Damage Characterization of EBC-SiC/SiC Ceramic Matrix Composites Under Imposed Thermal Gradient Testing

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Introduction

- Current aero-engine performance requirements necessitate light-weight materials that meet high-temperature strength and environmental durability requirements
- Melt-infiltrated (MI) SiC fiber-reinforced SiC ceramic matrix composites (**CMCs**) are a leading class of candidate materials.
- Environmental barrier coatings (**EBCs**) are applied to CMC substrates to protect Si-based components from rapid surface recession in high temp. H_2O containing environments.
- In many applications, EBC-CMC engine components will be subjected to multi-dimensional **thermal gradients** and complex stresses.

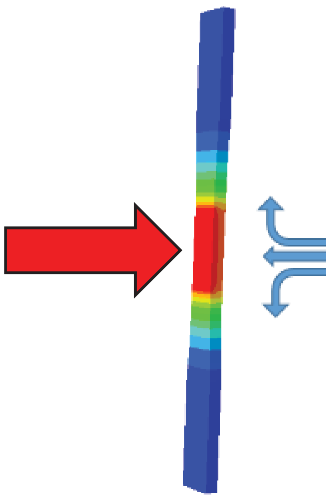
SiC/SiC vane sub-element coated with an environmental barrier coating and containing cooling holes in the trailing edge.

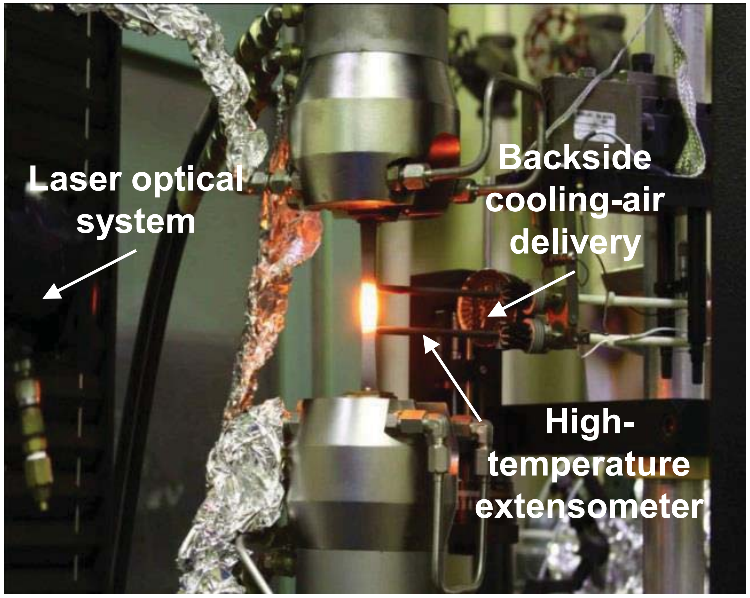
Tested SiC/SiC combustor liner from NASA's High Speed Research program

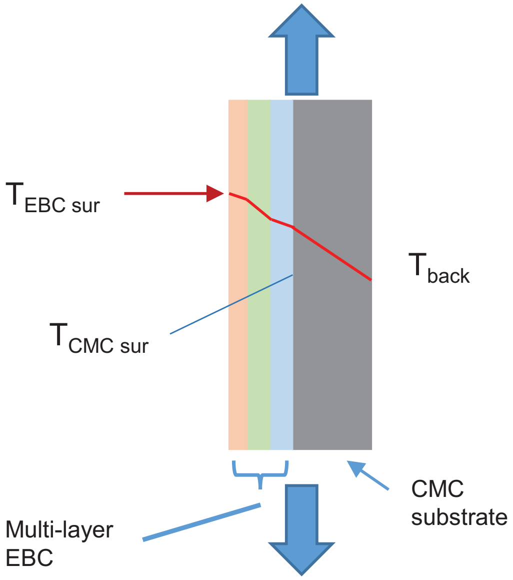
Motivation

- Desire for a simple NDE technique(s) to evaluate damage development in EBC-CMC systems during testing in thermal gradient environments
- Room-temperature (RT) tensile testing has successfully shown electrical resistance (ER) measurements as a damage monitoring technique for CMCs
- It is therefore advantageous to investigate ER response as a condition monitoring technique for testing under simulated, high-temperature, engine environments
- This study is aimed at investigating the electrical response of candidate materials under high-temperature thermal gradients in air:
    1. **Application of thermal loads**
    2. **Damage Accumulation**
    3. **Time-dependent effects**

High Heat Flux Laser-based tensile loading



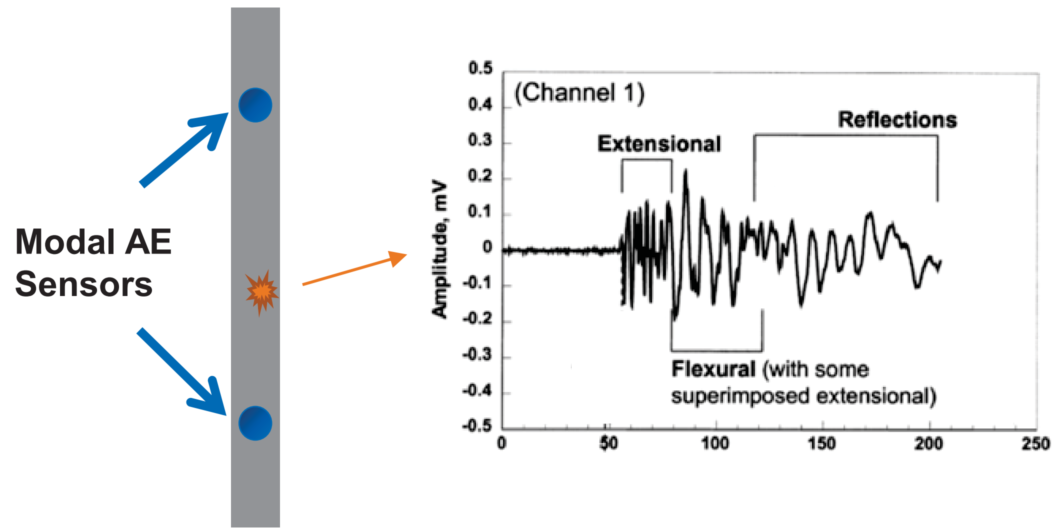


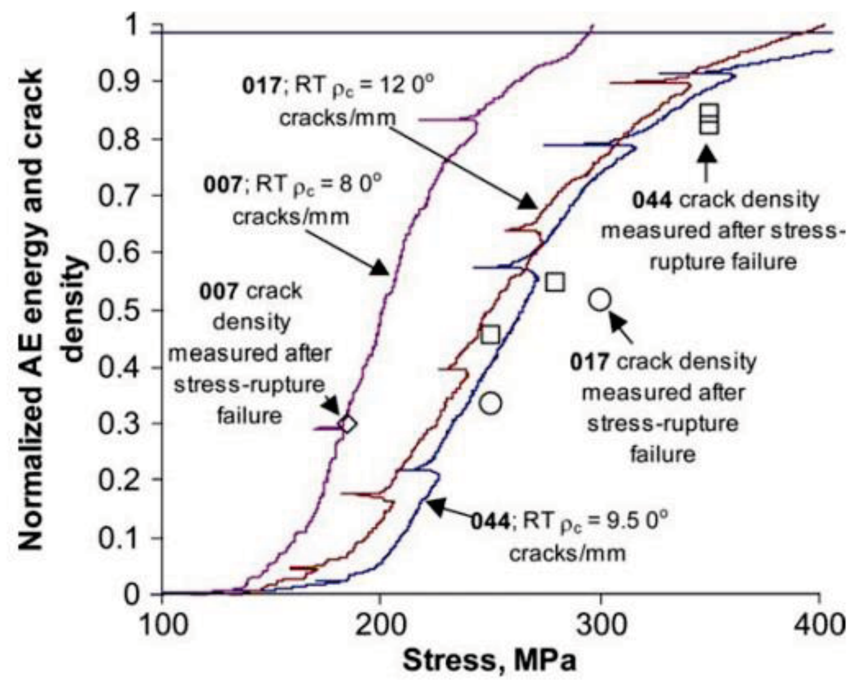


- Asymmetrical heating by a 3.5kW CO_2 high heat flux laser generates multi-axial (thru thickness and longitudinal) **thermal gradients**
- Thru thickness thermal gradients can be increased by the addition of active backside air-cooling
- The front and backside temperatures of the heated region are monitored by optical pyrometers
- The specimen is held by ceramic grip inserts in a screw driven test machine (Instron 5569)

