Ceramic matrix composites (CMC) are suitable for high temperature structural applications such as turbine airfoils and hypersonic thermal protection systems due to their low density high thermal conductivity. The employment of these materials in such applications is limited by the ability to accurately monitor and predict damage evolution. Current nondestructive methods such as ultrasound, x-ray, and thermal imaging are limited in their ability to quantify small scale, transverse, in-plane, matrix cracks developed over long-time creep and fatigue conditions. CMC is a multifunctional material in which the damage is coupled with the material’s electrical resistance, providing the possibility of real-time information about the damage state through monitoring of resistance. Here, resistance measurement of SiC/SiC composites under mechanical load at both room temperature monotonic and high temperature creep conditions, coupled with a modal acoustic emission technique, can relate the effects of temperature, strain, matrix cracks, fiber breaks, and oxidation to the change in electrical resistance. A multiscale model can in turn be developed for life prediction of in-service composites, based on electrical resistance methods. Results of tensile mechanical testing of SiC/SiC composites at room and high temperatures will be discussed. Data relating electrical resistivity to composite constituent content, fiber architecture, temperature, matrix crack formation, and oxidation will be explained, along with progress in modeling such properties.
Electrical Resistance of SiC/SiC Ceramic Matrix Composites for Damage Detection and Life-Prediction

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Why Electrical Resistance?

• Current nondestructive methods such as ultrasound, x-ray, and thermal imaging are limited in their ability to quantify small scale, transverse, in-plane, matrix cracks developed over long-time creep and fatigue conditions

• 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
  – Resistance has been shown to be effective at monitoring damage in composites such as C/SiC and CFRP
Objective

• To determine if and to what extent electrical resistance can be used as a self-sensing non-destructive evaluation technique for SiC-based fiber-reinforced composites
  – Composite characterization
  – Damage accumulation
    • Analysis technique
    • Monitoring as an inspection or possibly on-board device
  – Electro/Mechanical Modeling ➔ Performance and Life-modeling
Some studies in the literature
(mostly w/C fibers in epoxy, glass, or CVI SiC)

- Many papers last 20 years on C fiber reinforced polymer, concrete and carbon matrix composites
  - Strain and damage monitoring
- Recent measurement of conductivity of SiC/SiC for nuclear applications [e.g., Gelles & Youngblood (PNL); Shinavski, Katoh, and Snead, (Hyper-Therm & ORNL)]
What affects electrical resistivity?

\[ \rho = \text{Resistance} \cdot (\text{Area}/\text{Length}) \]

- The content and structure of composite
  - Constituents (fiber, interphase and matrix) and their relative resistivities
  - Nature and amount of porosity
  - Fiber architecture
- Temperature
- Stress
- The damage state
  - Already present (e.g., C/SiC) or as a result of stressed-oxidation (SiC/SiC)?
  - 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?
Room Temperature Damage Characterization

- 150mm specimens with contoured gage section
- Resistance measured by four-point probe method using an Agilent 34420 micro-Ohm meter
- Conductive silver paste was used to improve contact between specimen and voltmeter

\[ \text{Constant current} \]
\[ \text{dV} \]
Room Temperature Damage Characterization

- Load, unload, and reload in tension on an Instron Universal Testing Machine (4kN/min)
- Capacitance strain gage used with 1% range over 25mm (metal knife-edge contact extensometers were tried, but abandoned because of electrical interference)
- Resistance measurement made every second
- Acoustic emission monitored by 50kHz to 2MHz sensors just outside the gage section
Room Temperature Damage Characterization

**Syl-iBN/CVI Matrix Woven Composite (f = 0.3)**

As damage progresses, the resistivity of the composite increases
- 60% increase in resistivity at failure
- an order of magnitude higher than C fiber reinforced systems
- In situ damage detection is possible

- Upon unloading, resistivity does not return to original
  - Inspection of damage is possible after unloading

As $\sigma \uparrow$, $R \uparrow$ due to:
- Matrix cracking (follows AE)
- Piezoresistivity of fibers
- Fiber breaks

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Smith, Xia, Morscher, *Scripta Mater.* 2008
- Copper wire leads brazed (CuSil-ABA) on face and ends of specimen
- Resistance measured by four-point probe method
- Stress-rupture at 1315°C in air

Elevated Temperature Set-up

Constant Current Applied

Hot Zone

Furnace Region

Epoxy Grip Tab

Epoxy Grip Tab

$\text{dV}$
Elevated Temperature Set-Up
Elevated Temperature Damage Characterization

