Creep and environmental durability of EBC/CMCs under imposed thermal gradient conditions

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Abstract
Interest in SiC fiber-reinforced SiC ceramic matrix composite (CMC) environmental barrier coating (EBC) systems for use in high temperature structural applications has prompted the need for characterization of material strength and creep performance under complex aerospace turbine engine environments. Stress-rupture tests have been performed on SiC/SiC composites systems, with varying fiber types and coating schemes to demonstrate material behavior under isothermal conditions. Further testing was conducted under exposure to thermal stress gradients to determine the effect on creep resistance and material durability. In order to understand the associated damage mechanisms, emphasis is placed on experimental techniques as well as implementation of non-destructive evaluation; including electrical resistivity monitoring. The influence of environmental and loading conditions on life-limiting material properties is shown.

Introduction

In an effort to develop understanding of failure mechanisms associated with high-temperature creep of CMC’s under both isothermal and thermal gradient conditions was conducted.

The effect of EBC’s on environmental durability and stressed oxidation was examined.

Electrical resistance measurements were utilized in order to monitor in-situ material degradation, and model material behavior.

Materials

The samples in this study were 2-D woven SiC/SiC melt infiltrated ceramic matrix composites:
- 8 ply, 5 harness satin weave
- Tyranno ZMI reinforcing fibers with BN interphase coating
- Samples machine into dog-bone shape for testing

<table>
<thead>
<tr>
<th></th>
<th>ZMI-1</th>
<th>ZMI-2</th>
<th>ZMI-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width (mm)</td>
<td>10.686</td>
<td>10.129</td>
<td>10.455</td>
</tr>
<tr>
<td>Thickness (mm)</td>
<td>4.32</td>
<td>4.231</td>
<td>4.686</td>
</tr>
<tr>
<td>Fiber Volume Fraction, f</td>
<td>0.149</td>
<td>0.137</td>
<td>0.137</td>
</tr>
<tr>
<td>Coating</td>
<td>EBC - 1</td>
<td>uncoated</td>
<td>EBC - 2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Bond Coat</th>
<th>Ceramic Coating</th>
</tr>
</thead>
<tbody>
<tr>
<td>EBC - 1</td>
<td>HIO$_2$Si</td>
</tr>
<tr>
<td>EBC - 2</td>
<td>HIO$_2$Si + Yb$_2$Si$_2$O$_7$ (= 127μm) + Hf-RE silicate (= 254 μm)</td>
</tr>
</tbody>
</table>

Some of the specimens tested had been coated with multilayer environmental barrier coating systems

Experimental

- The specimen surface heating was provided by a high heat flux CO$_2$ laser system.
- Using an integrated rotating lens a uniform laser power distribution is achieved the gauge region of the sample face.
- In order to induce the desired through thickness thermal gradient, controlled cooling air was delivered to the back side of the CMC.
- The front side and back side temperatures of the specimen were monitored throughout testing by an 8μm and two-color pyrometer respectively.

Results

<table>
<thead>
<tr>
<th>Test Sample</th>
<th>Coating</th>
<th>RT Resistivity at T (Ω cm)</th>
<th>Resistivity at T (Ω cm)</th>
<th>Temperature (°C)</th>
<th>Stress σ at T (MPa)</th>
<th>Time (hrs)</th>
<th>Total Strain (%)</th>
<th>Creep Strain (%)</th>
<th>Resilience at T (Ω cm) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZMI-1</td>
<td>EBC-1</td>
<td>0.0648</td>
<td>0.130</td>
<td>1050</td>
<td>68.9</td>
<td>501</td>
<td>0.07902</td>
<td>0.03167</td>
<td>0.183</td>
</tr>
<tr>
<td>ZMI-2*</td>
<td>uncoated</td>
<td>0.0367</td>
<td>0.0935</td>
<td>1099.5/1033.87</td>
<td>68.9</td>
<td>209.3</td>
<td>0.11237</td>
<td>0.04782</td>
<td>0.189</td>
</tr>
<tr>
<td>ZMI-3*</td>
<td>EBC - 2</td>
<td>0.0636</td>
<td>0.113</td>
<td>1282/1010</td>
<td>68.9</td>
<td>526</td>
<td>0.13178</td>
<td>0.07678</td>
<td>0.167</td>
</tr>
</tbody>
</table>

In order to determine the sensitivity of the ER measurement during high-temperature testing. The dependence of electrical resistance on heating was examined for an uncoated specimen. This test was performed in the absence of mechanical loading and will be used in future work to model the electrical response of the composite system to thermo-mechanical loading.

Post - test examination

- Fracture Surface exhibits little to no fiber pull-out. Oxidation observed on fiber fracture surfaces through the entire thickness, indicated stressed oxidation assisted crack growth.
- Much higher density of cracks, as compared to coated samples, growing from both front and back sides, and running through thickness.
- Abundance of thru-thickness cracking.
- Majority of surface cracks observed begin on the non-coated surface and do not penetrate very deeply.
- Higher degree of fiber pull-out near coated surface, decreasing as you progress towards the back surface.
- More oxidation observed on plies adjacent to back surface.

Conclusions

- It has been seen that there is a significant difference in creep life and associated failure mechanisms of CMC materials due to the presence/absence of coating systems and thermal gradient conditions.
- Due to through-thickness thermal gradients, the cooler surface of the specimen sees lower levels of stress relaxation; essentially forcing more of the load onto a smaller cross-sectional area. This in turn leads to higher levels of stress induced cracking.
- Furthermore, as these crack continue to open increased levels of oxidation ingress becomes even increasing leading premature fracture of the material.
- The addition of the EBC coating seems to decrease oxidation ingress on the coated surface allowing for debonding between the fiber/BN interface or the BN/matrix interface leading to longer creep life. Ultimately however, the exposed rear surface once again appears to be the main source of oxidation ingress due to its increased stress state and lack of coating.
- The isothermally tested specimen exhibited the highest level of fiber pullout indicative of higher creep tolerance. This phenomenon once again demonstrates the detrimental effect of thermal gradients on creep properties.
- The coupled ER measurements appear to be sensitive to both temperature increase as well as increased damage state of the material leading to confidence in its use as a non-destructive evaluation and damage modeling technique.
- Further investigation into failure mechanism and environmental contributions to premature failure is necessary in order for adequate understanding of material failure modes.

Acknowledgements

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