Characterization of SiC/SiC Composites in Support of Environmental Degradation Modeling

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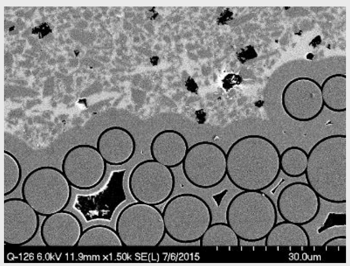

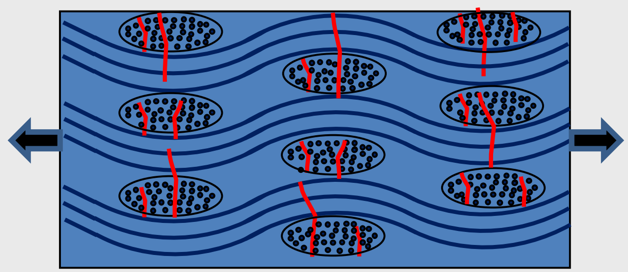

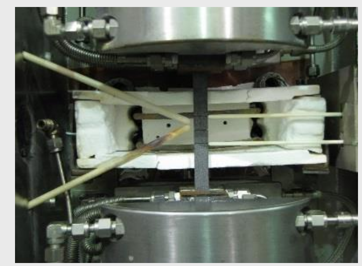

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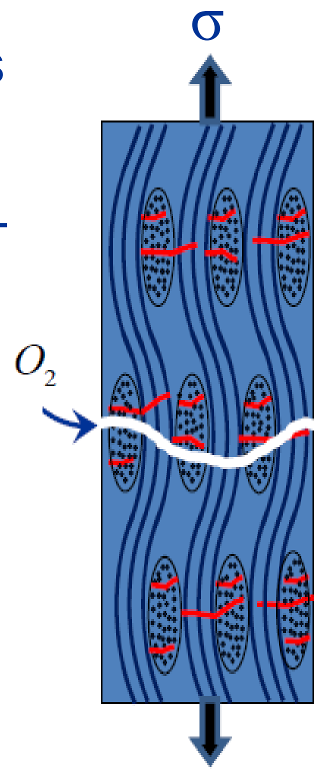
Initial Premise:

Consider 2 Different Scenarios for SiC/SiC at "Intermediate Temperature"

- At 815°C (in air), an **uncracked** melt infiltrated (MI) Sylramic™ SiC_f /BN/ SiC tensile specimen **loaded beneath the proportional limit stress (PLS) should not show much if any strength degradation**
- We assume slow crack growth is not an issue for Sylramic™ SiC fiber at 815°C (1500°F), and that sealed sample edges prevent oxidation from occurring.



- However, at 815°C **the life of a specimen will decrease once a through-thickness crack is present and a significantly high load is applied to the specimen, due to oxidation of the BN interface and tensile strength loss**

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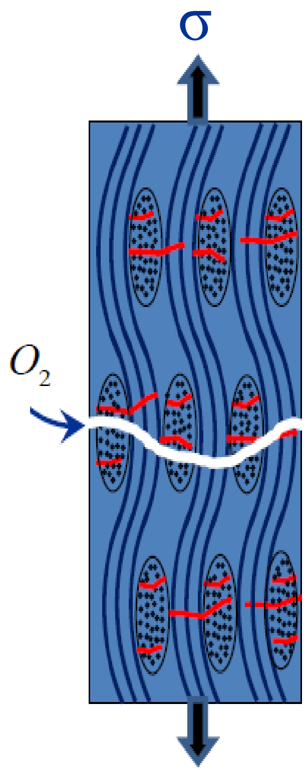


? **Can the comparison of *experimentally-observed phenomena* with *model predictions* lead to improved understanding of material degradation mechanisms?** ?

Environmental Degradation of SiC/SiC Composites

Overall Objective (Phase I—Intermediate Temperature)

Determine critical oxidation mechanisms and develop accurate models for the effect of oxidation on the time-dependent strength and life of SiC_f /BN/ SiC composites at Intermediate Temperatures

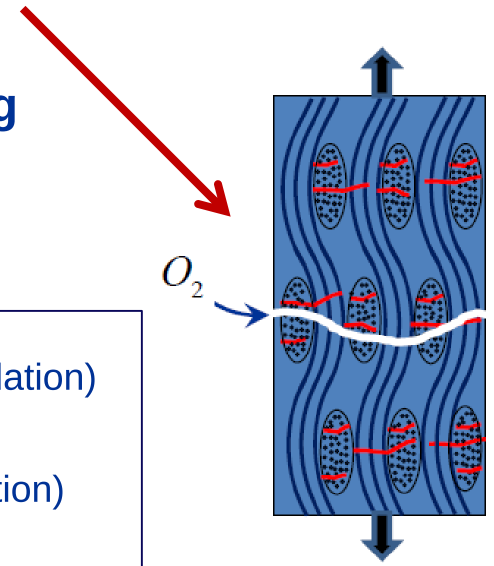
Approach (Phase I—Intermediate Temperature)

Perform parallel and correlative experimental and numerical analysis studies

- Develop numerical diffusion/oxidation model
- Develop mechanical models that simulate the effect of oxidation on the strength of SiC_f /BN/ SiC composites
- Perform experimental and microstructural studies to:
  - Investigate key oxidation mechanisms
  - Provide input to the model and obtain data for model validation

SiC/SiC Oxidation Model Overview

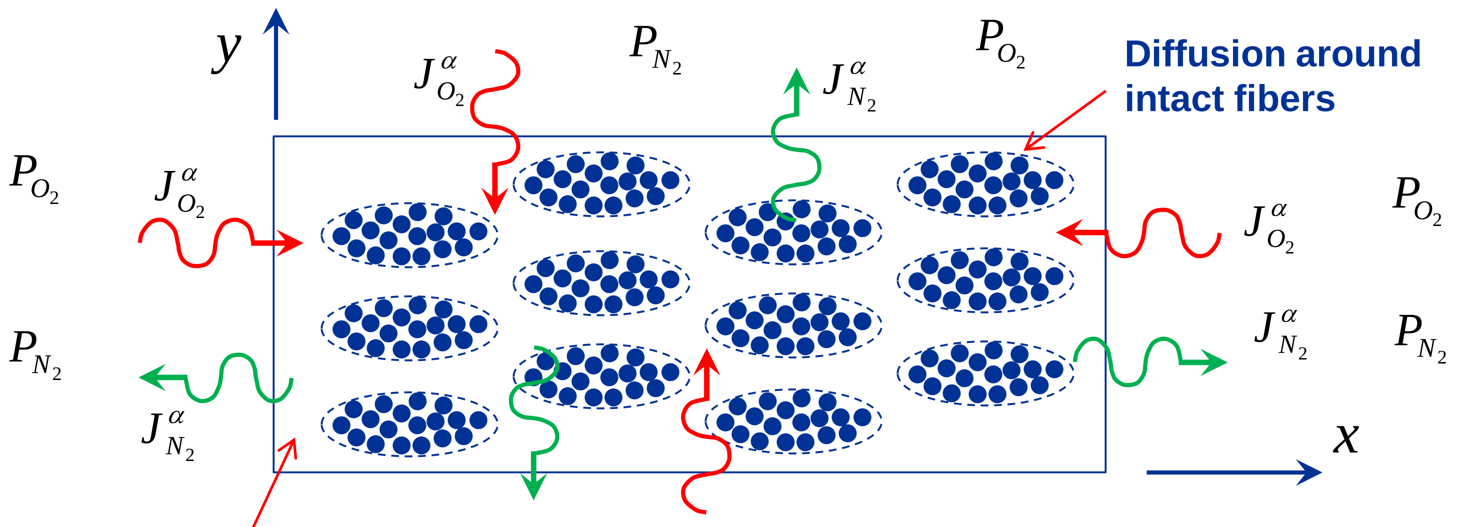
- **Model the diffusion of oxygen within a through-crack bridged by SiC fibers**
- **Model the oxidation of SiC fibers and BN fiber coating *at the local level* and calculate the extent of oxidation at each location within the cross-section**



Fibers	$SiC + 2O_2(g) \rightarrow SiO_2 + CO_2(g)$	(passive oxidation)
	$SiC + O_2(g) \rightarrow SiO(g) + CO(g)$	(active oxidation)
Fiber coating	$2BN + \frac{3}{2}O_2(g) \rightarrow B_2O_3 + N_2(g)$	
	$SiO_2 + B_2O_3 \rightarrow B_2O_3SiO_2$	(borosilicate glass)

- **Restrict the focus to temperatures below 1000°C *to avoid creep effects***
- **Restrict the focus to oxidation involving dry air *to avoid volatilization of boria***

SiC/SiC Oxidation Model Overview (cont.)

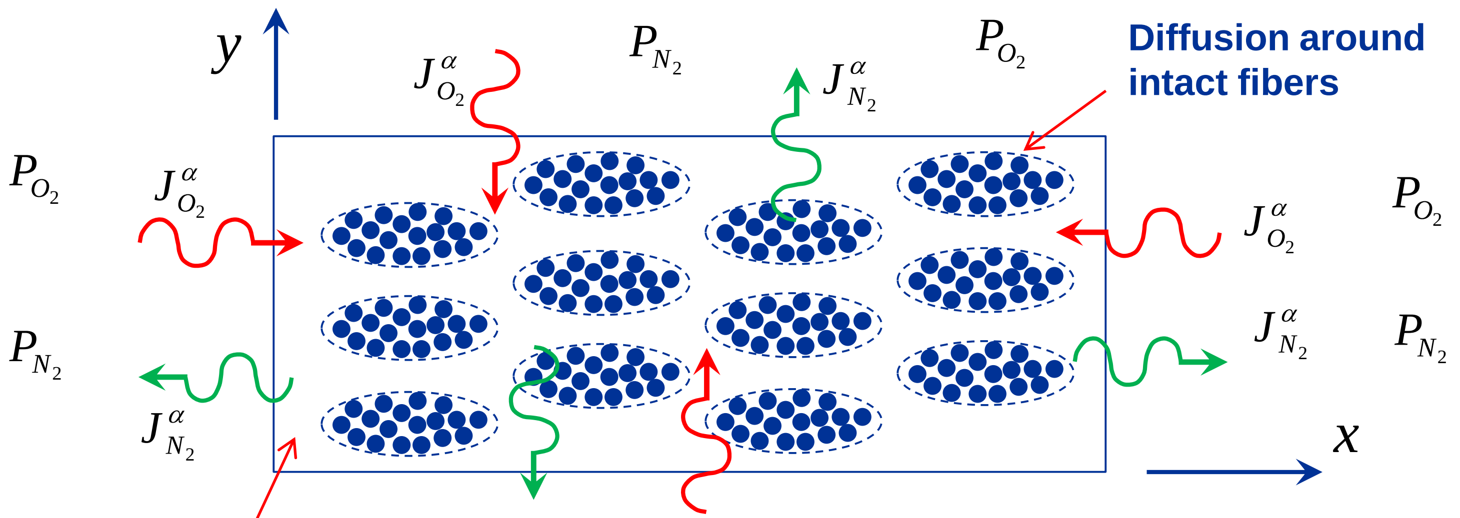


Open Channel diffusion (molecular or Knudsen)

Diffusion within the crack plane, looking at 0° tows

$$2BN + \frac{3}{2}O_2 \rightarrow B_2O_3 + N_2$$

SiC/SiC Oxidation Model Overview (cont.)