- Determine effects of temperature and time on resistivity:

\[ \text{Resistivity, } \Omega \text{-m} \]

\[ \text{Time, hr} \]

\[ \text{Temp, } ^\circ\text{C} \]

- Two hour hold at 1315\(^\circ\text{C}\): NO CHANGE IN RESISTIVITY
Elevated Temperature Procedure

EXPERIMENT:

• Raise furnace temperature to 1315°C
  – Resistivity decreases with temperature (SiC)

• Raise stress to desired level
  – Resistivity increases with stress

• Hold stress until failure
  – Resistivity increases with time at stress
Elevated Temperature Damage Characterization

Syl-CVI SiC (Vendor 1); 1315C; 86 MPa

Syl-iBN-CVI SiC (Vendor 2); 1315C; 172 MPa

What is occurring?
- Matrix cracking & growth
- Creep of matrix
  - Load transfer to fibers
- Oxidation in matrix cracks
- Fiber breaks

(0.041Ω-m) (0.0188Ω-m)
In principle, as unbridged cracks form and oxidizing species fill matrix cracks and/or pores and/or oxidation reactions cause recession of composite, resistance changes should occur. If they can be quantified, then this technique offers a way of health monitoring.
Coupled Electrical/Mechanical Model at Tow Level

Electrical Model:
Local resistances

Resistance vs.
stress/strain/damage

Mechanical Model:
Damage, local stresses

Stress vs. strain, failure

Random Fiber-Fiber contacts in composites

• Electric characteristic length:
The average distance between electrical contacts

\[ \delta_{ce} = \frac{1}{f_c} = \frac{LN}{2N_c} \]

- \( f_c \) = fiber contact density
- \( N_c \) = the number of fiber contacts
- \( N \) = the number of fibers
- \( L \) = length of composites

An analogy to mechanical characteristic length slip length \( \delta_c : \delta_c = r \sigma_c / \tau_y \)

\( \delta_{ce} \) is measureable:

\[ \beta \delta_{ce} = d \sqrt{\frac{\rho_T}{\rho_L}} \frac{\pi}{4V_f} \]

- \( \rho \) ------ Resistivity
- \( \beta \) ------ geometric contact factor
- \( d \) ---- diameter of fiber
- \( V_f \) ---- volume fraction of fiber
**3D Electrical/Mechanical Models**

**Electrical Model**

Current equilibrium:

\[ I_1 + I_2 + I_3 + I_4 + I_5 + I_6 + I_7 + I_8 = 0 \]

*From neighbors (contact current)*

\[ I = (v_{i+1} - v_i)/R_i \]

**Mechanical model**

Force equilibrium:

\[ F_1 + F_2 + F_3 + F_4 + F_5 + F_6 + F_7 + F_8 = 0 \]

*From neighbors (shear forces)*

Axial Force: \[ F = AE(u_{i+1} - 2u_i + u_{i-1})/\delta \]

Shear force: \[ F = hG(u_i - u_{in})/d \]
Coupled Model at Laminate Scale

Unit Cell model:
- Single fiber bundle surrounded by matrix
- Through-thickness matrix cracks bridged by fiber bundles
- Matrix containing 90° fiber are conductive

Fiber tow
Conductive matrix

Electrical Model
Mechanical model

Matrix crack

σ
σ
Mechanical Behavior of SiC/SiC Composites

Fitting stress-strain curve by adjusting matrix Weibull strength $\sigma_0$ and interfacial shear strength $\tau$

- $\tau=25$ MPa, with the typical experimental range.
- Predicted matrix crack density curve well matches cumulative AE energy.

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By varying electrical parameters, the model should fit experimental data, but there is an unknown problem.
Summary and Conclusions

- Electrical resistance in SiC-based CMCs is very sensitive to constituent content, fiber-architecture, and stress/strain history
- Electrical resistance offers a useful way to characterize SiC-based CMCs, both as-produced and after mechanical damage
- 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
    - which can then be related to life-prediction models based on stress, time, and damage accumulation and their relationship to electrical response
Future Work

- Compare different composites – varying composite constituents and fiber architecture
- Quantify elevated temperature microstructural change with resistivity change
- Determine lead attachment schemes for different applications and conditions
- Extend electro/mechanical model to:
  - Include high temperature properties
  - Include 90° tows