Detecting Damage in Ceramic Matrix Composites Using Electrical Resistance

ABSTRACT
The majority of damage in SiC/SiC ceramic matrix composites subjected to monotonic tensile loads is in the form of distributed matrix cracks. These cracks initiate near stress concentrations, such as 90° fiber tows or large matrix pores and continue to accumulate with additional stress until matrix crack saturation is achieved. Such damage is difficult to detect with conventional nondestructive evaluation techniques (immersion ultrasonics, x-ray, etc.). Monitoring a specimen's electrical resistance change provides an indirect approach for monitoring matrix crack density. Sylramic-iBN fiber-reinforced SiC composites with a melt infiltrated (MI) matrix were tensile tested at room temperature. Results showed an increase in resistance of more than 500% prior to fracture, which can be detected either in situ or post-damage. A relationship between resistance change and matrix crack density was also determined.
Detecting Damage in Ceramic Matrix Composites Using Electrical Resistance

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Why electrical resistance?

- Small scale, transverse, in-plane, matrix cracks are the initial form of damage to develop over long-time creep and fatigue conditions.
- Ultrasound, x-ray, and thermal imaging are limited in their ability to quantify such cracking.
- Electrical resistance of SiC/SiC composites is one technique that shows special promise towards this end.
  - Both the matrix and the fibers are semi-conductive.
  - Changes in matrix or fiber properties should relate to changes in electrical conductivity along the length of a specimen or part, i.e., perpendicular to the direction of damage.
Previous work on resistance of SiC/SiC

- Many journal papers available for fiber-reinforced polymers
- Few available for CMC
- Our previous work has focused on CVI SiC/SiC
  - Room temp tensile
    - (Smith, Morscher, Xia; Scripta Mater. 2008)
  - Elevated temp creep
    - (Smith, Morscher, Xia; Int. J. Applied Ceram. Tech 2010)
  - Interrupted creep
    - (Smith, Morscher, Xia; Int. J. Applied Ceram. Tech 2010)
- Results showed feasibility of using resistance for NDE of CVI SiC/SIC
- Now, we look at MI matrix
Objective

- Many variables contribute to resistance
- Need to determine the contribution of each mechanism to the overall resistance for modeling purposes
- One panel of consistent material
  - Samples subjected to varied environments and loading
  - Here, results are given for room temperature samples
- Main goal: Determine how sensitive resistance change is to matrix cracks
What affects electrical resistance?

- **The content and structure of composite**
  - Constituents (fiber, interphase and matrix) and their relative resistances
  - Nature and amount of porosity
  - Fiber architecture

- **Temperature**

- **Stress**

- **The damage state**
  - Transverse and/or interlaminar cracking

- **The oxidation and/or recession state**

- **Lead attachment** – on the face, on an edge, an extension from within the structure?
Procedure

- 150 mm long samples with contoured gage section
- Load, unload, and reload in tension on an Instron Universal Testing Machine (4kN/min)
- Capacitance strain gage used with 1% range over 25mm
- Resistance monitored with Agilent 34420A micro-Ohm meter
- Acoustic emission monitored by 50kHz to 2MHz sensors just outside the gage section
Specimen Configuration

- Silver paint on surface for lower contact resistance
- Grip region wrapped with copper mesh
  - Evenly distributes load
  - Used as electrical contact
- Resistance measured by four-point probe method
- Gripped with metal wedge grips covered with polymer sheet (for electrical insulation)
Procedure

- 3 samples (from the same panel)
  - Sample 1: Load- unload- reload hysteresis to failure
  - Sample 2: Load to 5 kN
    - 1/3 of total AE energy
    - Interrupt and polish
  - Sample 3: Load to 6 kN
    - 2/3 of total AE energy
    - Interrupt and polish

- All samples polished and plasma etched to examine cracks
Room Temperature Damage Characterization

*Syl-iBN/MI Matrix Woven Composite (f = 0.38)*

As damage progresses, the resistance of the composite increases
- 500% increase in resistance at failure
- **In situ damage detection is possible**

Resistance is permanently affected (never returns to zero)
- **Inspection of damage is possible after unloading**