Diffusion around intact fibers

Open Channel diffusion (molecular or Knudsen)

Diffusion within the crack plane, looking at 0° tows

$$2BN + \frac{3}{2}O_2 \rightarrow B_2O_3 + N_2$$

Perform coupled solution of local mass conservation equation for each gas specie

Oxidation of BN

oxygen

$$\phi \frac{\partial \rho_{O_2}}{\partial t} + \vec{\nabla} \cdot \vec{J}_{O_2}^{\alpha} = -\frac{n}{\delta} A_c J_{O_2}^{\beta}$$

nitrogen

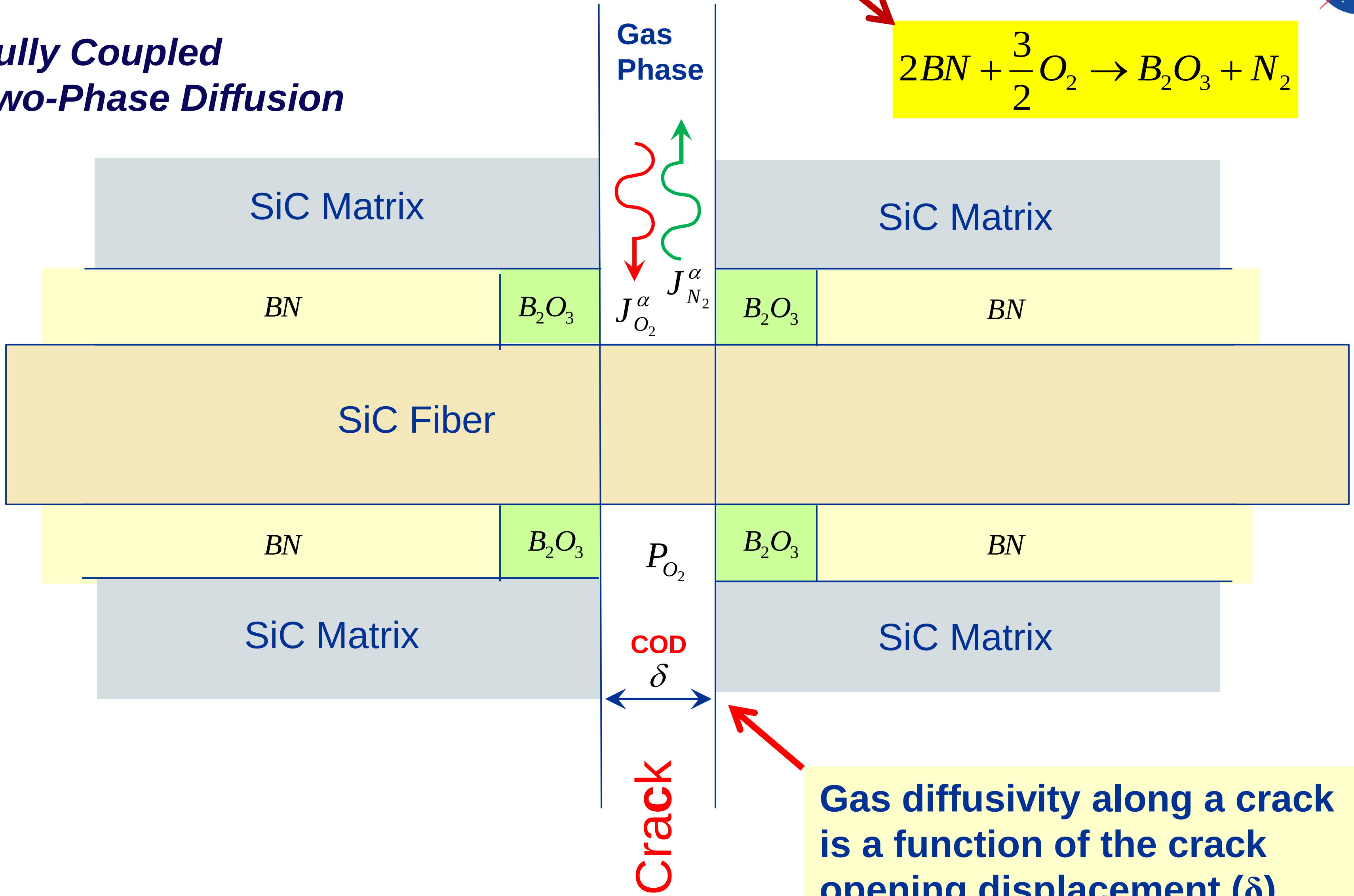
$$\phi \frac{\partial \rho_{N_2}}{\partial t} + \vec{\nabla} \cdot \vec{J}_{N_2}^{\alpha} = \frac{n}{\delta} A_c J_{N_2}^{\beta}$$

Solution yields the partial pressure of oxygen and nitrogen at each location

Modeling Approach (Local View of BN Oxidation)

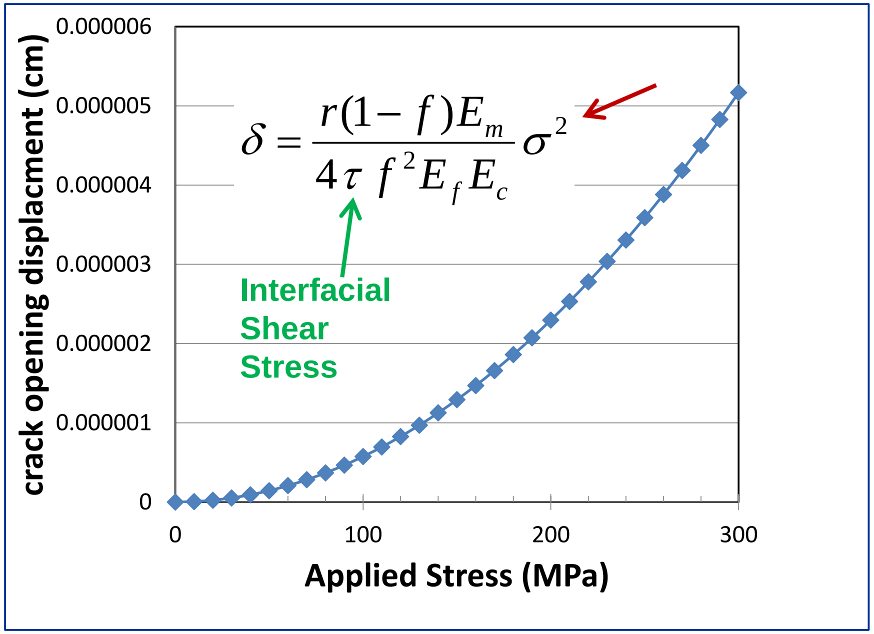
Fully Coupled Two-Phase Diffusion

$$2BN + \frac{3}{2}O_2 \rightarrow B_2O_3 + N_2$$

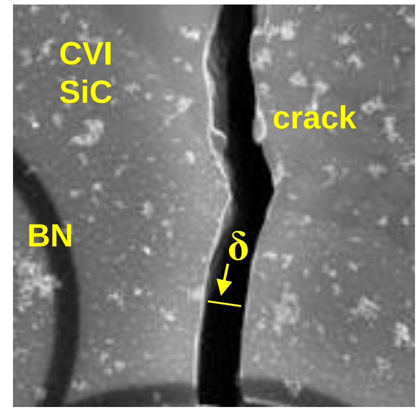


Gas diffusivity along a crack is a function of the crack opening displacement (δ)

Crack Opening Displacement (δ)



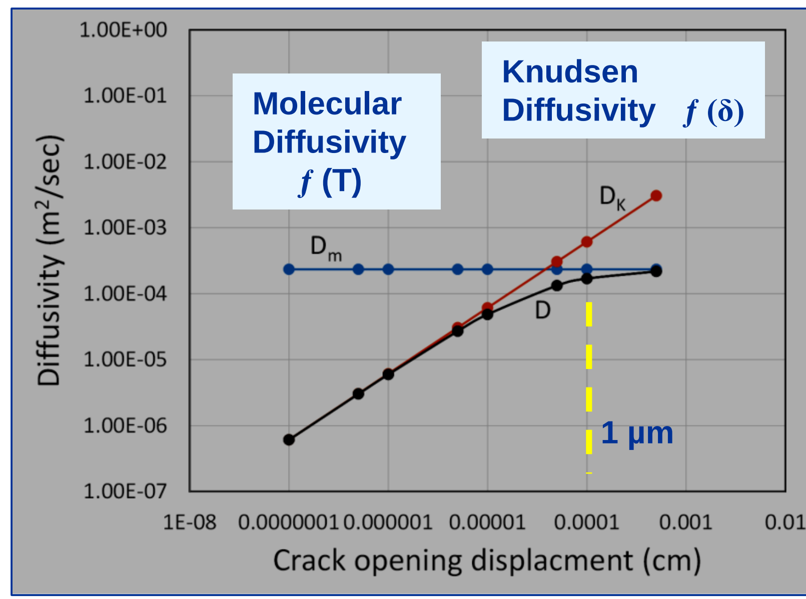
Crack opening displacement is a second-order function of the applied stress