Modal Acoustic Emission (AE) - briefly

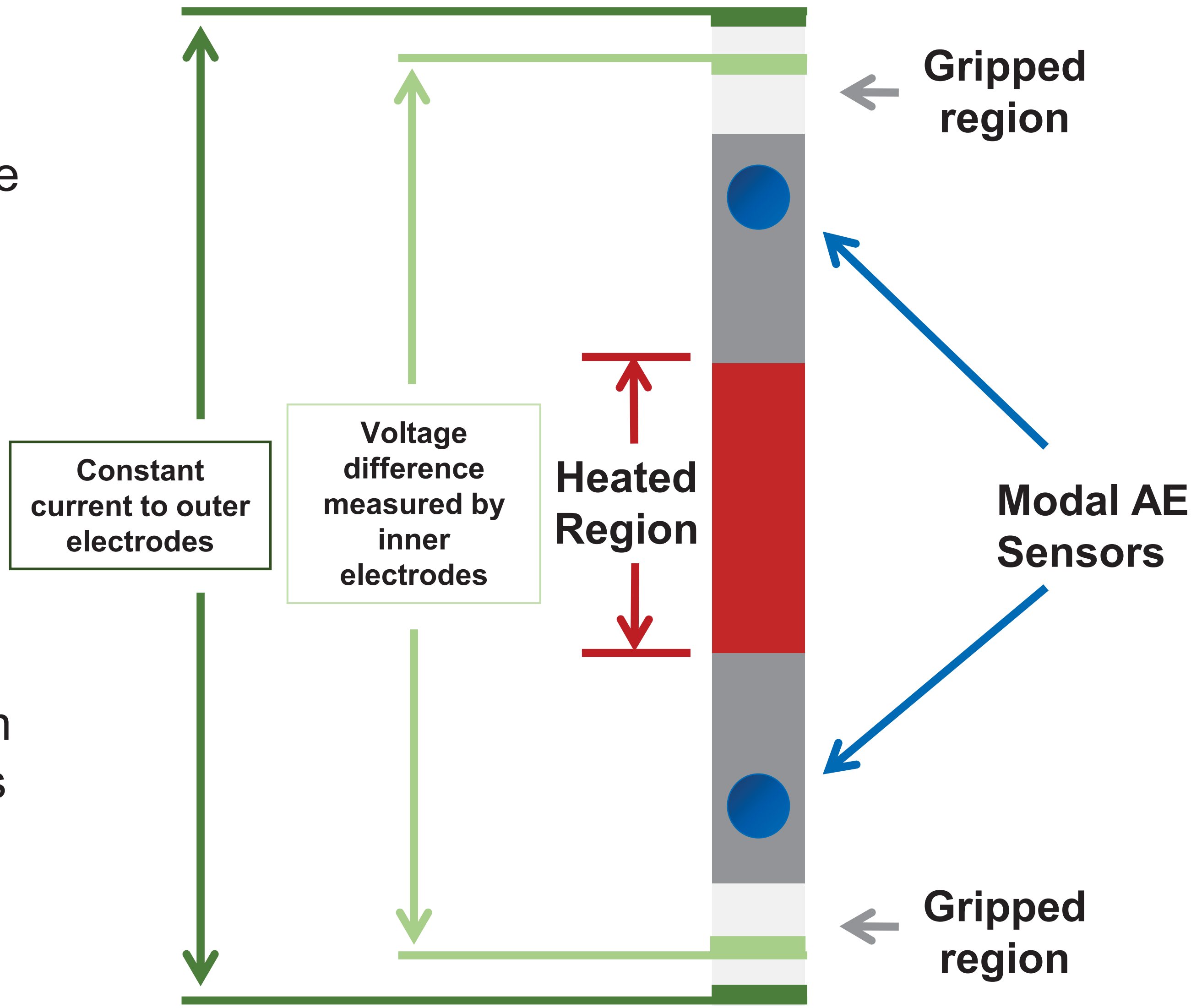
- The fracture energy of solids is released as elastic stress waves, and these waves can be captured through the use of wide-band acoustic sensors
- Accumulated acoustic energy has been shown to be directly related to transverse crack density [Morscher 2004]
  - i.e. as damage accumulates, so does the energy sensed by the acoustic sensors
- Modal AE is therefore used to characterize stress dependent crack accumulation





High Temperature ER Measurement and Setup

- ➢Electrical Resistance measured by four-point probe method (micro-Ohmmeter, Agilent Model E34420A; 10 mA constant current)
- ➢ For high temperature testing, the **ER** measurements are taken from the gripped areas
- ➢ When implemented, **AE** sensors are attached ±40mm from center (event location is determined and only events in the gage are used in AE analysis)

