Electrical Resistance Technique to Monitor SiC Composite Detection

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Abstract

Ceramic matrix composites are suitable for high temperature structural applications such as turbine airfoils and hypersonic thermal protection systems. The employment of these materials in such applications is limited by the ability to process components reliable and to accurately monitor and predict damage evolution that leads to failure under stressed-oxidation conditions. 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. Since both the matrix and the fibers are conductive, changes in matrix or fiber properties should relate to changes in electrical conductivity along the length of a specimen or part. The effect of matrix cracking on electrical resistivity for several composite systems will be presented and some initial measurements performed at elevated temperatures under stress-rupture conditions. The implications towards electrical resistance as a technique applied to composite processing, damage detection (health monitoring), and life-modeling will be discussed.
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-based 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.
  - Ideal for:
    - Characterization: different fiber-type, fiber architecture, interphase, and matrix.
    - Structural diagnostic tool.
    - Extension to self-sensing capability.
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 effects electrical resistivity?

\[ \rho = \text{Resistance} \cdot \left( \frac{\text{Area}}{\text{Length}} \right) \]

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

Fibers broken in some cracks
Experimental

- 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
Application I: Composite Characterization

The relative resistivities of the constituents, porosity, and fiber-architecture all play a role in the overall resistivity of the composite… which should show consistent differences when varied.

• We varied fiber-types, matrix-types, and fiber architectures…
  – Matrix Variation: 5 harness satin, 8 ply, Sylramic-iBN, BN interphase with
    • Melt-infiltrated (CVI + SiC particles + Si) matrix – NASA N24A COMPOSITE
    • CVI SiC only
    • PIP matrix (S300 Variation)
  – Fiber type, with a CVI SiC matrix…
    • Sylramic-iBN,
    • Hi-Nicalon Type S
    • Hi-Nicalon
  – Architecture, with a CVI SiC matrix…
    • Unidirectional lay-up,
    • 0/90 lay-up
    • 0/±60 lay-up
Application I: Composite Characterization

**Comparison of Matrix Types**

- 2D Five-harness satin, 8 ply lay-up
- Resistivity, Ohm-cm
- Syl-iBN MI
- Syl-iBN PIP
- Syl-iBN CVI
- GEPS GC
- COIC
- Hyper-Therm

**Comparison of Fiber Types**

- 2D Five-harness satin, 8 ply lay-up
- Resistivity, Ohm-cm
- Syl-iBN CVI
- Hi-NicS CVI
- Hi-Nic CVI

**Comparison of Architecture**

- Syl-iBN; CVI matrix; unidirectional ply lay-up
- Hyper-Therm, Inc.

- Resistivity, Ohm-cm
- Unidirectional
- [0+/−60][0 oriented]
- [0+/−60][90 oriented]
- [0/90][0 outside]
- [0/90][90 outside]
Application I: Composite Characterization (Extension to Quality Control)

• Therefore, applicable to monitoring processing of SiC/SiC composites. For example, if processing parameters can be related to resistivity (conductivity), this could be used as a quality control technique.
Application II: Damage Characterization (Room Temperature)

- 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
Application II: Damage Characterization (Room Temperature)

*Sylramic-iBN/MI 2D Woven Composite (f=0.4)*

- As damage progresses, the circuit changes for electrical conductivity, increasing the resistivity of the composite.
  - *The change is on the order of several hundred percent... this is a huge sensitivity factor!*
Application II: Damage Characterization
(Room Temperature)

*Sylramic-iBN/MI 2D Woven Composite (f=0.4)*

- Resistance at zero-stress (inspection application) very similar to AE, i.e., *matrix crack density for lower stress/strain conditions*
- Peak stress/strain resistance changes significantly with stress/strain → potential for in-situ monitoring and recognition of transient stress events

![Graphs showing normalized cumulative AE or R/Ro vs peak stress and strain history](image)
Application II: Damage Characterization
(Room Temperature)

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

- CVI matrix composite shows similar trends as MI; however the relative change in resistance with stress is about an order of magnitude less than for MI, but still an order of magnitude higher than C fiber reinforced systems
  - It should be noted, though the composite had the same number of plies as MI (8); the CVI composite was thicker $\Rightarrow f = 0.3$
Application II: Damage Characterization
(Elevated Temperature)

- Wire leads, either Pt or Cu, brazed (CuSil-ABA) on face or edge of specimen
- 2-wire and/or 4-wire connections
- Stress-rupture at 1315°C
Elevated Temperature Set-Up
EXPERIMENT:

- Raise furnace temperature to 1315°C
  - Resistivity decreases with temperature (SiC)
  - Resistivity measured every second
- Raise stress to desired level
  - Resistivity increases with stress
- Hold stress until failure
  - Resistivity increases with time at stress
Application II: Damage Characterization (Elevated Temperature)

- Determine effects of temperature and time on resistivity:

Two hour hold at 1315°C: NO CHANGE IN RESISTIVITY
So far, two different responses for two different composite-types

- Syl-CVI SiC (HyperTherm); 1315C; 120 MPa
  - Increasing resistivity with strain accumulation.
  - Transient resistivity measurement just prior to rupture.

- Increasing resistivity w/strain; Very eventful failure
So far, two different responses for two different composite-types

Syl-CVI SiC (GE); 1315C; 86 MPa

- Increasing resistivity w/strain;
- Non-eventful failure

Syl-CVI SiC (HyperTherm); 1315C; 120 MPa

- Higher conductivity composite
- Increasing resistivity w/strain;
- Very eventful failure

So far, two different responses for two different composite-types
Application II: Damage Characterization
(Extension to Time-Temperature-Stress Conditions)

• 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.
Electro/Mechanical Modeling

e.g., Z.H. Xia and W.A. Curtin, “Modeling of mechanical damage detection in CFRPs via electrical resistance”, *Comp. Sci. Tech.*, 2007

- Fibers as resistors
- “electrical ineffective length” defined by broke fibers

Next step is to model electro/mechanical performance considering conductivities of constituents and fiber architectures
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
- Develop electro/mechanical model for different composite systems relating electrical and mechanical performance with an aim toward life-modeling