Gas diffusivity along a crack is a function of the crack opening displacement

$$\frac{1}{D} = \frac{1}{D_m} + \frac{1}{D_K}$$

Molecular diffusivity ***Knudsen diffusivity***



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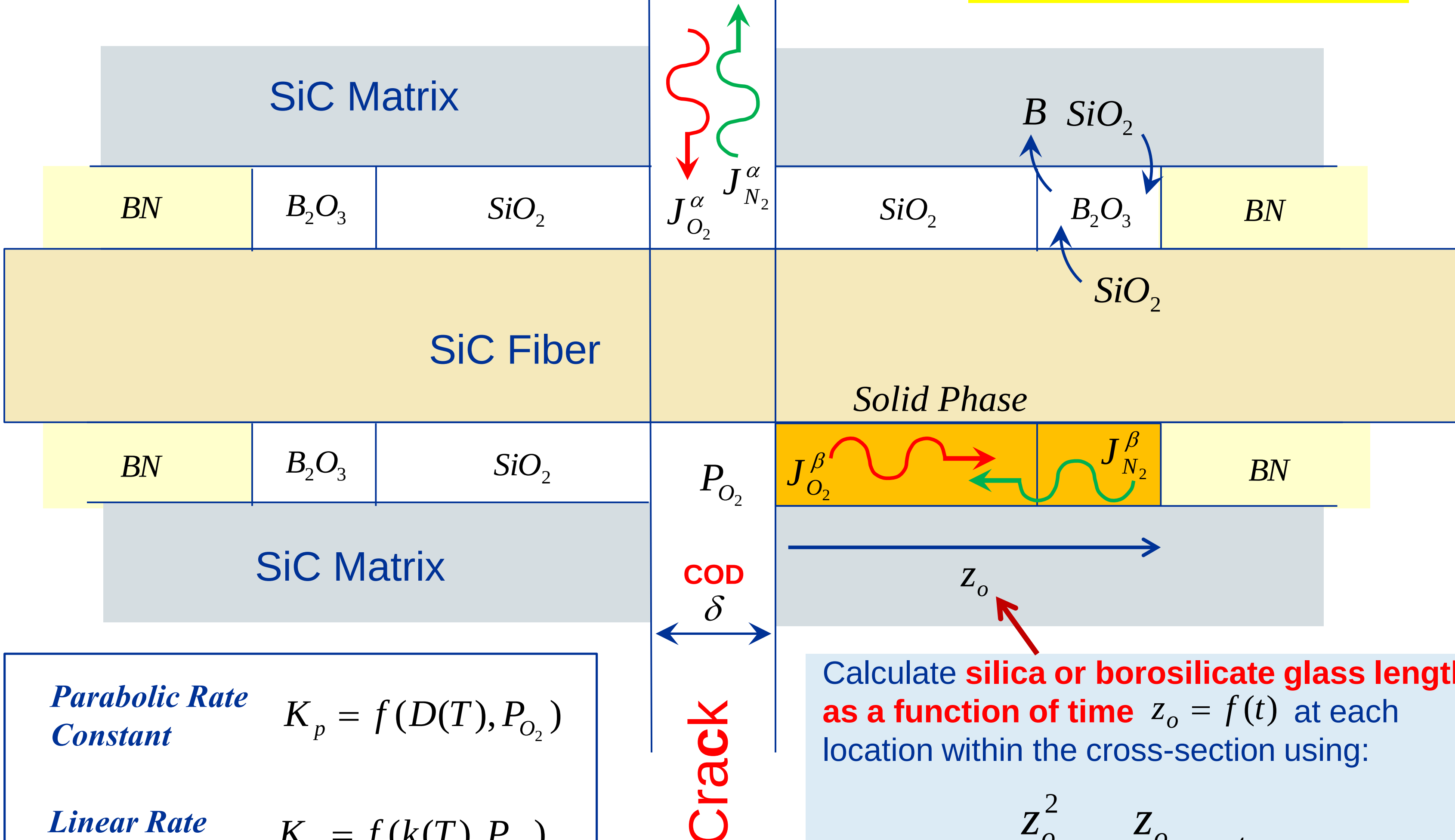
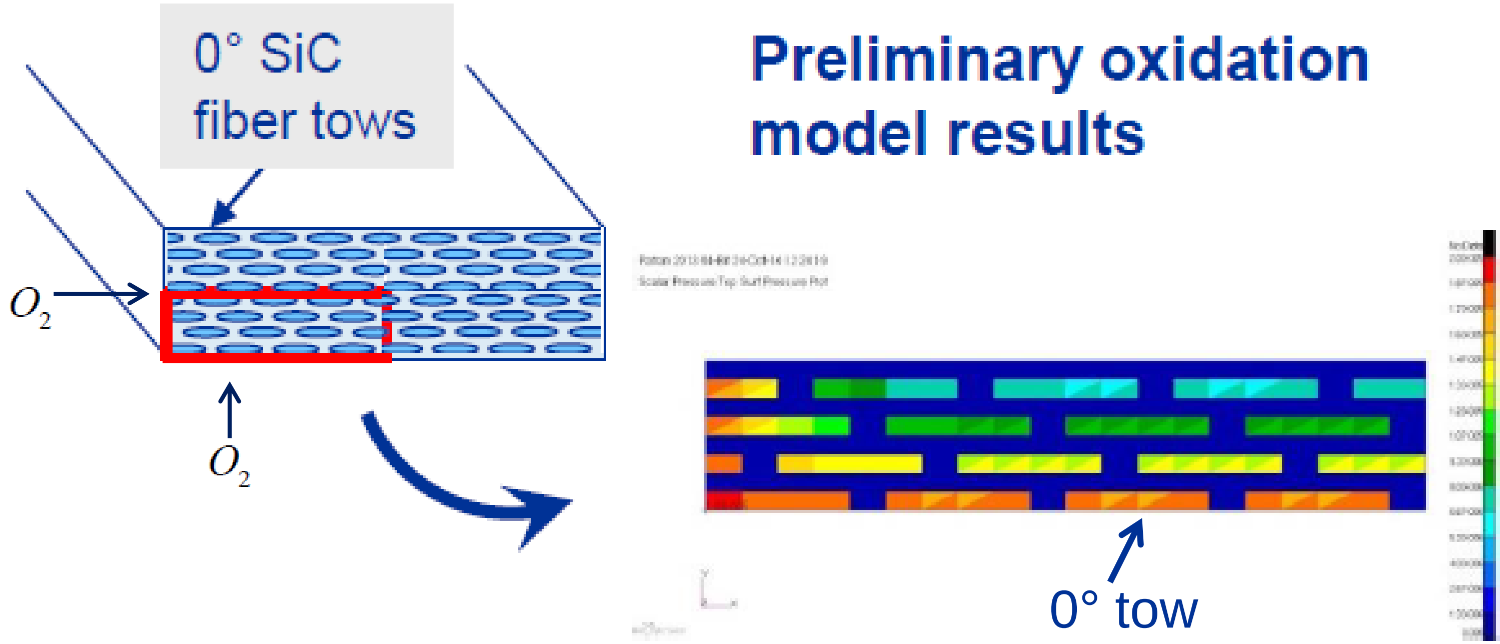


Parabolic Rate Constant	$K_p = f(D(T), P_{O_2})$
Linear Rate Constant	$K_L = f(k(T), P_{O_2})$

Calculate **silica or borosilicate glass length as a function of time** $z_o = f(t)$ at each location within the cross-section using:

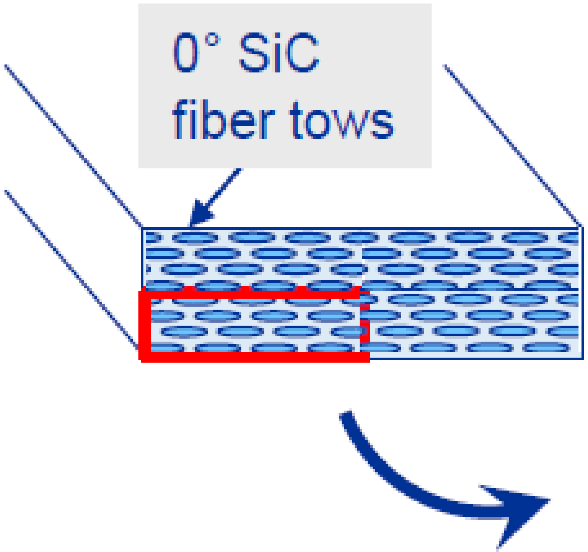
$$\frac{z_o^2}{K_p} + \frac{z_o}{K_L} = t$$

Oxidation Patterns in Crack Plane

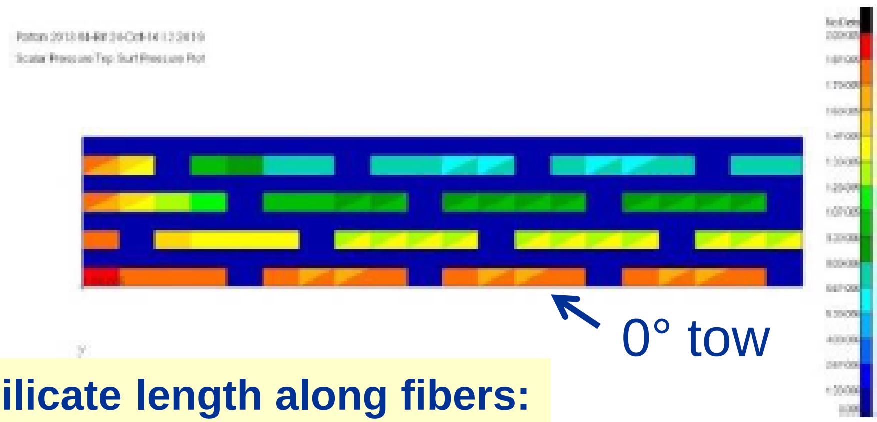


Fringe plot of boria/borosilicate length along fibers:
1 atm dry air @1000°C for 29 hours, 115 MPa applied stress

Oxidation Patterns in Crack Plane



Preliminary oxidation model results



Fringe plot of boria/borosilicate length along fibers:
1 atm air @1000°C for 29 hours, 115 MPa applied stress

?

- **Does this accurately represent the oxidation of BN that occurs?**
- **How does the “fusing” of the SiC fiber to the matrix affect CMC life?**
- **How would you characterize the microstructure of a stress rupture sample (failed or unfailed) to help verify this model?**

Questions to Address and Challenges

Questions

- What is the critical length of the oxide along the SiC fibers which is required for local embrittlement?
- Amount of fiber fusing (%) required to cause CMC failure to occur? **Is this the most critical issue affecting SiC/SiC durability at Intermediate Temperatures?**
- What is the COD along the crack plane (does it vary, and why)?
- Selection of a suitable precracking stress, and what is the nature of the crack or cracks formed by loading a notched or dog-bone tensile sample to 206.9 MPa?

Microstructural Characterization and Testing Challenges

- Fractography: characterizing a cross section that contains sixty-four 0° tows.

↓

- **Characterizing tensile samples that have been tested to failure at 815°C and correlating damage such as oxidation of the BN interface and embrittlement from fibers fusing to the matrix to composite loss of strength and failure. How to approach this, and amount of effort required?**
- Measuring crack opening displacement (COD) at various loads, given the predicted COD of approximately 1 μm or less.
- Tensile testing in controlled environments.

Assessing Degradation of SiC/SiC CMCs to *Support* Modeling Effort

Melt Infiltrated (MI) SiC/SiC Material Currently Being Utilized:

Fabricated by GEPSC (GE Power Systems Composites):

- Panels were fabricated in 4Q 2003 and shipped to NASA on **11/19/03**
- Dimensions: 6” x 9” x 0.082” (2.09 mm) panel
- **Sylramic™ SiC fiber** reinforcement (or is it Sylramic™-iBN?)
- **20 epi 5 HSW** (harness satin weave) fabric
- Lay-up: NASA provided the 8 ply preform, and GE “assembled” the preform into CVI (chemical vapor infiltration) tooling
- **CVI Si-BN (doped) interface** + CVI SiC matrix deposited
- Followed by SiC slurry cast / MI (melt infiltrated silicon) matrix
- Minimal porosity due to slurry cast MI matrix. Some canned porosity in fiber tows.

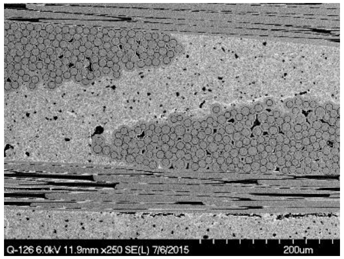
Characteristics

- Panels had a final bulk density of 2.78 g/cc, via Archimedes method (measured by GE). NASA bulk density estimates of approx. 2.8 g/cc, determined using smaller pieces (based on weights and dimensions).
- **NASA estimated the fiber vol. fraction = 0.38** (using equation (Morscher, Refr. 1) that accounts for fiber type, number of plies, epi, and panel thickness).

Summary and Status of Experimental and Characterization Tasks Identified to Support "Intermediate T" SiC/SiC Oxidation Modeling

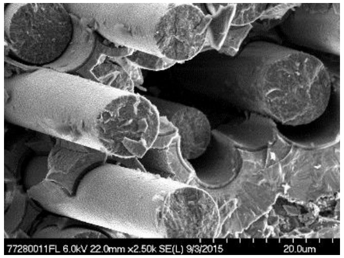
Significant Progress

- Characterize the microstructure and RT mechanical properties of the MI SiC/SiC that is being used
- Measure crack opening displacement as a function of applied stress (collaboration with University of Michigan)



In-Progress

- Initially, conduct experiments to obtain parabolic and linear rate constants at 815°C for boria and borosilicate glass formation
- Conduct stress rupture tests on notched, precracked MI SiC/SiC specimens and assess stress rupture life as a function of applied stress at different temperatures and oxygen partial pressures
- Examine the microstructural features of fracture surfaces and polished cross sections to characterize (quantify) oxidation patterns **challenging!**



Assessing Degradation of MI SiC/SiC CMCs to *Support* Modeling Effort

I.

Oxidation of Small Square Pieces

Heat treatment: GRC Rig 7, dry air, 815°C, up to 240 hrs

Examine CMC Surfaces w/SEM, then Mount/Polish. Characterize Oxidation of BN (Depth/ Comp.), Look for Fiber Degradation

Calculate Parameters for Oxidation Model

II.

Critical Stressed Oxidation of Notched, Precracked Tensile Samples

Stressed oxidation testing: Rig 7, dry air, 815°C, start: 206.9 MPa

Characterize Cracking and Oxidation of Tows; Effect on Strength and Life

Input for Oxidation Model

III.

1) Characterize Microstructure and 2) RT σ_f of MI SiC/SiC CMC

Polish samples and perform RT tensile test/ fractography

1) Examine Microstructure and Try to Quantify Characteristics. 2) Determine σ, E, ε_f, and Examine Fracture Surface

Input for Oxidation Model

IV.

DIC at GRC—Examine Notched Tensile Samples Tested in Load Frame at Room T

DIC: Macroscale and Possibly Microscale Approach (No SEM) Learning Process.

Apply Compound to Cracked Areas, Allow to Dry Under Load

Examine Cracking, Attempt to Measure Crack Widths

V.