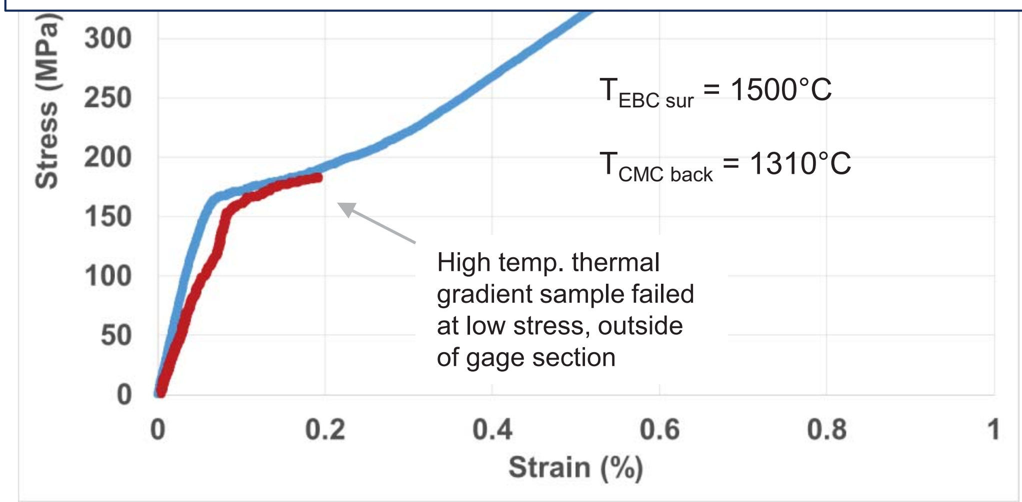


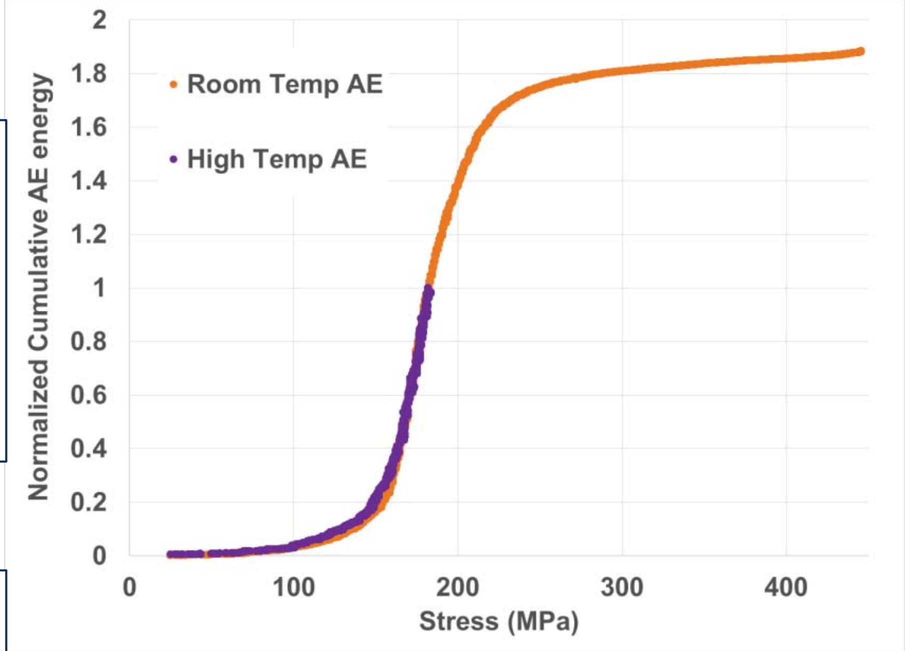
High heat flux laser-based tensile loading

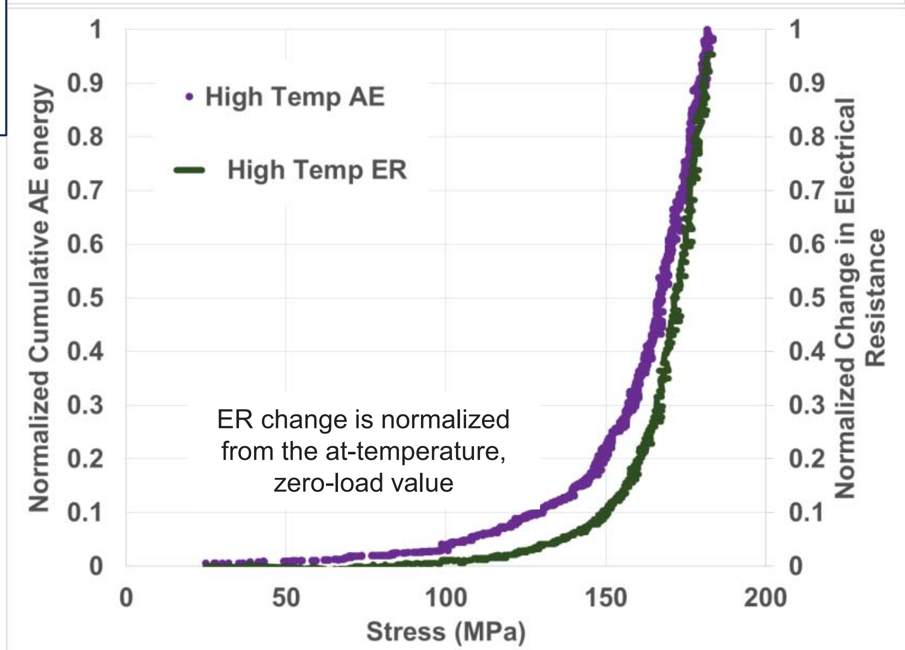
Though the high temperature thermal gradient sample failed early, the AE shows behavior similar to room temperature (RT). We can assume similar stress dependent matrix cracking behavior.

YbSi (127 µm), Hf-RE silicate (203 µm)

ER appears to be quite sensitive to damage onset and accumulation as well. The response appears to slightly lag the AE curve.