**SAMPLE 1**
0.26 Ω-mm initially

As $\sigma \uparrow$, $R \uparrow$ due to:
- Matrix cracking (follows AE)
- Fiber breaks (at high stresses)
- Piezoresistivity of fibers (small effect)
Room Temperature Damage Characterization

**Syl-iBN/MI Matrix Woven Composite** (f = 0.38)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Max Stress, MPa</th>
<th>Stiffness, GPa</th>
<th>Initial Resistivity, Ω-mm</th>
<th>Resistance change, %</th>
<th>Etched Crack Density, mm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>430</td>
<td>323</td>
<td>0.260</td>
<td>504</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>247</td>
<td>246</td>
<td>0.267</td>
<td>17.9</td>
<td>0.9</td>
</tr>
<tr>
<td>3</td>
<td>300</td>
<td>237</td>
<td>0.251</td>
<td>78.5</td>
<td>3</td>
</tr>
</tbody>
</table>

Note: all samples have eight plies with a BN interphase, 800 fibers per tow, and total fiber volume fraction of 0.38

![Graph showing stress-strain relationship and AE energy vs strain]
Resistance change related to cracking

- Resistance change vs. crack density is parabolic relationship
- Higher sensitivity at high stress (cracks and fiber breaks)

\[ y = 3.55x^2 + 14.80x + 1.20 \]
\[ R^2 = 1.00 \]

- AE energy is proportional to crack density (Morscher, comp. Sci. 64)
- Measured crack density is shifted below predicted
Acousto-Ultrasonic (AU) Scanner

- Received signal analyzed using 18 parameters (Freq. and time based)
- Correlate certain parameters to distributed damage (AU developed/optimized for composites)
Sample AU Results

Transducers (1.25 MHz, broadband)

Main AU scan area (dashed line, with sub-areas defined by dotted lines) and result screen.

### Table: AU Results for Specimens 2 and 3

<table>
<thead>
<tr>
<th></th>
<th>Specimen 2</th>
<th>Specimen 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before test</td>
<td>After 247 MPa</td>
</tr>
<tr>
<td>Mean square value, V^2</td>
<td>0.014 (0.007)</td>
<td>0.0031 (0.0014)</td>
</tr>
<tr>
<td>Centroid, MHz</td>
<td>0.349 (0.010)</td>
<td>0.350 (0.015)</td>
</tr>
<tr>
<td>Diffuse field decay rate, V^2/µsec</td>
<td>0.011 (0.005)</td>
<td>0.015 (0.005)</td>
</tr>
</tbody>
</table>

*Data represents the average for the overall scan area (standard deviations indicated within parenthesis).
Most consistent results were based on the mean square value of the power spectrum
- Parameter represents the energy of the received ultrasonic signal (area under the power spectrum)

Scanning AU technique (especially the MSV term) was very sensitive to early matrix cracks
- Damage increased material’s attenuation, thereby reducing MSV

Greater than 70% reduction of MSV observed after damage inducing loads

Earlier study:
- In-situ monitoring during damage inducing tensile tests showed that large changes occurred early on due to both micro-cracking and transverse cracks
- Values stayed constant until near failure, fiber fractures caused further changes
- Plateau was reached before transverse crack saturation and fairly early in respect to the AE accumulated energy plots
- Similar behavior here, as witnessed by the indifference to the specific crack counts

To fully appreciate the capabilities of the new scan system, additional points of interruption are needed
Summary and Conclusions

- Electrical resistance in SiC-based CMCs is very sensitive to stress/strain history
- Electrical resistance offers a useful way to characterize SiC-based CMCs
- This technique offers potential as
  - a method of quality control for processing these composites
  - a method to monitor the health of SiC-based CMC components
    - in-situ or as an inspection technique
    - Can then be extended to Life-prediction models
- Resistance shows parabolic relationship with cracking at room temperature
Future Work

- Quantify the effect of high temperature microstructural changes on resistance
  - Sensitivity to: crack formation, fiber breaks, oxidation, creep
- Compare different composites – varying composite constituents and fiber architecture
- Determine lead attachment schemes for different applications and conditions