DIC Within SEM—Mature Approach With Higher Spatial Resolution (U. Michigan)

DIC: Microscale Approach (SEM) Also a Learning Process—Different MI SiC/SiC Material

Examine CMC Cracking at RT and Measure Crack Opening Widths. Learn About Technique

Synergy

Input for Oxidation Model: Cracking Behavior, Crack Opening Width, Ƭ (tau)

5 Different Tasks: Combined Input Supports Oxidation Modeling

Assessing Degradation of MI SiC/SiC CMCs to *Support* Modeling Effort

III.

1) Characterize Microstructure and 2) RT σ_f of MI SiC/SiC CMC

Polish samples and perform RT tensile test/ fractography

↓

1) Examine Microstructure and Try to Quantify Characteristics.
2) Determine σ, E, ε_f, and Examine Fracture Surface

↓

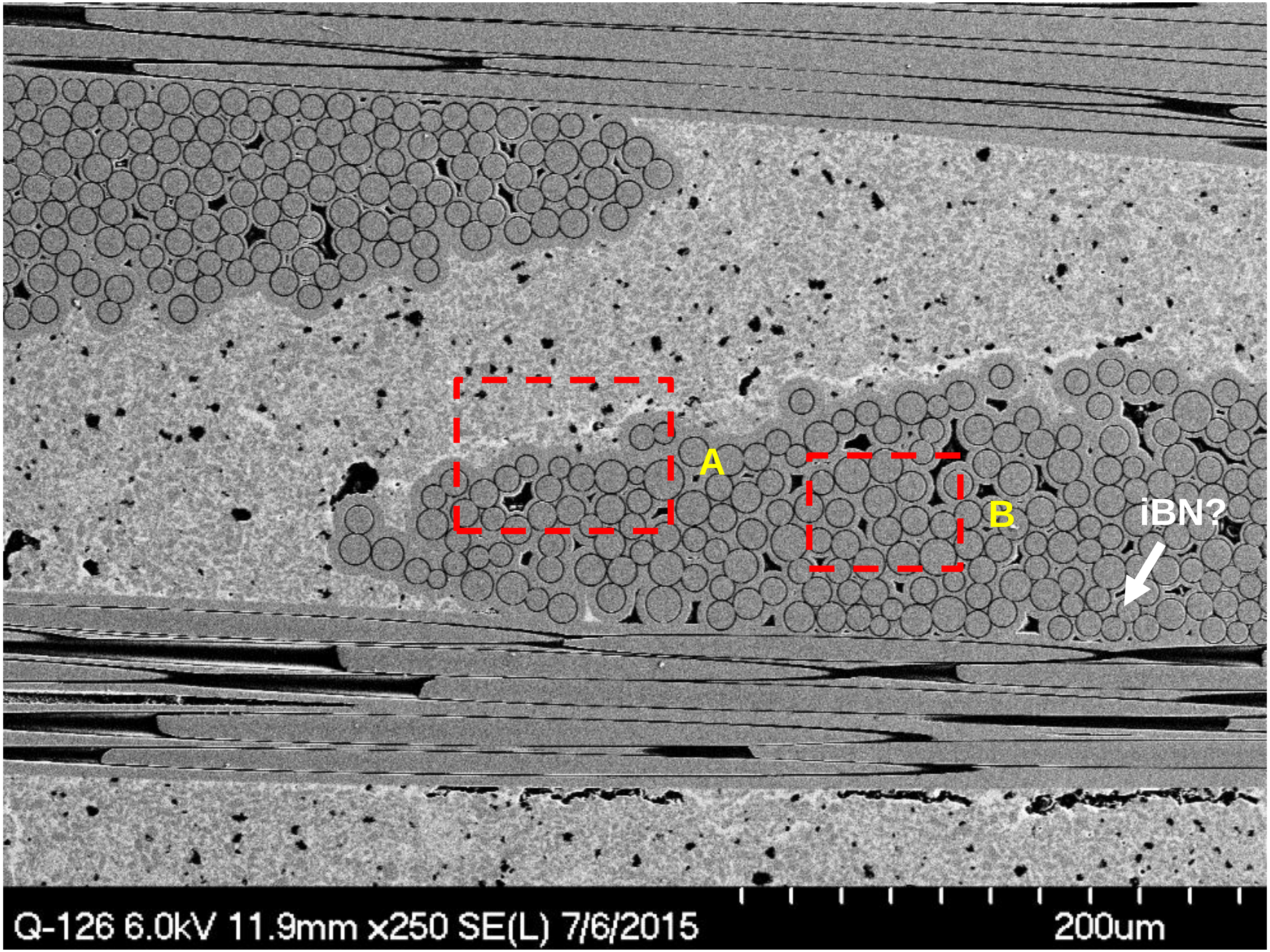
Input for Oxidation Model

Characterize Microstructure of Starting MI SiC/SiC CMC

- **Have characterized polished sections of the as-fabricated material with FESEM (Field Emission Scanning Electron Microscopy—Hitachi S-4700, Tokyo, Japan).**

1277-28-001 As-Fabricated MI SiC/SiC Material

Polished Section—Examined With FESEM



1277-28-001 As-Fabricated MI SiC/SiC Material

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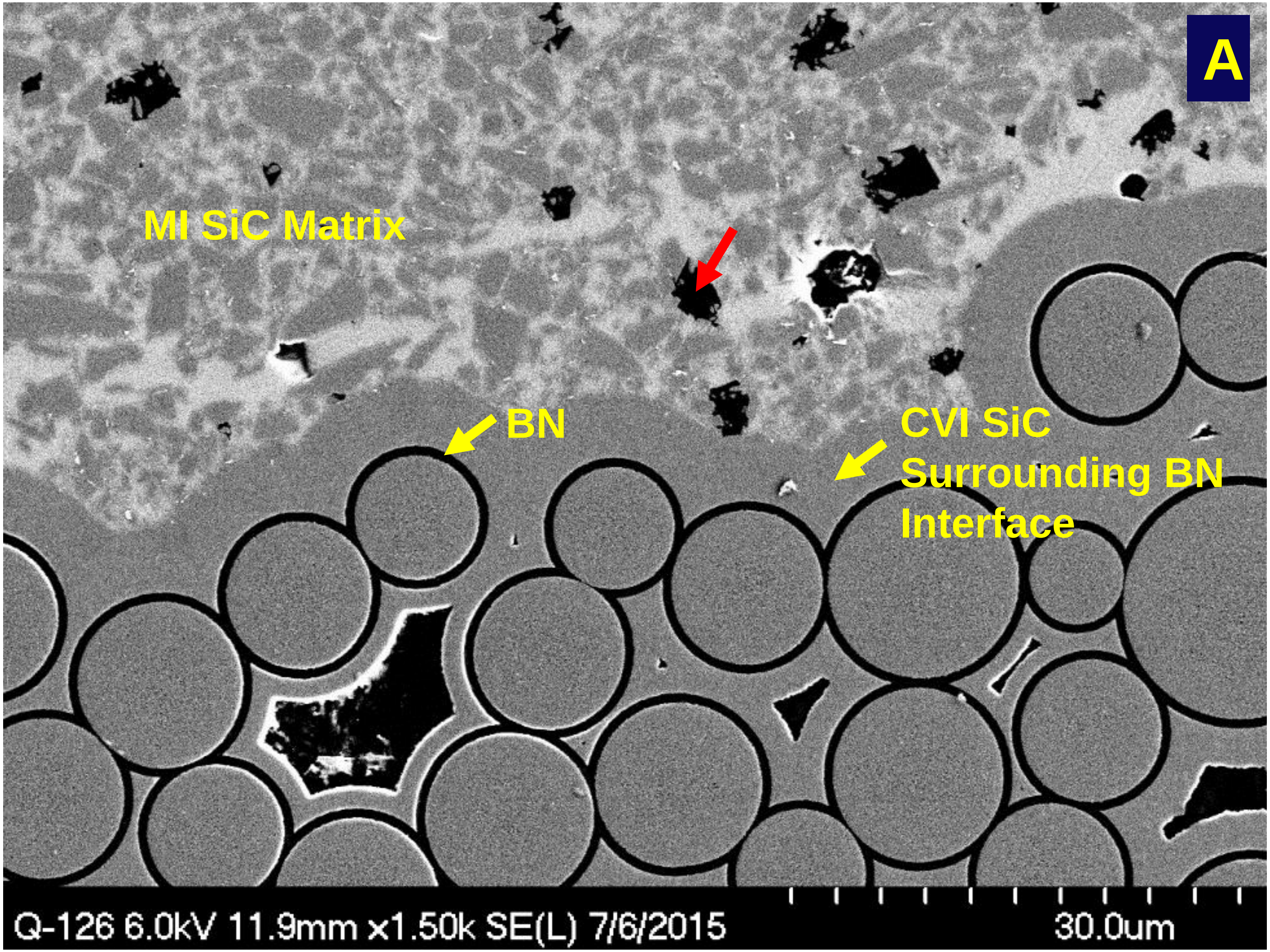

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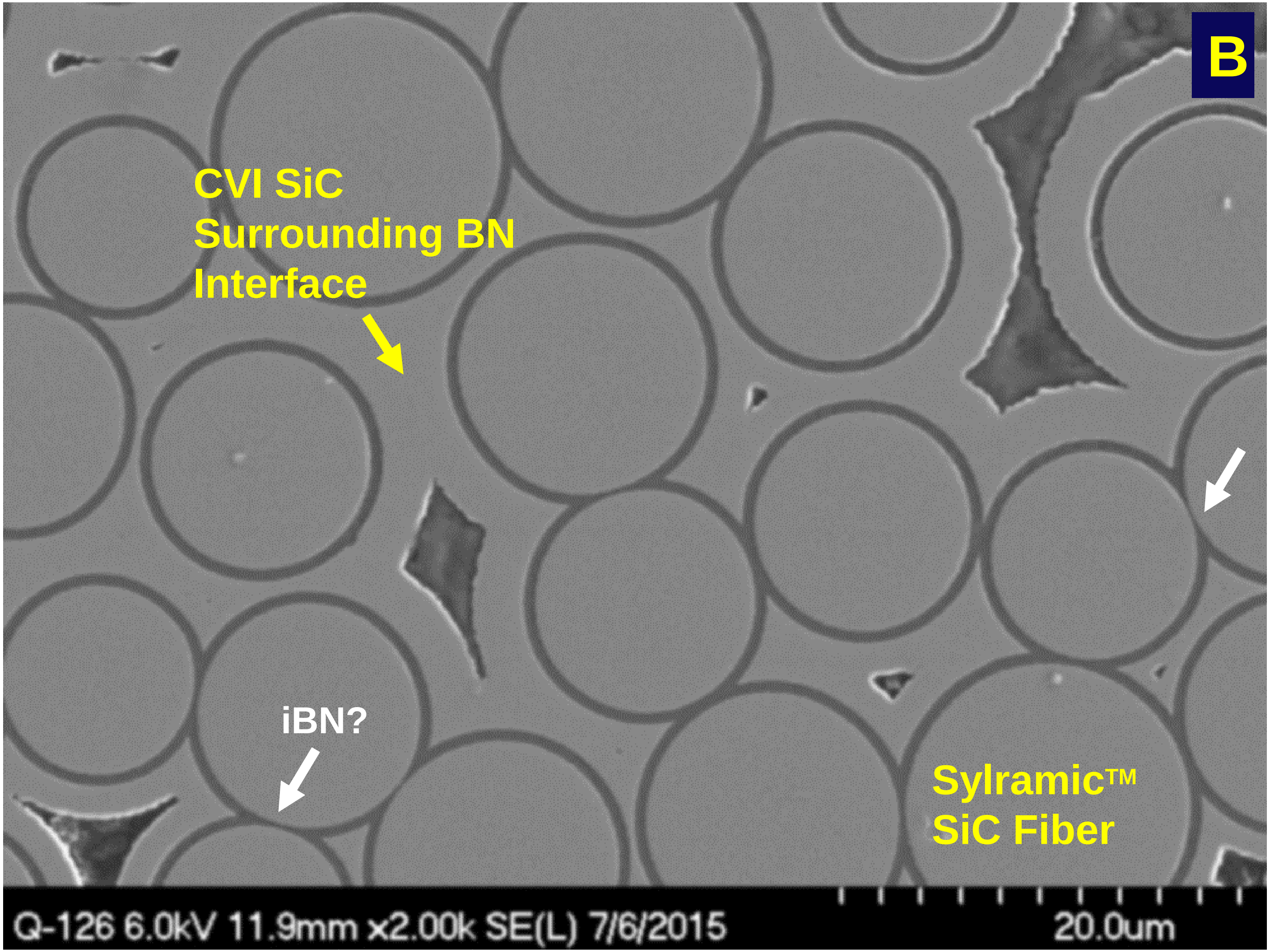