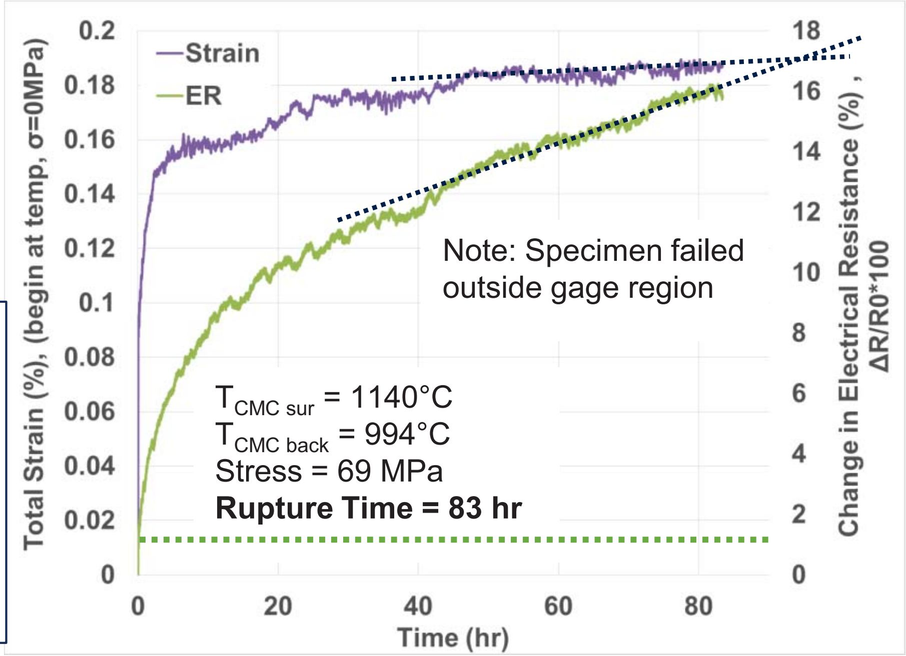


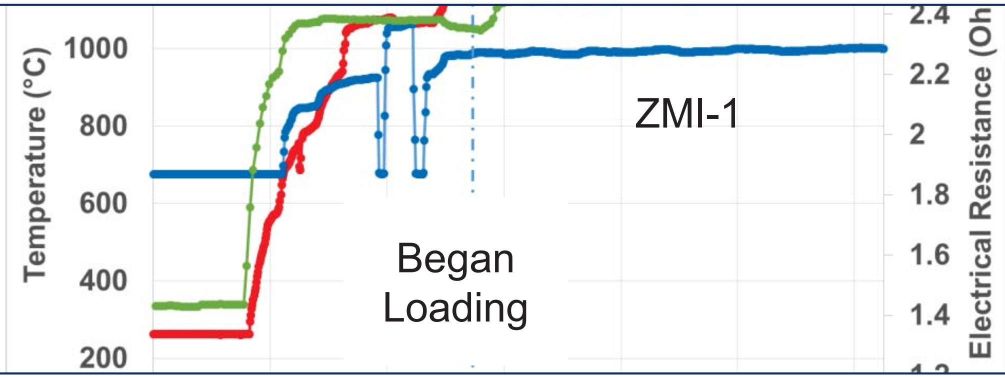


High heat flux laser-based tensile creep

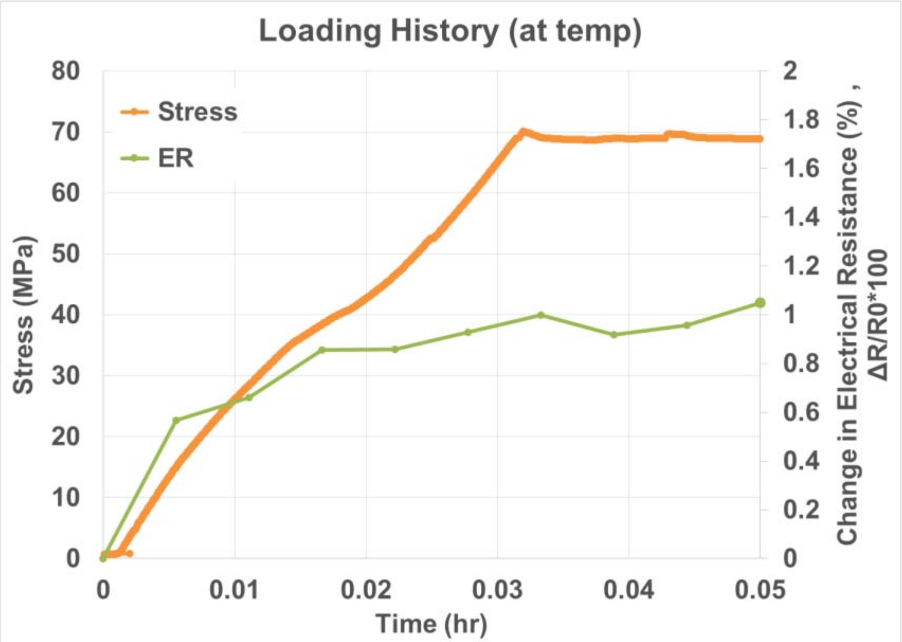
- Materials produced by Si Melt Infiltration (**MI**) process
- **Fiber Architecture**: **ZMI** fiber, 0/90 2D woven, SiC/BN/SiC
- **Uncoated**

Minor change in ER during loading suggests little damage to specimen. However, the ER does seem to be sensitive to time-dependent effects (even more so than the strain measurement). ER ~16% change over 83hr rupture time.





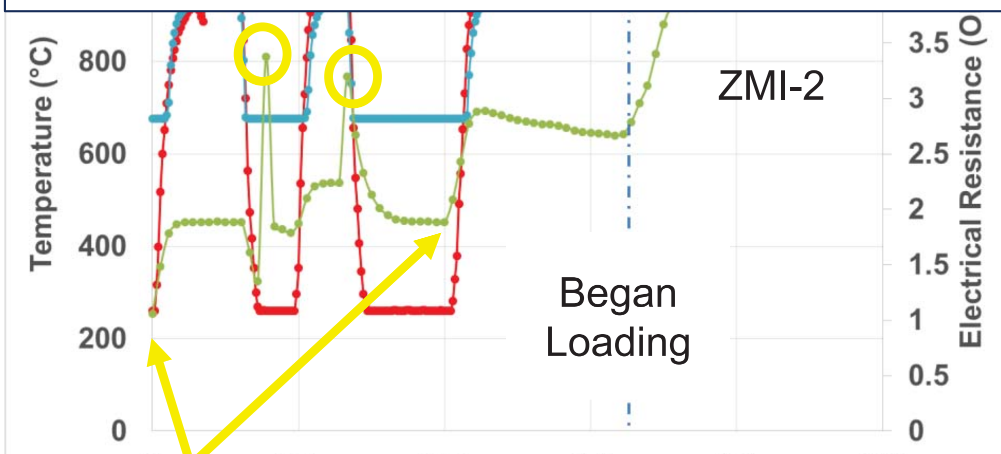
ER response during heat-up shows dependence of electrical resistivity to temperature.



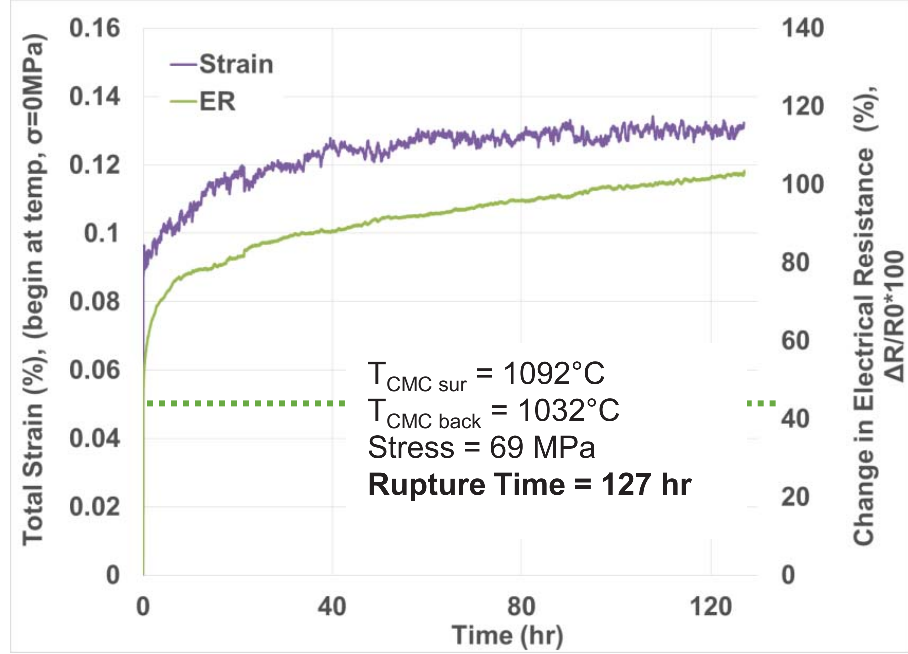
High heat flux laser-based tensile creep

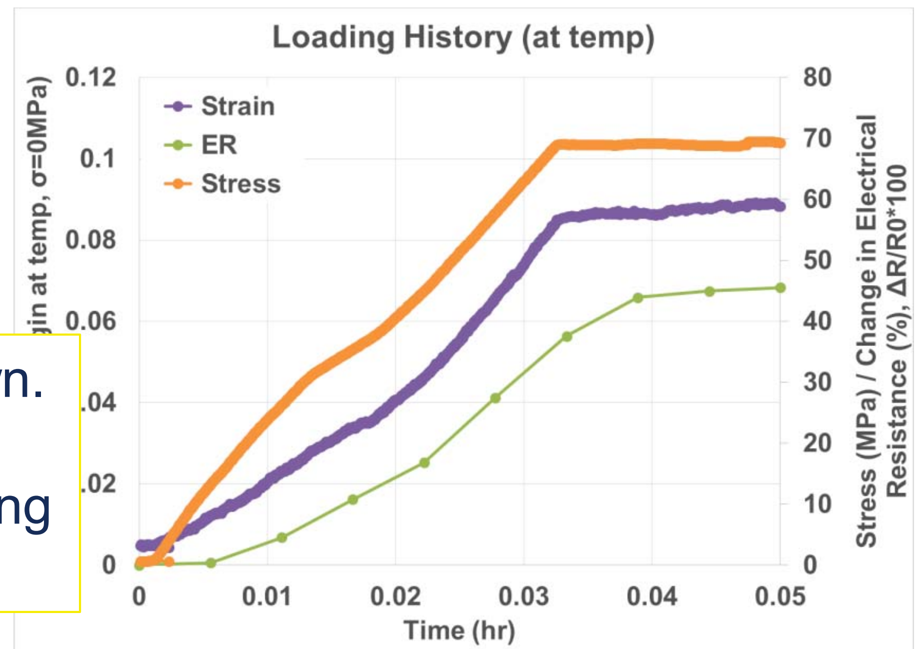
- Materials produced by Si Melt Infiltration (**MI**) process
- **Fiber Architecture**: **ZMI** fiber, 0/90 2D woven, SiC/BN/SiC
- **Uncoated**

Pre-stressed specimen showed ~45% ER increase upon loading, with an addition ~55% increase over the 127hr stress rupture time.



