Correlation of electrical resistance to CMC stress-strain and fracture behavior under high heat-flux thermal and stress gradients

Matthew Appleby* †, Gregory Morscher*, Dongming Zhu†

*The University of Akron, Akron OH
† NASA Glenn Research Center, Cleveland OH

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Electrical Resistance (ER) monitoring has been shown as a tool for detecting room temperature tensile damage accumulation in woven melt-infiltrated (MI) SiC/SiC CMCs. However, under stress gradients and complex thermo-mechanical loading, mechanisms controlling contributions to ER change are still not well understood.

In this study, experiments were chosen to explore the capabilities of ER as a relevant non-destructive evaluation (NDE) technique for testing under high-temperature thermal gradient conditions, with stress concentrations.

To overcome the disadvantage of far-field in-situ ER measurement, notched samples provided a highly localized strain field in gage section for ER correlation.

Acoustic Emission (AE) measurements and Digital Image Correlation (DIC) were also performed to correlate with ER results.
Experimental Material

- SiC/SiC CMC material (Hyper-Therm HTC)
  - 8 plies, balanced 5 harness satin 2D woven 0°/90°, SiC/BN/SiC
  - Hi-Nicalon Type-S fiber reinforced
  - Produced by molten silicon melt infiltration (MI)
  - Machined into 6 in. tensile bars

Room Temperature
- SINGLE NOTCH
  - w = 12.74 mm (10.77 mm)
  - t = 2.17 mm
  - \( f_0 = 0.147 \)

High Temperature
- DOUBLE NOTCH
  - w = 12.82 mm (10.25 mm)
  - t = 2.27 mm
  - \( f_0 = 0.141 \)
Experimental Technique

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

High temperature testing:
- Face of specimen gage-section heated by a 3.5kW CO₂ 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
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

Post-test ER “inspection” is performed by taking discrete measurements along the specimen length
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

$$x = \frac{v}{2}(t_{\text{bottom}} - t_{\text{top}})$$

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-test microscopy revealed high crack densities in the vicinity of the notch, as would be expected from locally increased stress.

However, due to a large density of porosity, another localized strain field was produced, leading to the ultimate failure location.

Crack densities were therefore calculated for the notched region and the far-field regions:

<table>
<thead>
<tr>
<th>Region</th>
<th>Crack Density, mm(^{-1})</th>
<th>(far-field/notch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-12.7mm &lt; x &lt; -5mm</td>
<td>2.1</td>
<td>(0.85)</td>
</tr>
<tr>
<td>± 5mm</td>
<td>2.5</td>
<td>1</td>
</tr>
<tr>
<td>5mm &lt; x &lt; 12.7mm</td>
<td>1.70</td>
<td>(0.68)</td>
</tr>
</tbody>
</table>
Damage Location

- Damage location determined from AE events in good agreement with DIC strain-field mapping of gage section at peak load

\[ \sigma = 324 \text{ MPa} \]
Acoustic energy has been shown to be directly related to transverse matrix cracking in CMCs.

AE events separated by each region.

The cumulative energy was normalized in all regions by the total energy recorded in the notched region (± 5mm from mid-plane of specimen).

The area around the notch shows increasing damage at lower stress and higher total energy released; which is in good agreement with AE/DIC location analysis.
In-situ electrical behavior

- While the elastic region shows little increase, ER begins to increase rapidly with damage onset.
- The electrical resistance of the entire specimen increases over 500% with an increase in nominal strain of the gage section of 0.5% at failure.
- ER is monitored over the entire specimen showing both the contribution to localized and far-field damage (including all matrix crack and fiber sliding)
- However, as previously noted, the damage accumulation is being dominated by the stress concentration region
In order to determine the contribution of ER increase from the gage region vs. the rest of the specimen, discrete ER measurements were taken along the length of the sample for the “pristine” and “post fracture” cases.

As expected, the results show that the highest ER increase corresponds to the area of increased damage in the gage section.
High Temperature – Double Notch

- Laser-heated gage section
  \[ T_{\text{surface}} = 1230^\circ \text{C} \]
  \[ T_{\text{back}} = 1000^\circ \text{C} \]

- 0.53% nominal strain increase from thermal loading, with an addition 0.29% nominal mechanical strain to failure

- Specimen failed along notch plane, with post-test microscopy showing increased crack density in notch region

- Specimen exhibits far less damage accumulation than room temperature (single notch) specimen

<table>
<thead>
<tr>
<th></th>
<th>± 5mm</th>
<th>± 5mm &lt; x &lt; ± 12.7mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crack Density, mm⁻¹</td>
<td>1.0</td>
<td>0.81</td>
</tr>
<tr>
<td>(far-field/notch)</td>
<td>1</td>
<td>(0.81)</td>
</tr>
</tbody>
</table>

Fracture at mid-plane
Once again damage location determined from AE events in good agreement with DIC strain-field mapping of gage section at peak load.

At low stresses, AE activity occurs only in the notch region (± 5mm), while only becoming significant in the far-field above 150 MPa.
Similar to the room temperature specimen the area around the notch sees an increase in AE energy at a lower load level.

However, this sample does not reach matrix crack saturation, leading to the decreased values in matrix crack density in both the notched and far-field regions (as compared to room temp. test).

Normalizing by their respective lengths you can see that the region outside the notch has a lower energy density (73% of notch region), which is in good agreement with the reported crack density measurements.
Notch strain decomposition

- In an effort to further investigate the contribution of the stress concentration region surrounding the notch area with ER increase, the increased strain fields in the notch region were isolated (based on color thresholds) using image analysis software.

- The area fractions of each of these strain values is then used to determine the weighted average of elevated strain in the notch region for a given applied stress.

\[ \sigma = 240 \text{ MPa, } \varepsilon_{\text{notch}} = A_f(i) \times \varepsilon(i) = 1.05\% \]
High Temperature In-situ ER behavior

- **Notch strain decomposition** analysis was performed on images of 103, 132, 151, 172, 200, 217, 226, and 240 MPa (net-section stress) respectively.

The values of increased strain in the notch area appears to correlate well with the increase in total ER of the sample; suggesting notch area ER dominance.
Room temperature inspection of high-temperature tests

The post high-temperature fast-fracture ER inspection analysis did not demonstrate the highest ER increase around the notch region as expected

This seemingly anomalous behavior was further examined by observing the ER response to laser-heating only (i.e. no mechanical loading) to similar temperatures
Post-heat only data shows an increase in electrical conductivity of the heated zone upon cooling back to room temperature.

Clearly more needs to be understood about high temperature microstructural changes in the matrix (relief of residual stress, diffusion of impurities, etc.).

Competing factors between high temperature exposure and damage could lead to good area for further development.
Conclusions

- AE and DIC in good agreement with location of damage zones generated from localized stress concentrations seen in post-test microscopy.

- ER measurement shown to be an effective tool for **in-situ damage monitoring** of MI SiC/SiC CMCs with stress concentrations at both *room temperature* and 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).

- Post-test ER measurements of room temperature sample indicated usefulness of ER as inspection technique.
  - Highest ER changes seen in areas of increased crack densities.
  - In agreement with elevated damage regions shown by AE event locations and DIC strain mapping.
  - Could be useful technique for component level damage inspection.

- Post-test inspection for high temperature samples however, requires better understanding of change in room temperature ER after high temperature exposure.
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Questions?