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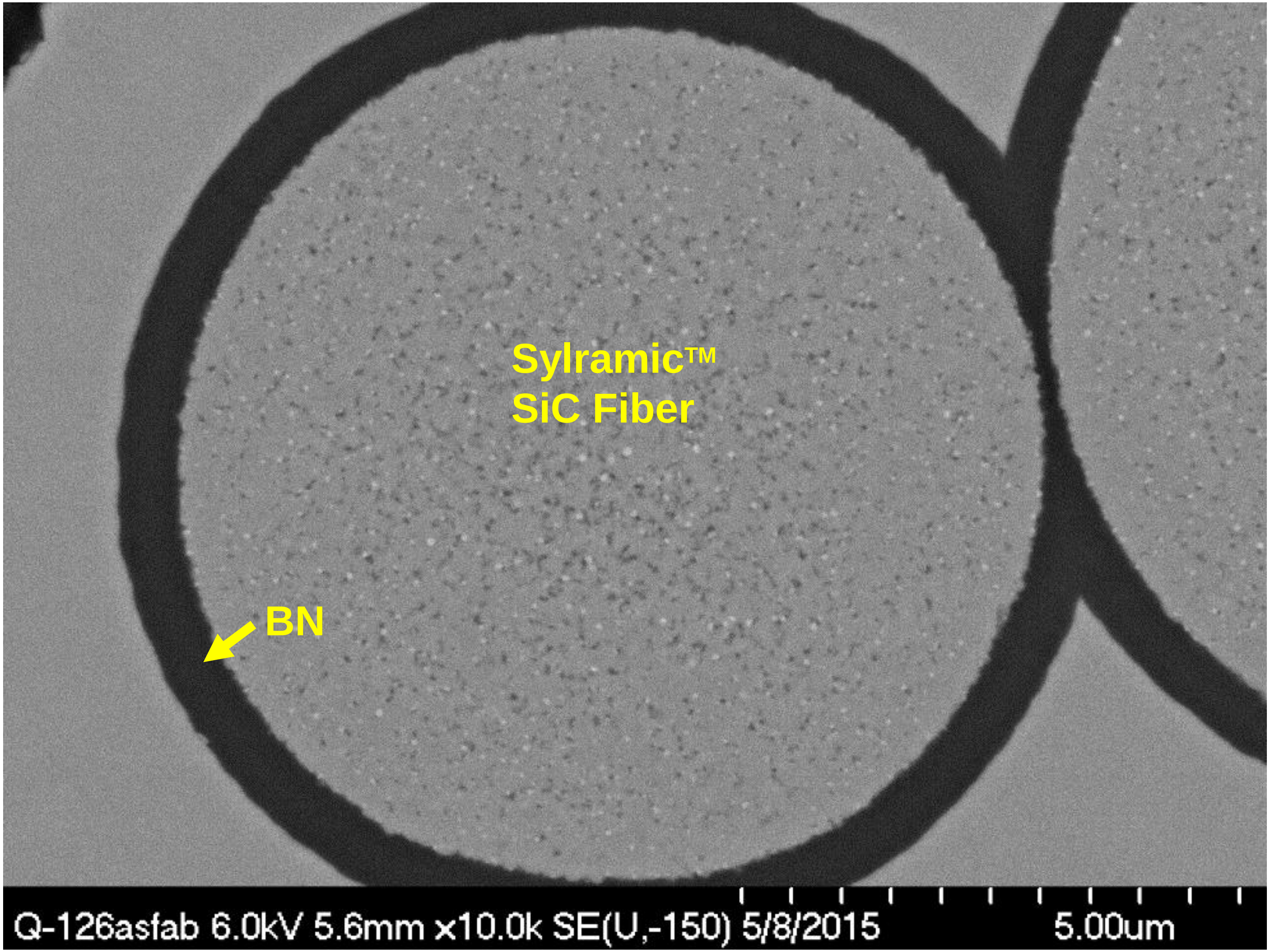
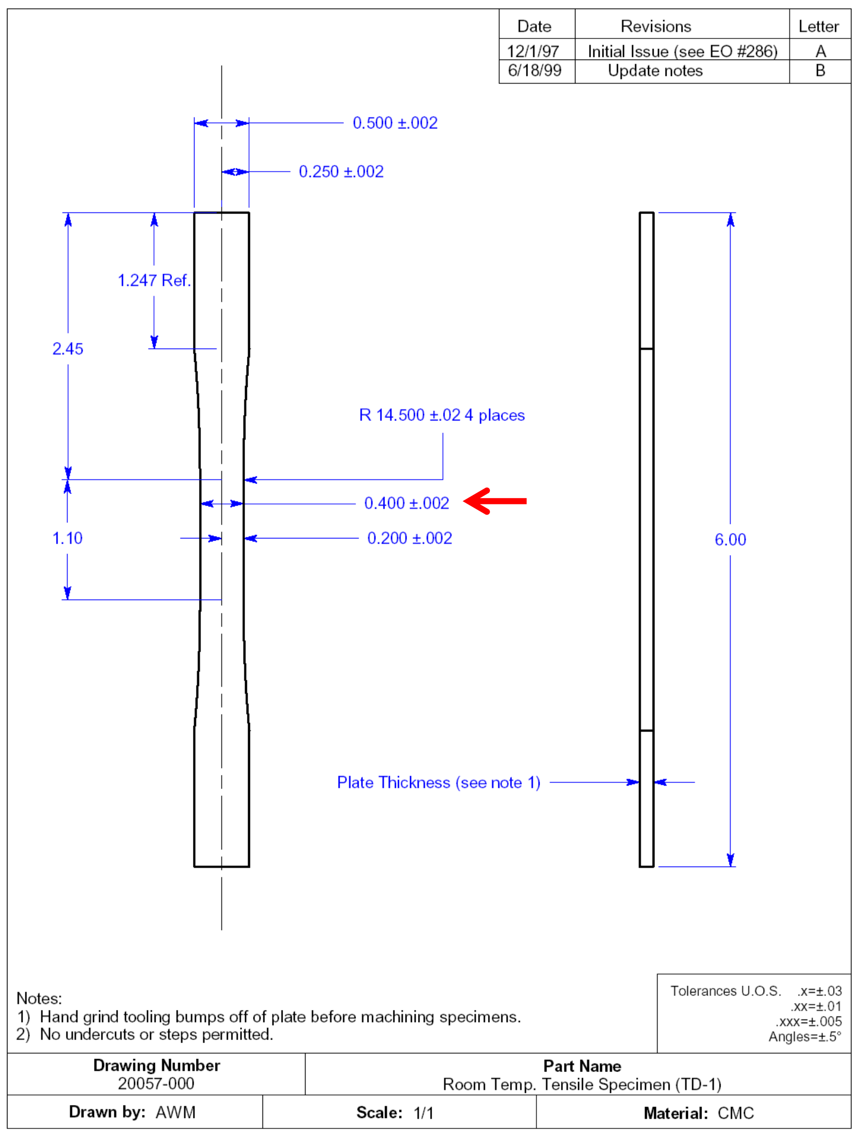


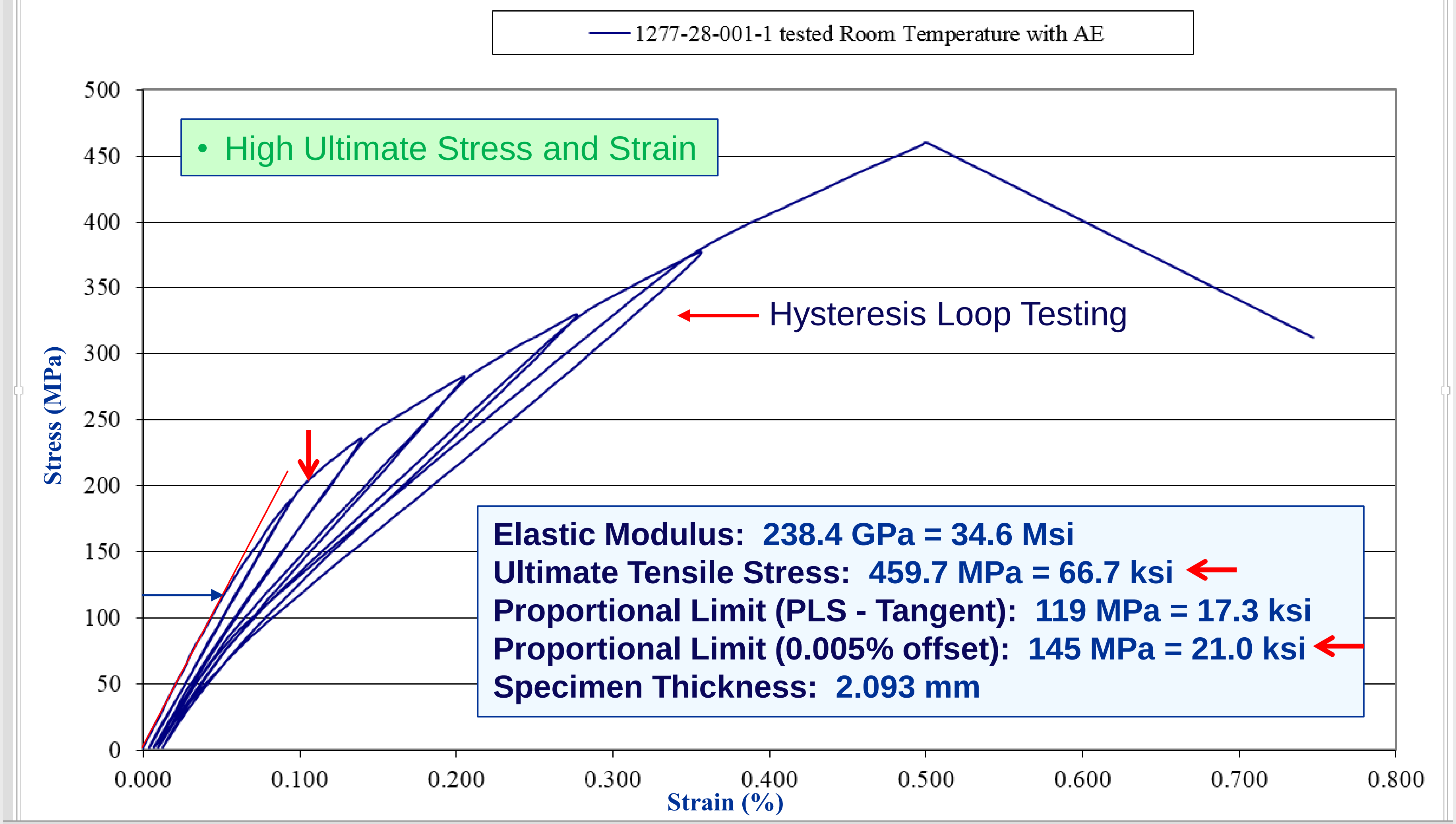
Determination of Room Temperature Fast Fracture Strength: EPM Tensile Geometry (Dog-bone Sample)



<u>gage section:</u>
20% reduction in width, with tapering from 0.5” (grip) to 0.4” (gage)