Laser Fault during heat up caused rapid cool down. Rig could not maintain zero load-hold condition. "Load-spike" to sample generated damage resulting in permanent increase in ER at RT (~100%)

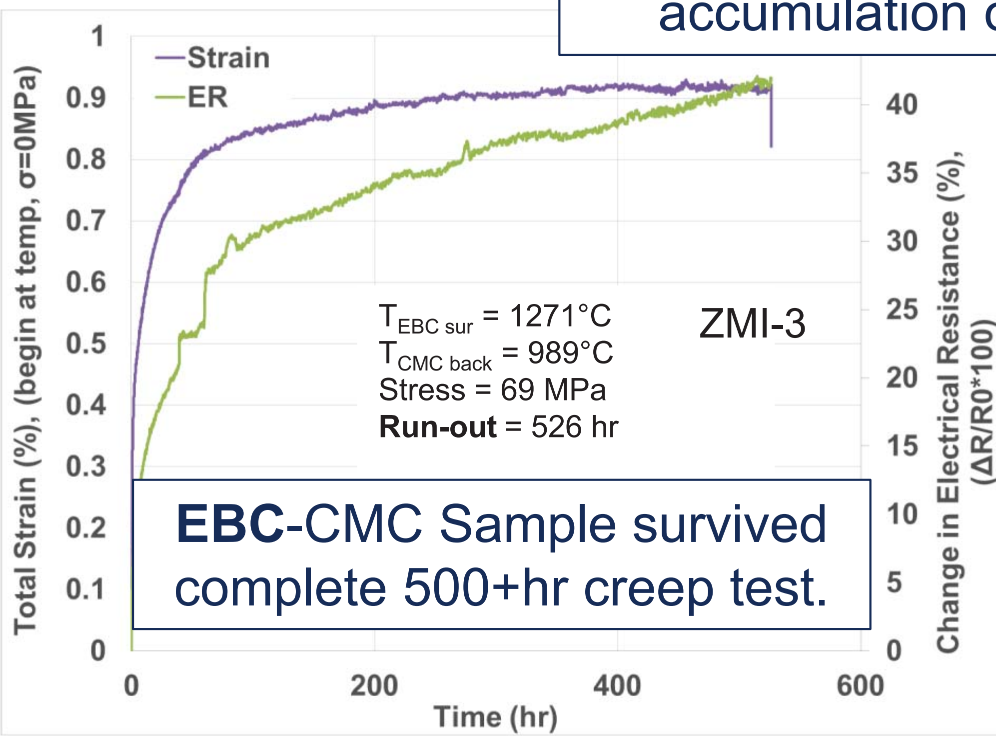




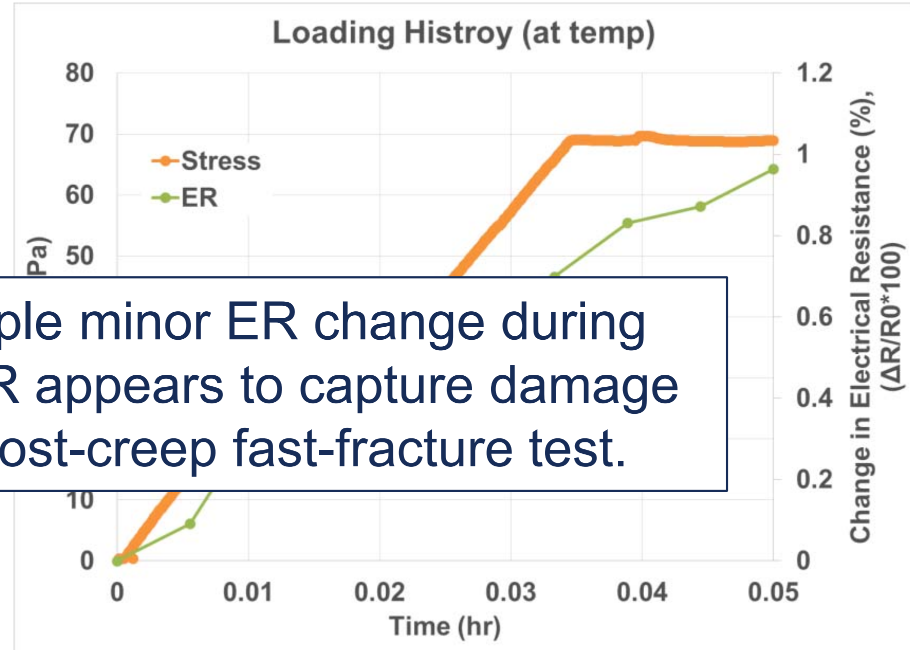
High heat flux laser-based tensile creep

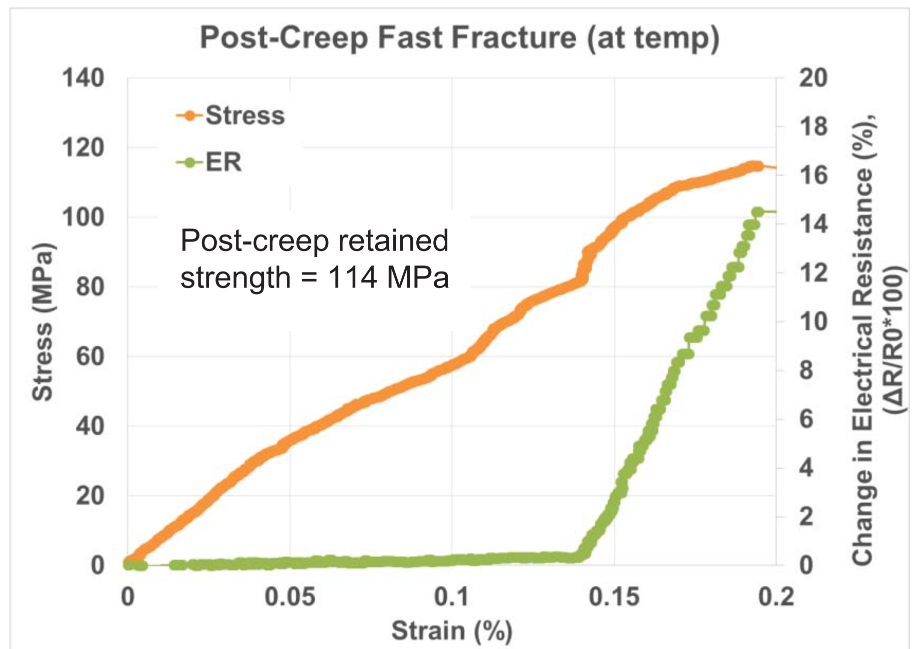
- Materials produced by Si Melt Infiltration (**MI**) process
- **Fiber Architecture**: **ZMI** fiber, 0/90 2D woven, SiC/BN/SiC
- **EBPVD EBC multi-layer syste**
  - HfO_2+Si bond coat, $Yb_2Si_2O_7$ (1

Like the first example minor ER change during loading, however ER appears to capture damage accumulation of post-creep fast-fracture test.



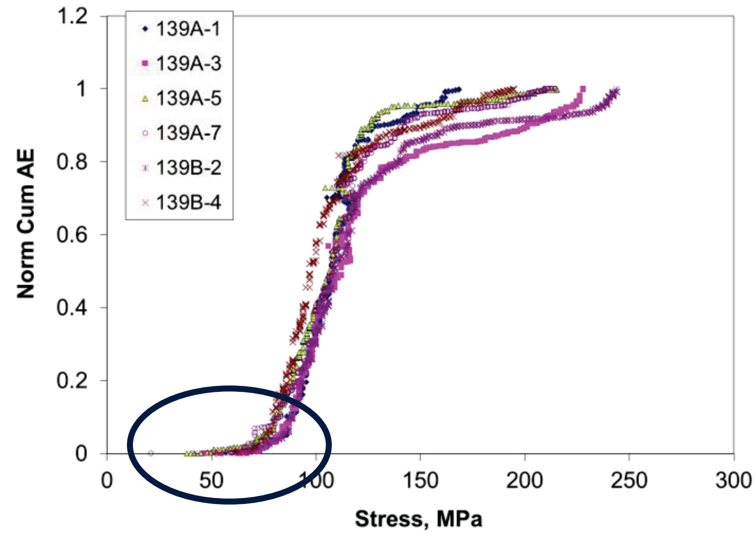
EBC-CMC Sample survived complete 500+hr creep test.

