top and bottom sensors for each AE event.
- The FC of damage events in similar MI SiC/SiC laminates has been shown to be in the range of 600 kHz – 1200 kHz [Maillet and Morscher, Mech Syst. Signal Processing 2015].
- Coated sample exhibited a dense, low freq. (<375 Hz) cluster beginning ~125MPa
  - Highlighted in red

![Graphs showing frequency centroid vs. stress for top and bottom sensors.](image)

Similar FC content on both sensors.
Closer investigation of the low frequency cluster seen in the coated sample shows that are spatially dispersed throughout the gage.

These low FC events also exhibit similar low energy content.
Conclusions

- Decrease in retained tensile properties post HPBR exposure clearly shows degradation of uncoated specimen, and in turn the increased performance benefit of the NASA EBC system.

- ER measurement shown to be an effective tool for in-situ damage monitoring of MI SiC/SiC CMCs under high-temperature thermal gradients.
  - Damage onset indicated by steep ER increase in both cases.
  - Increases in ER response show high sensitivity (100’s of % increase to failure).

- AE energy distribution in good agreement with DIC strain mapping in terms of damage location and distribution.

- AE waveform analysis revealed some differences in frequency content and energy between the coated and uncoated samples.
  - While further study is required, there is evidence that AE analysis can be used to differentiate EBC from CMC damage events.
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  ➢ NASA Transformational Tools and Technologies Project

Questions?