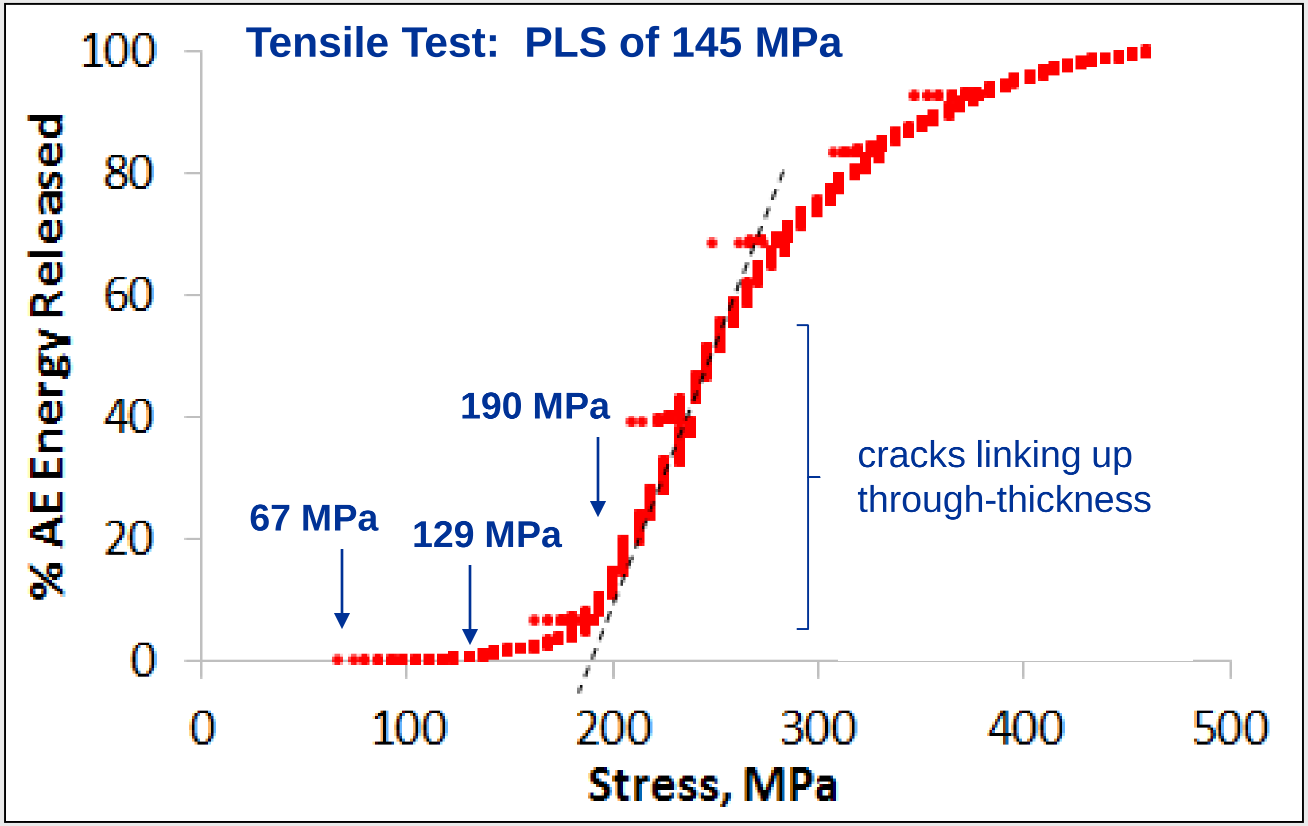
Determination of Room Temperature Fast Fracture (FF) Strength

MI SiC/SiC "EPM" Dog-bone Tensile Sample—RT FF



1277-28-001-1 As-Fabricated MI SiC/SiC Material Tested at RT

Acoustic Emission (AE) Curve

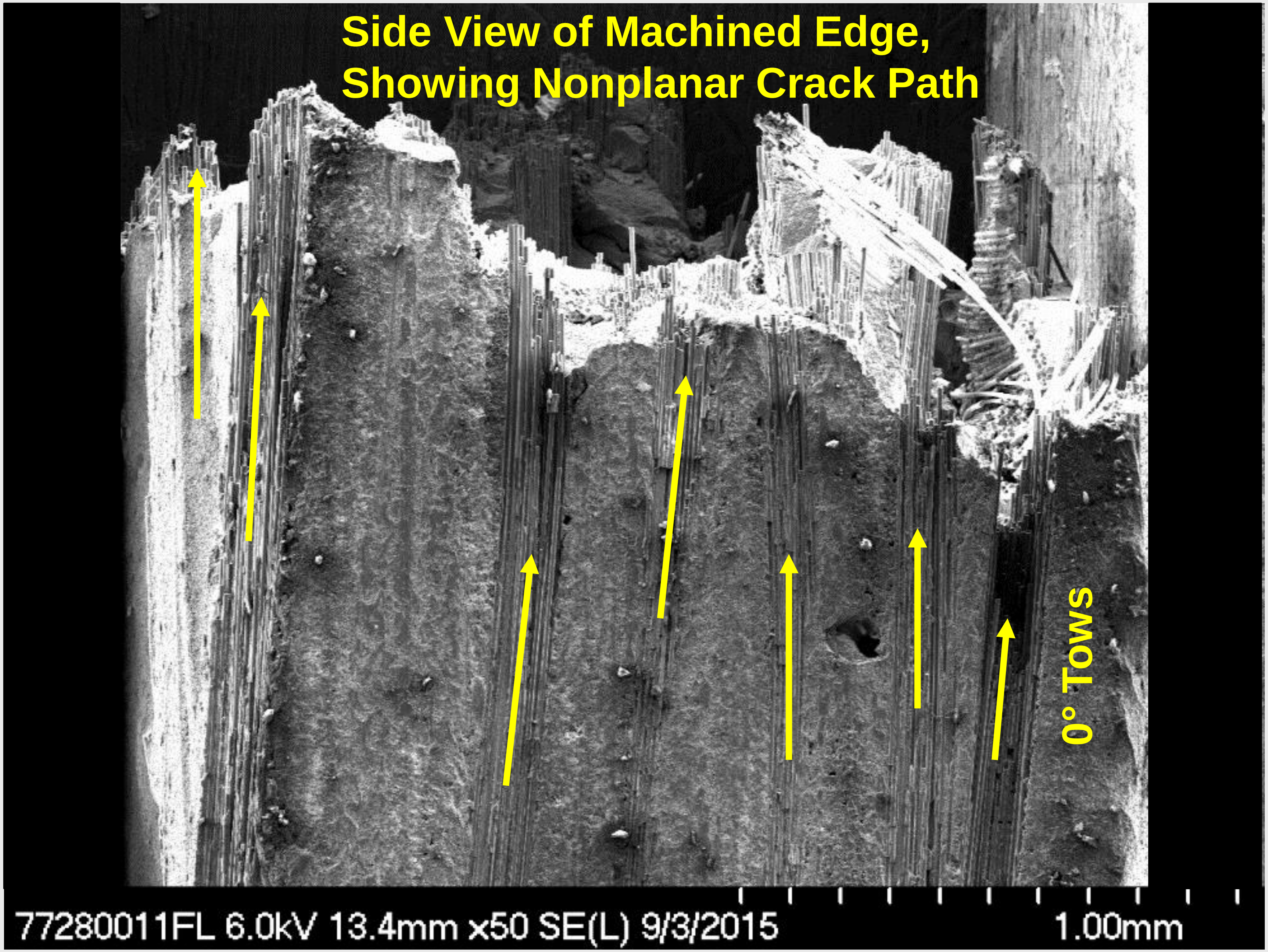


- **The first event occurred at 67 MPa. Assume: cracking in 90º tow.**
- **The first "loud" event occurred at 129 MPa. Energy: highest order magnitude. Assume this corresponds to the cracking of the CVI matrix material.**
- **AE onset occurred at 190 MPa.**
- **In stressed oxidation testing of notched tensile samples: precracked at 206.9 MPa.**

1277-28-001-1 As-Fabricated MI SiC/SiC Material FF Tested at RT

Fracture Surface—Examined With FESEM

Tensile Direction



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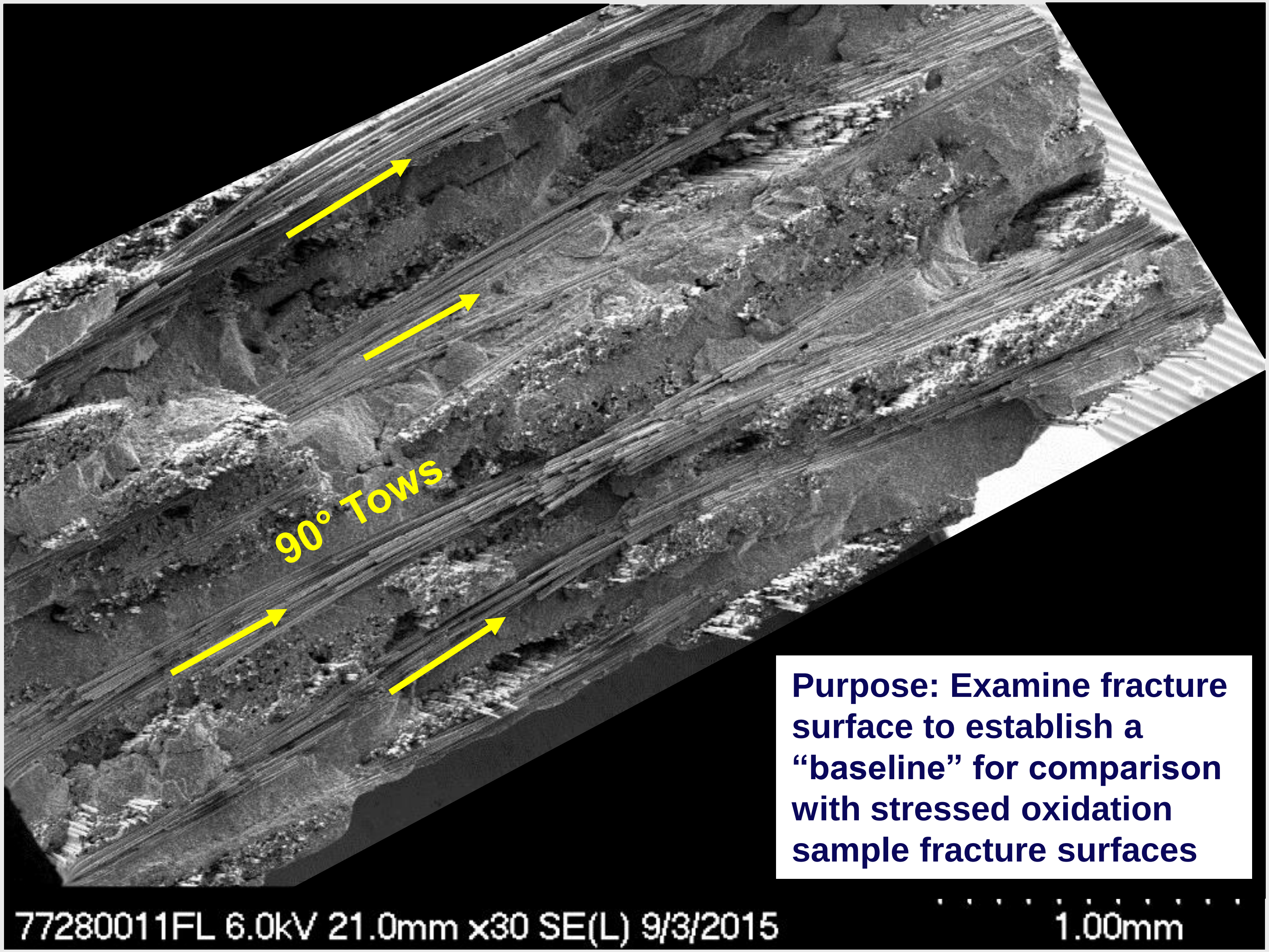
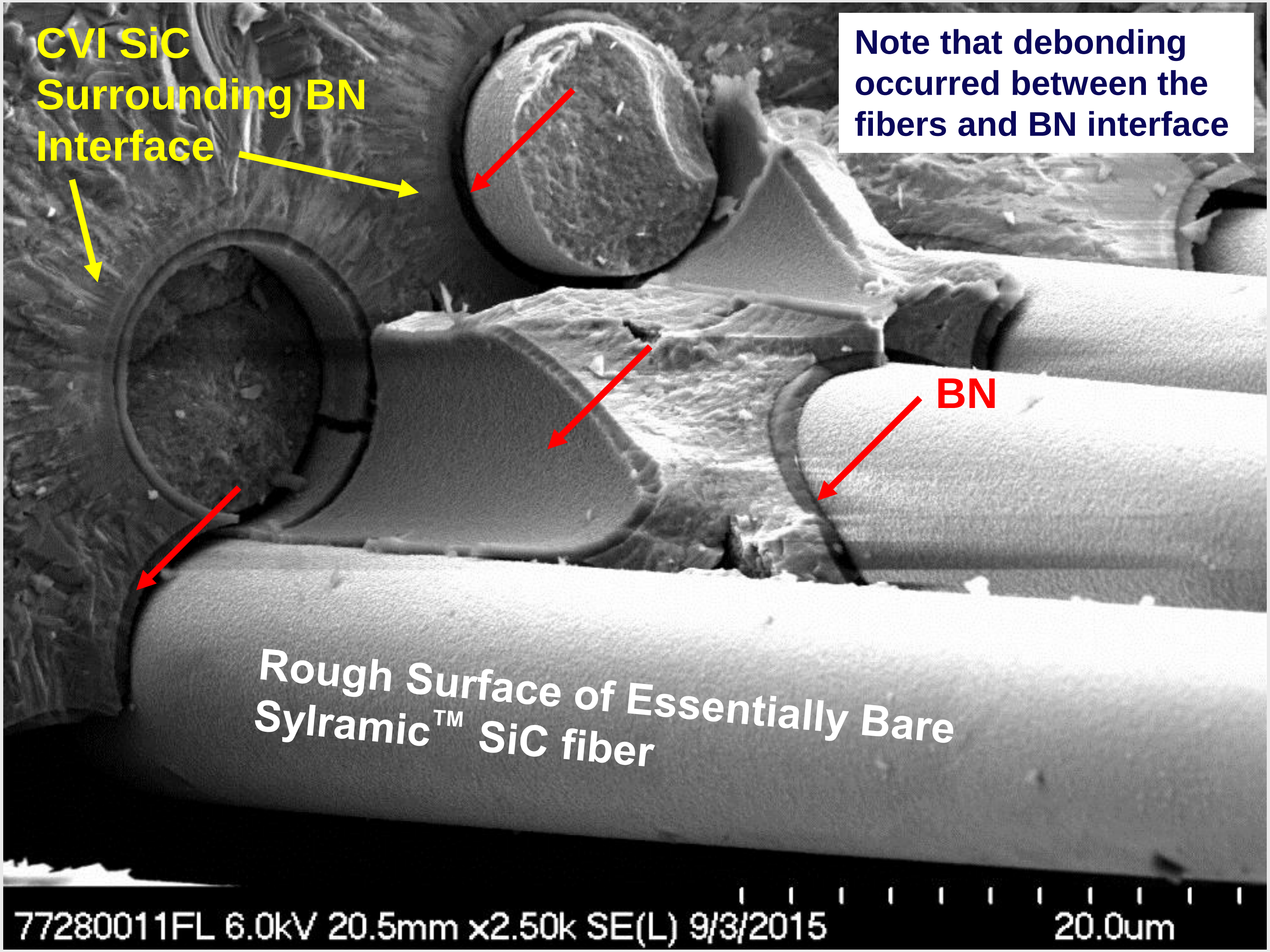
Fracture Surface—Examined With FESEM