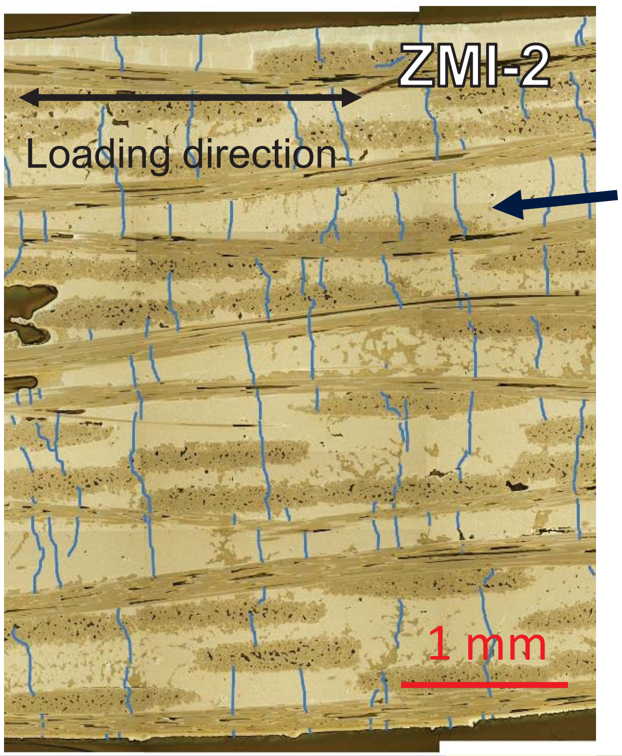




Post-test damage assessment

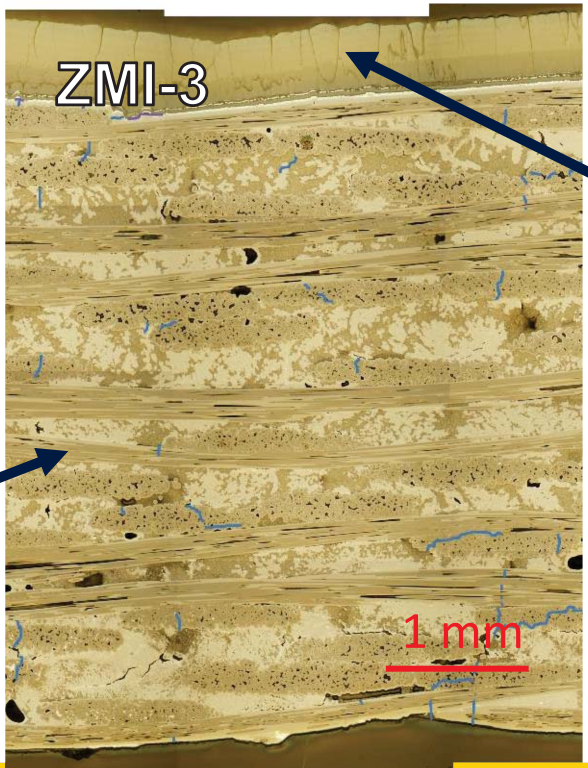
- **Recall all test performed at constant stress of 69 MPa**
- RT AE data of specimens from the same panel show microcracking initiation at ~35MPa and rapid crack accumulation beginning around 75 MPa
- Can compare crack density/morphology via post tested optical microscopy





Pre-stressed sampled showed high density of bridged matrix cracks (2.5 cracks/mm)

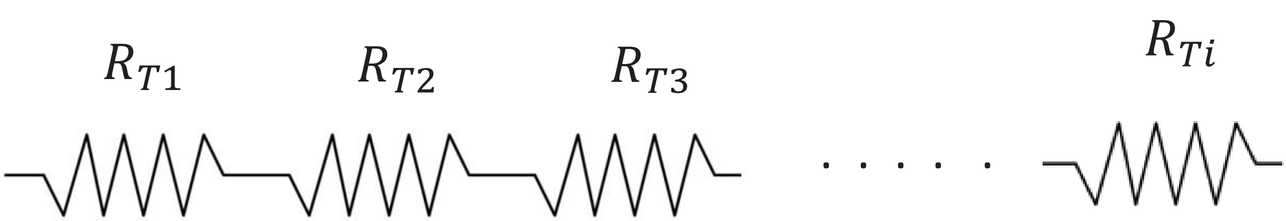
The other samples showed un-bridged cracking (0.6 cracks/mm ZMI-1, 0.45 cracks/mm ZMI-3)



EBC demonstrated good environmental durability (sintering cracks observed, but no large scale coating delamination)

Thermal-Electrical Modeling (undamaged CMC)

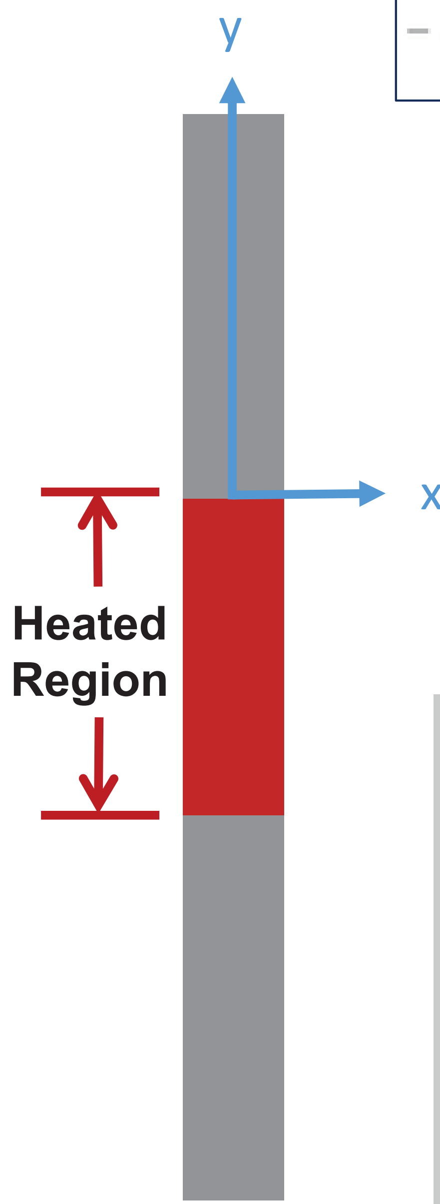
- Recall that ER measurements are taken from the gripped ends of the specimen.
- Therefore, experimental ER data vs. temperature measures the **total resistance** of the specimen under a thermal gradient
  - i.e. not directly give us discrete values of ER vs. T
- A specimen could however be described as a series of temperature dependent resistors. This circuit could then used to calculate the total resistance of a undamaged CMC specimen.

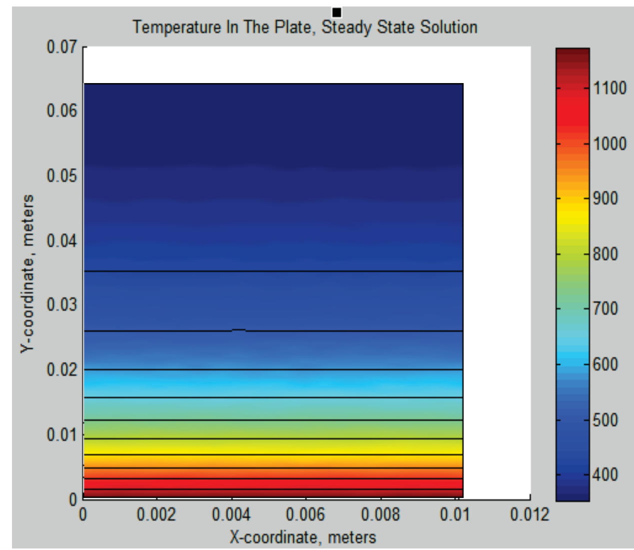


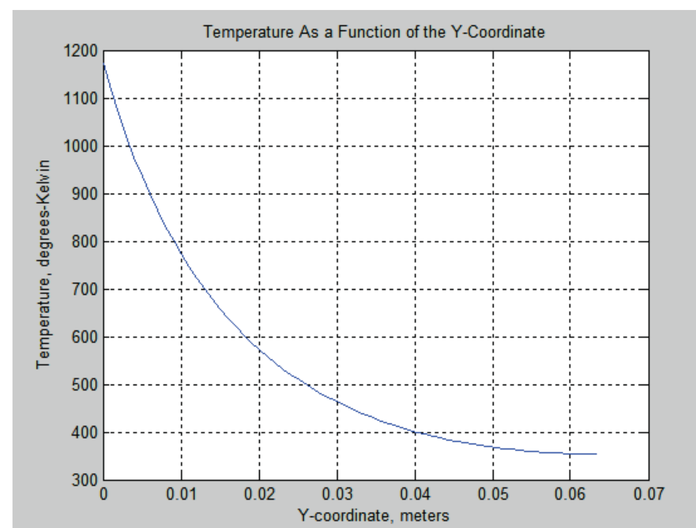
Heat Transfer Analysis

$$-kt_z\nabla^2 T + 2h_c T + 2\epsilon\sigma T^4 = 2h_c T_a + 2\epsilon\sigma T_a^4$$

- A steady-state 1D heat transfer model was developed to determine the longitudinal thermal gradient from heated zone to ER electrode:
  - Radiation and convection losses on specimen face
  - Constant thermal conductivity
  - Constant cross-section
  - Constant temperature in laser-heated region (has been verified experimentally through thermography)
  - $T(0) = T_{\text{heated region}}$
- Temp. distribution solved numerically using damped Newton iteration technique

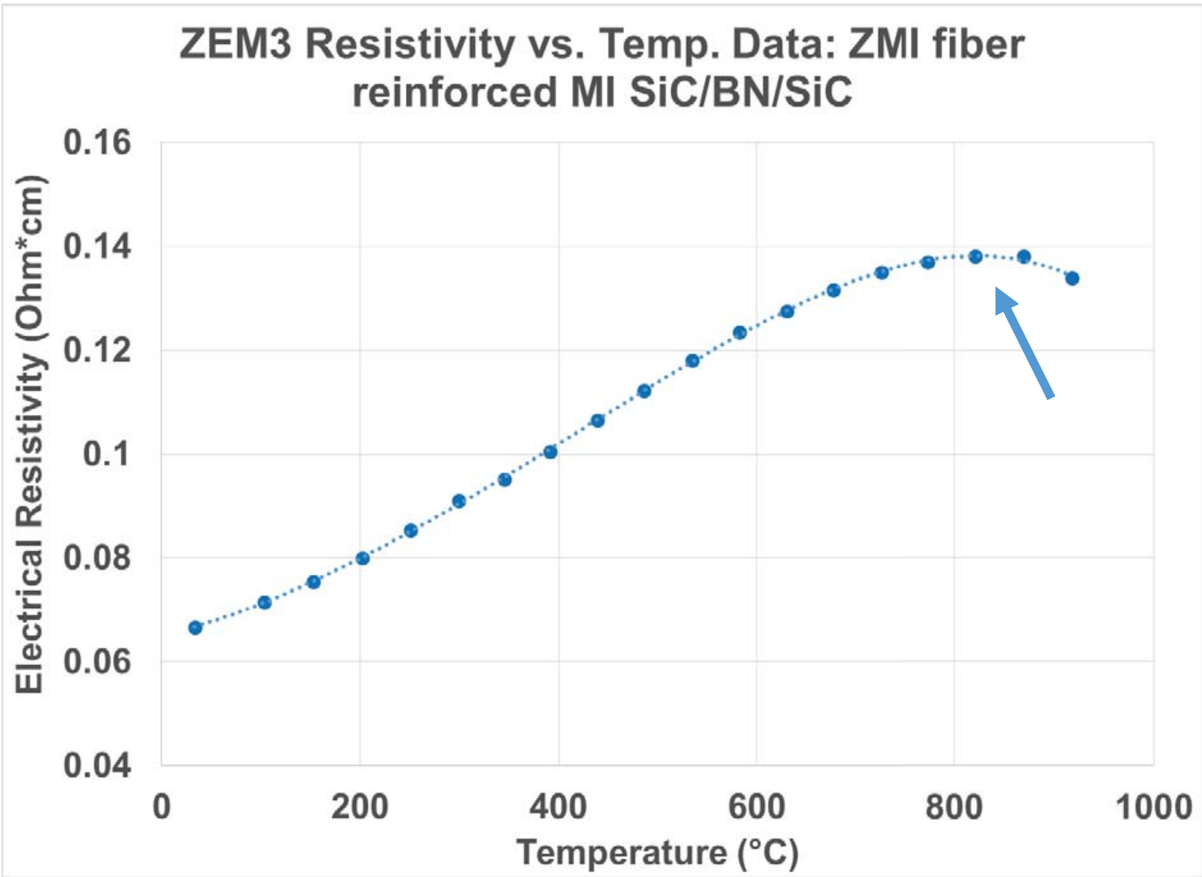


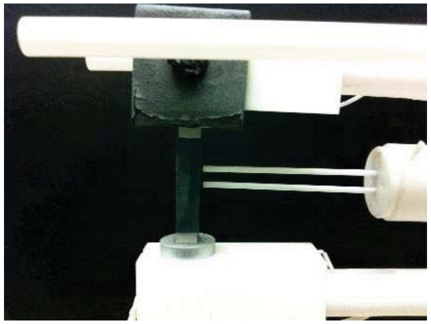




Thermal-Electrical Modeling

- The previously modeled temperature distribution can then be populated with experimental resistivity data (**20-900°C**) measured using the commercially available ZEM3 unit.