Purpose: Examine fracture surface to establish a "baseline" for comparison with stressed oxidation sample fracture surfaces

1277-28-001-1 As-Fabricated MI SiC/SiC Material FF Tested at RT

Fracture Surface—Examined With FESEM



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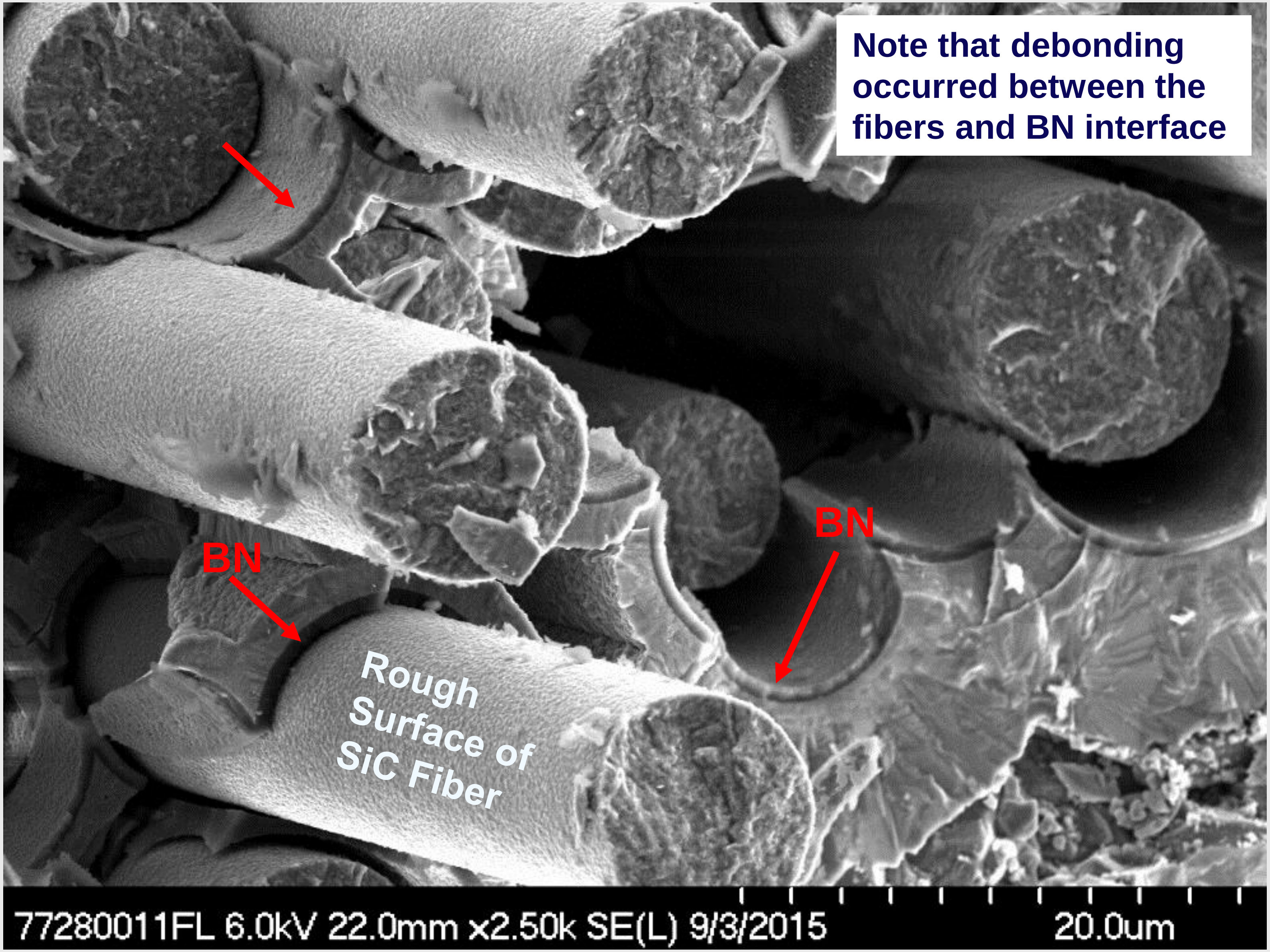

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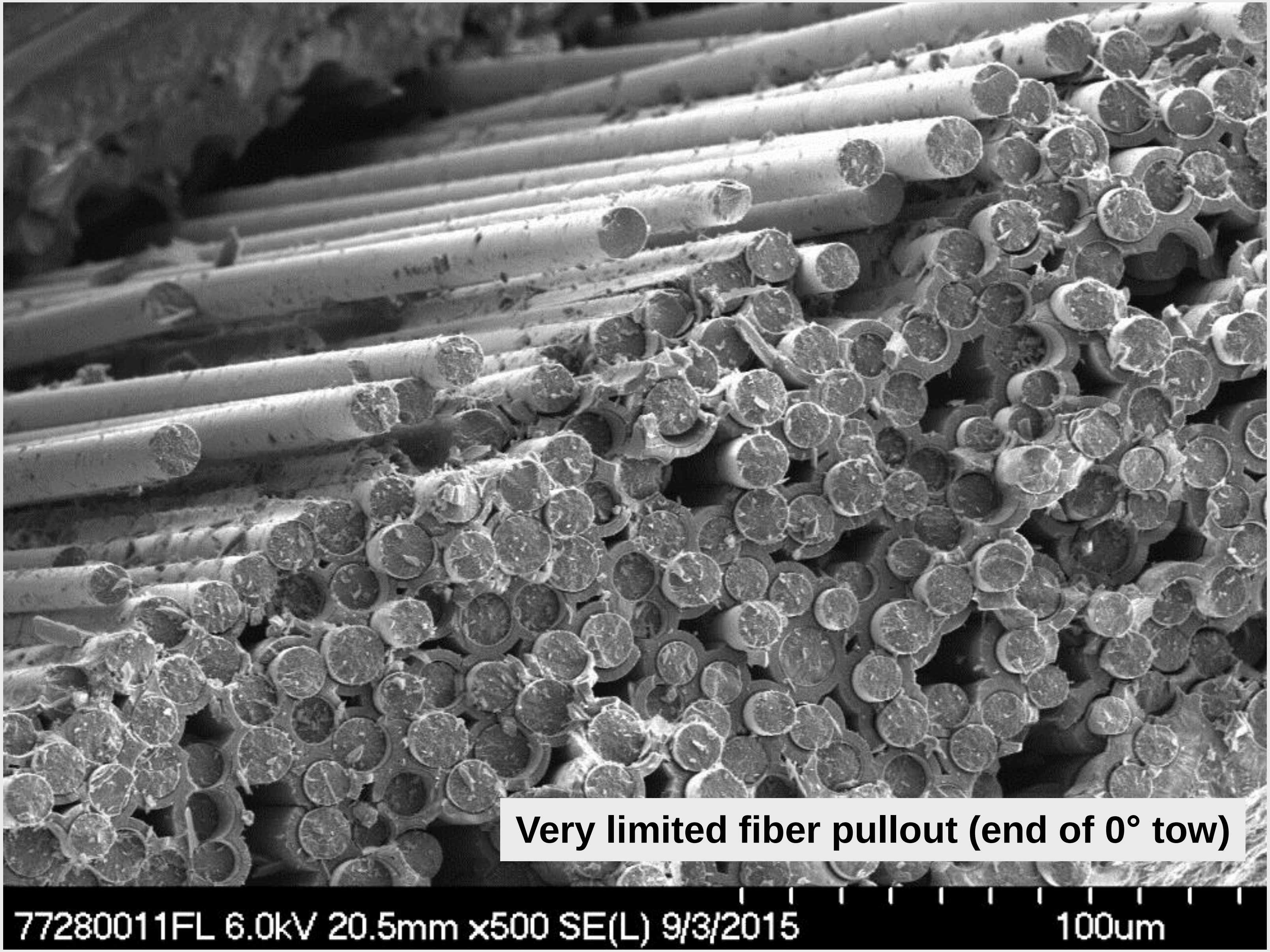