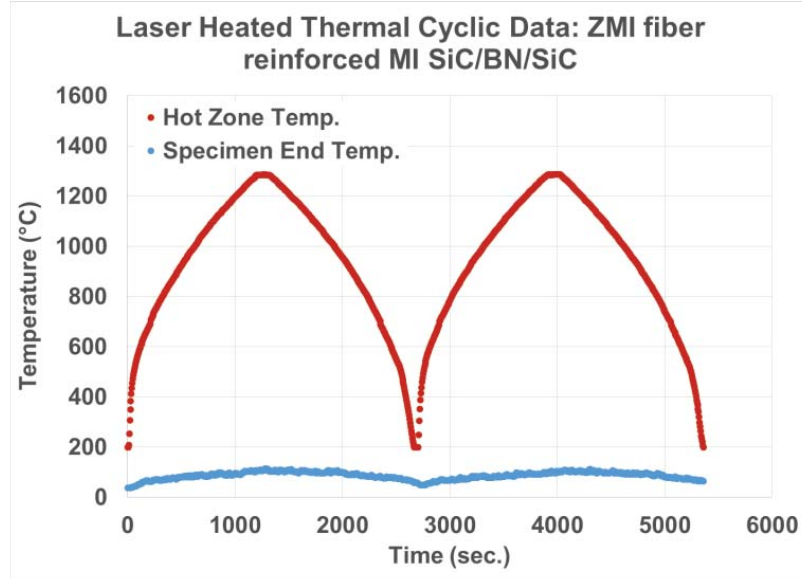


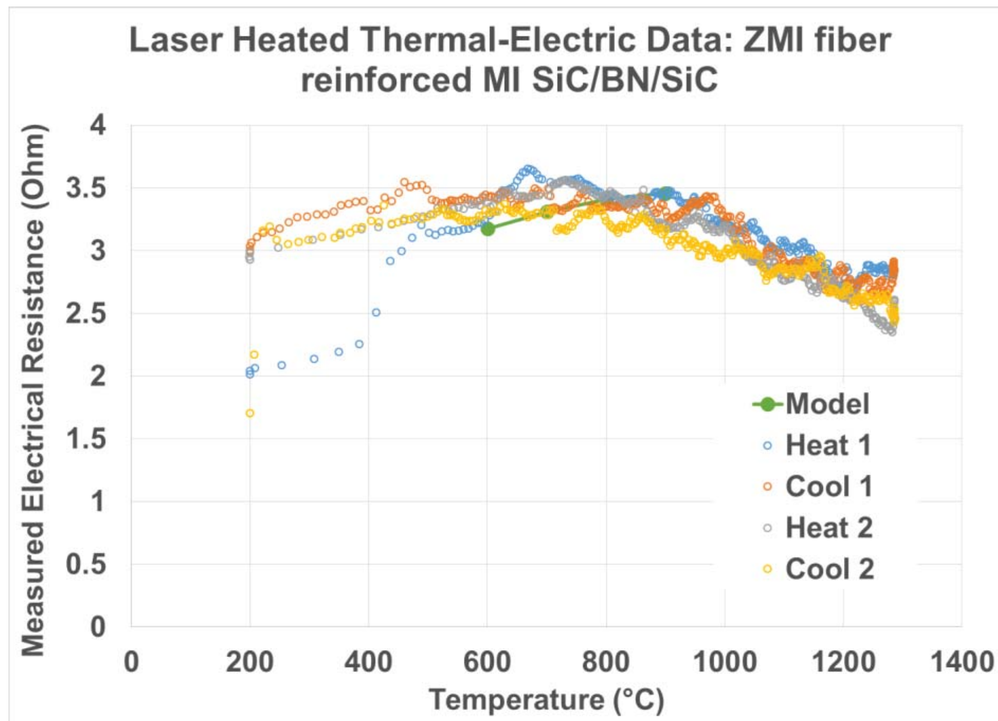
A small sample of CMC is heating isothermally in a furnace, and a 4pt resistance measurement is taken to determine temperature dependent resistivity

Thermal-Electrical Model Experimental Verification

- A ZMI fiber reinforced CMC tensile bar was laser heated under zero stress conditions with no applied cooling air
- The ZEM3 data for this material was used to calculate the total resistance of the specimen based on the temperature profile generate from the heat transfer analysis
- This was then compared to experimental data with good agreement
- This allows us to model the total thermal-electric response of the specimen and compare it with the data measured during testing from the grip section

$2R_{T(y)}$ $R_{Heated\ zone}$



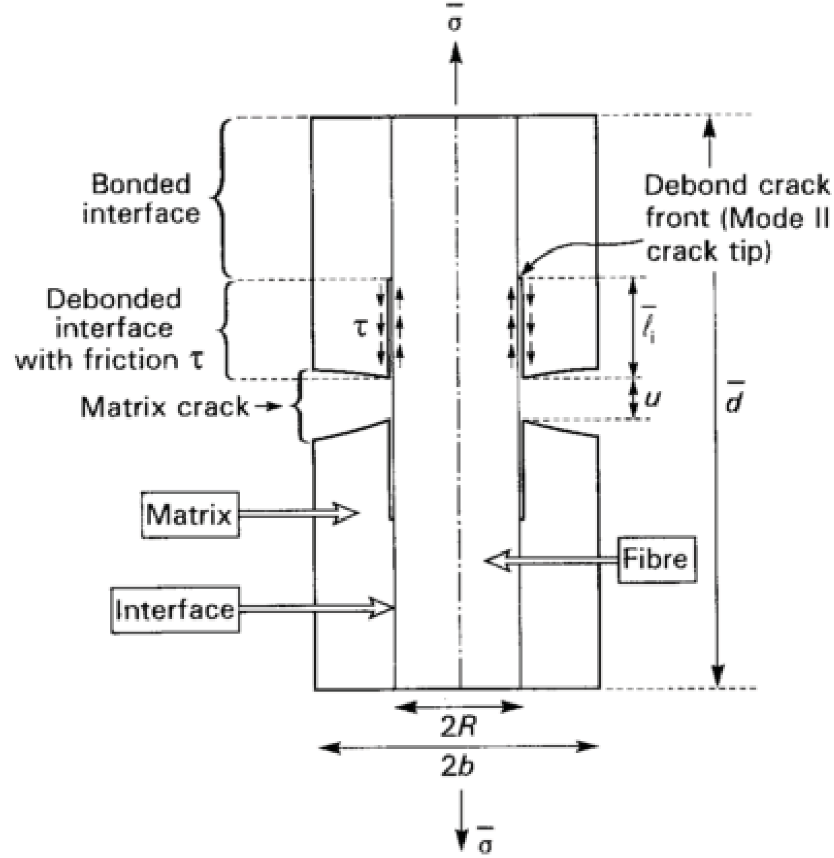


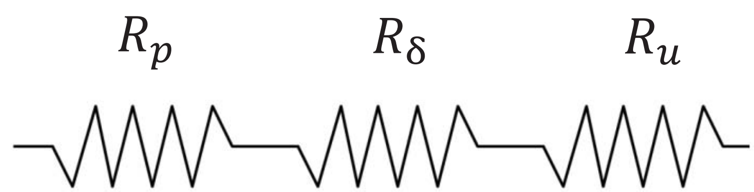
Early ER-Damage Model

- Current room temp. ER model (Baker, Maillet, Morscher, Appleby) under development
  - Based on brittle composite cracking/fiber-sliding micromechanics model
- From known stress based crack density (from AE), the composite is modeled as a series circuit of 3 types of resistors

$R_{undamaged} = R_p \quad R_{debond} = R_\delta \quad R_u = R_{fiber}$

- R_δ is a sliding contact resistance at the fiber/matrix interface; proposed as a function of radial stress on the fiber and interfacial shear stress τ
- Added complexity of high temperature thermal gradient testing
  - **Temperature dependence of electrical resistance of constituents: fiber, matrix and interphase**
  - **Thru thickness thermal stress gradient induced by asymmetric heating**
  - **Addition of EBC coating**





Conclusions

- **Convoluted Electrical Response of EBC/CMC's**
  - Thermal, Mechanical, Environmental and Time-Dependent
- **ER able to assess temperature changes**
- **ER sensitive to damage onset and accumulation**
  - Deviation from AE curve suggests ER sensitivity to fiber sliding effects as well as crack density
  - Proved to be useful in identifying damage from excessive thermal stresses caused during laser malfunction (not evident directly from load data)
  - Proved helpful for overall health monitoring and material inspection
- **ER increase during creep** caused by time-dependent deformation and environmental effects under constant stress condition
  - More sensitive to material condition than strain measurement alone
- **Need to further investigate EBC contribution and effects**
  - e.g. benefit of stable EBC to increased high temperature stress-rupture life

Current and Future Work

- **Carefully crafted "proof of concept" testing and more sophisticated models are still required to fully understand the nature of electric response.**
- Further development of thermal-electric model to include through thickness thermal gradient
- Use of thermal-electric model and high-temperature experimental data to separate contributions of constituents (fiber, matrix, coating) to ER
- Use thermal-electric constituent data to further develop ER damage model
- Current Testing focusing on EBC cracking/delamination detection using AE monitoring possible EBC contribution to ER response shows promise; however further testing required.

The University of Akron

Thank You