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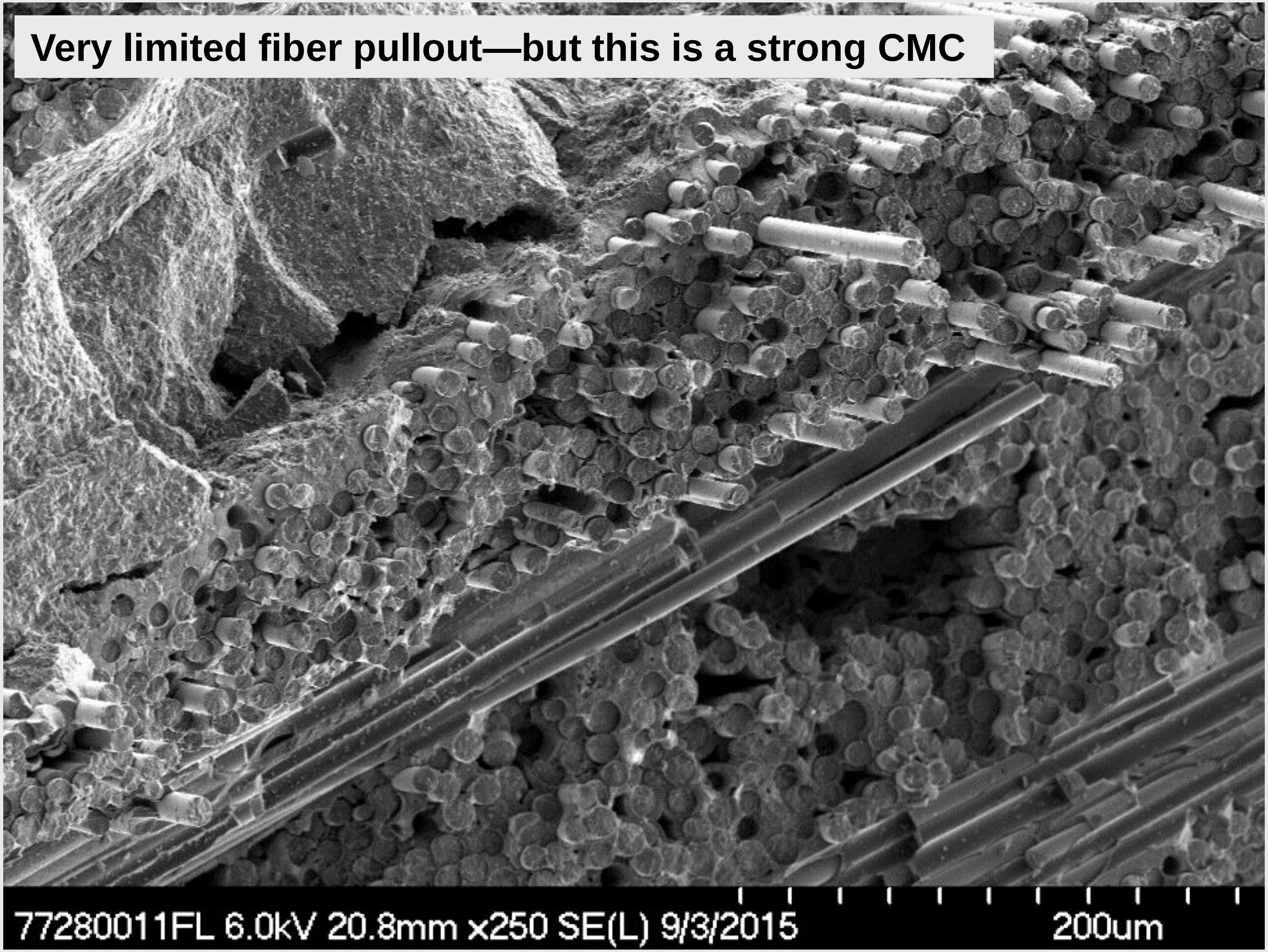


Assessing Degradation of MI SiC/SiC CMCs to *Support* Modeling Effort

V.

DIC Within SEM—Mature Approach With Higher Spatial Resolution (U. Michigan)

DIC: Microscale Approach (SEM)
Also a Learning Process—Different MI SiC/SiC Material

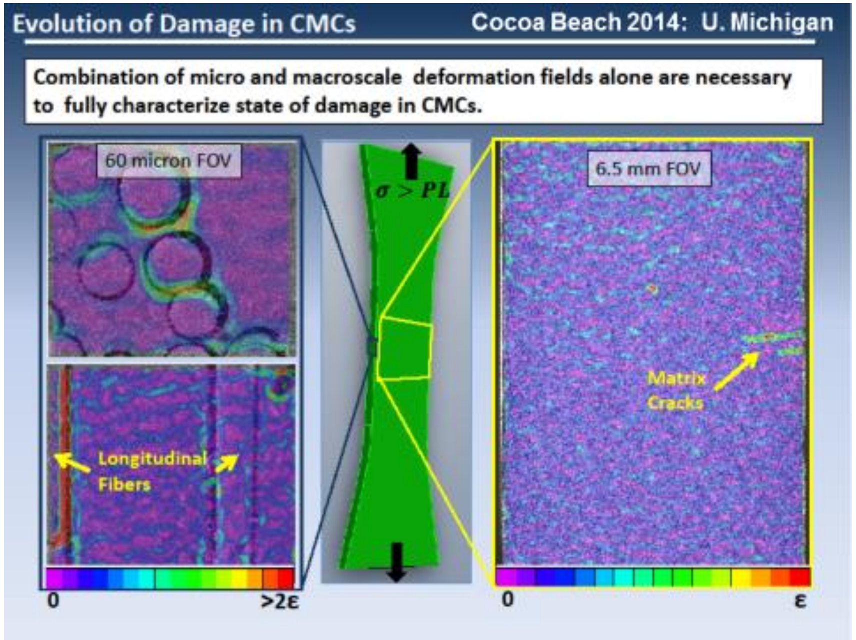
Examine CMC Cracking at RT and Measure Crack Opening Widths. Learn About Technique

Input for Oxidation Model: Cracking Behavior, Crack Opening Width, Τ (tau)

Making SiC/SiC Crack Opening Measurements—
U. of Michigan (UM) Utilized Small Loading Stage, SEM, and DIC

Problem and Approach Selected to Obtain COD Values

- **Crack opening displacement (COD) values (widths) were needed to support GRC modeling of the oxidation of SiC_f /SiC CMCs at Intermediate Temperatures (≈815ºC).**
- **Since these displacements were predicted to be *very small* (Refr. 2), GRC funded the U. of Michigan to utilize a small tensile loading fixture in an SEM (scanning electron microscope) to examine cracking in a melt infiltrated (MI) SiC_f /SiC.**



This technique has previously been used by UM in the characterization of cracking in MI SiC/SiC CMCs: "Characterization of Fracture in CMCs at the Microstructural Length Scale," J. Tracy and S. Daly, Cocoa Beach 2014

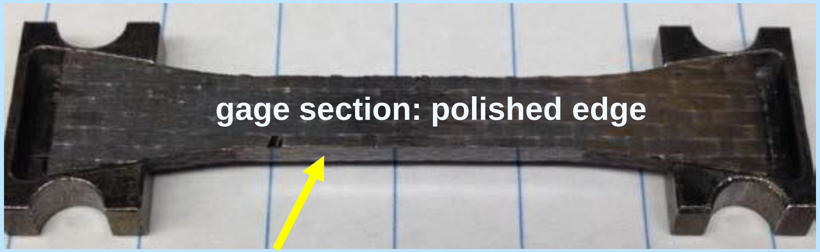
MichiganEngineering

Making SiC/SiC Crack Opening Measurements—
U. of Michigan (UM) Utilized Small Loading Stage, SEM, and DIC

Objective

- Using a MI SiC_f /SiC sample provided by GRC, determine when matrix cracking occurs, and collect images of cracks at specific stress levels (10 to 30 ksi).

2" SiC/SiC CMC tensile specimen



dovetail grip for edge loading

- These images and DIC (digital image correlation) would be used to determine crack opening displacement.

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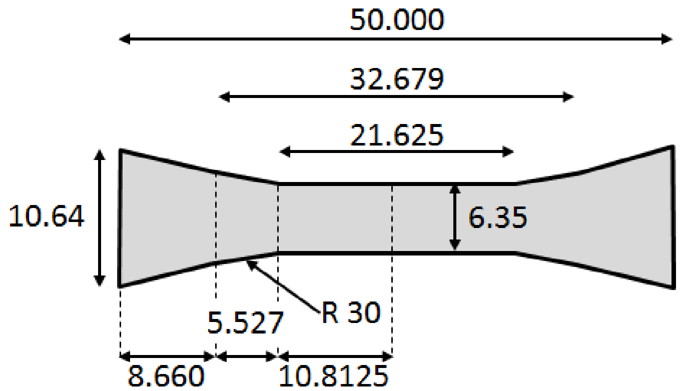

all measurements are in mm

Making SiC/SiC Crack Opening Measurements—
U. of Michigan (UM) Utilized Small Loading Stage, SEM, and DIC

Results

- SEM/DIC (digital image correlation) provided the ability to detect, observe, and image cracks on the polished edge of the sample at high magnification.
- First matrix cracking took place between 20 and 25 ksi, accompanied by an observable relaxation in strain near matrix cracks (detected using DIC).
- Following precracking at 25 ksi stress, images were captured at 10, 15, 20, 25, and 30 ksi.
- Matrix crack opening measurements at the maximum load (30 ksi) ranged from 0.2 to 1.5 µm.
- A report was provided to GRC, and a draft journal article has been prepared.
- Dr. Kathy Sevener presented this study at the Cocoa Beach 2016 Conference (Refr. 3).

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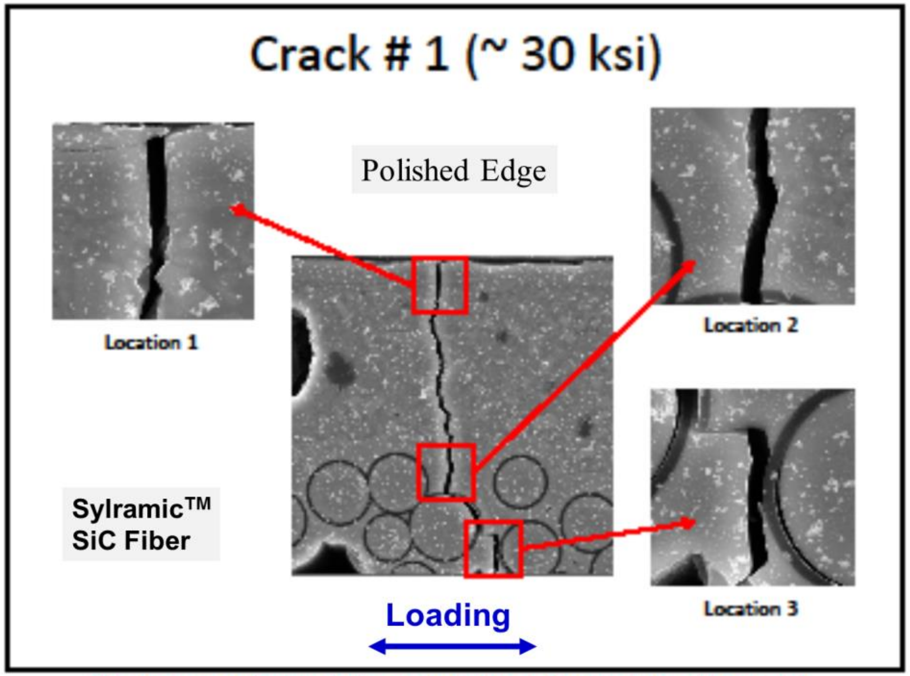

Measurements made in several locations along the crack

Summary and Future Plans

- A melt infiltrated (MI) SiC/SiC is being characterized (microstructure and RT mechanical properties) and oxidized / stress oxidation tested in dry air at 815ºC.
- This characterization is being performed to determine critical oxidation mechanisms and support the development of accurate models for the mechanical-oxidation-creep interactions that affect the strength and life of SiC_f /BN/SiC CMCs.
- In the future we might transition to characterizing a 2D Hybrid SiC/SiC (CVI and PIP matrix) with either Sylramic™-iBN SiC fiber or Hi Nic S SiC fiber reinforcement, because that material is more relevant to our current GRC focus on 2700°F CMCs.

References

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