2023 FECM/NETL SPRING R&D PROJECT REVIEW MEETING

Track 2: Advanced Energy Materials for Hydrogen Turbines (Advanced Energy Materials)

Moderator:
Heather Hunter
April 18, 2023

Low-Cost/High Performance Silicon Carbide Composite Shells for Gas Turbines

U.S. DEPARTMENT OF ENERGY
Fossil Energy and Carbon Management

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DOE SBIR Phase I Award: SC0022704

Design, Modeling, and Experimental Validation for life-optimization of Hydrogen Turbine CMC Components

Joseph Pegna

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United States

www.fffibers.com

Main takeaways:

BACKGROUND INFORMATION / PREREQUISITE:

- Laser Printed, High Purity / Low Cost SiC fibers.
- Micro-Trellis [1]: *3-D non-woven fiber architecture*
- Embedded Wire Chemical Vapor Deposition [2]
  - *Fast, low-cost, CVI/CVD for SiC-SiC CMC shells*
- Joining [2]
  - *SiC-SiC / Metal Joining*
  - *SiC-SiC homogeneous joining*
- SiC-SiC Composites = 'wild west'. *Urgent need for QA standards*

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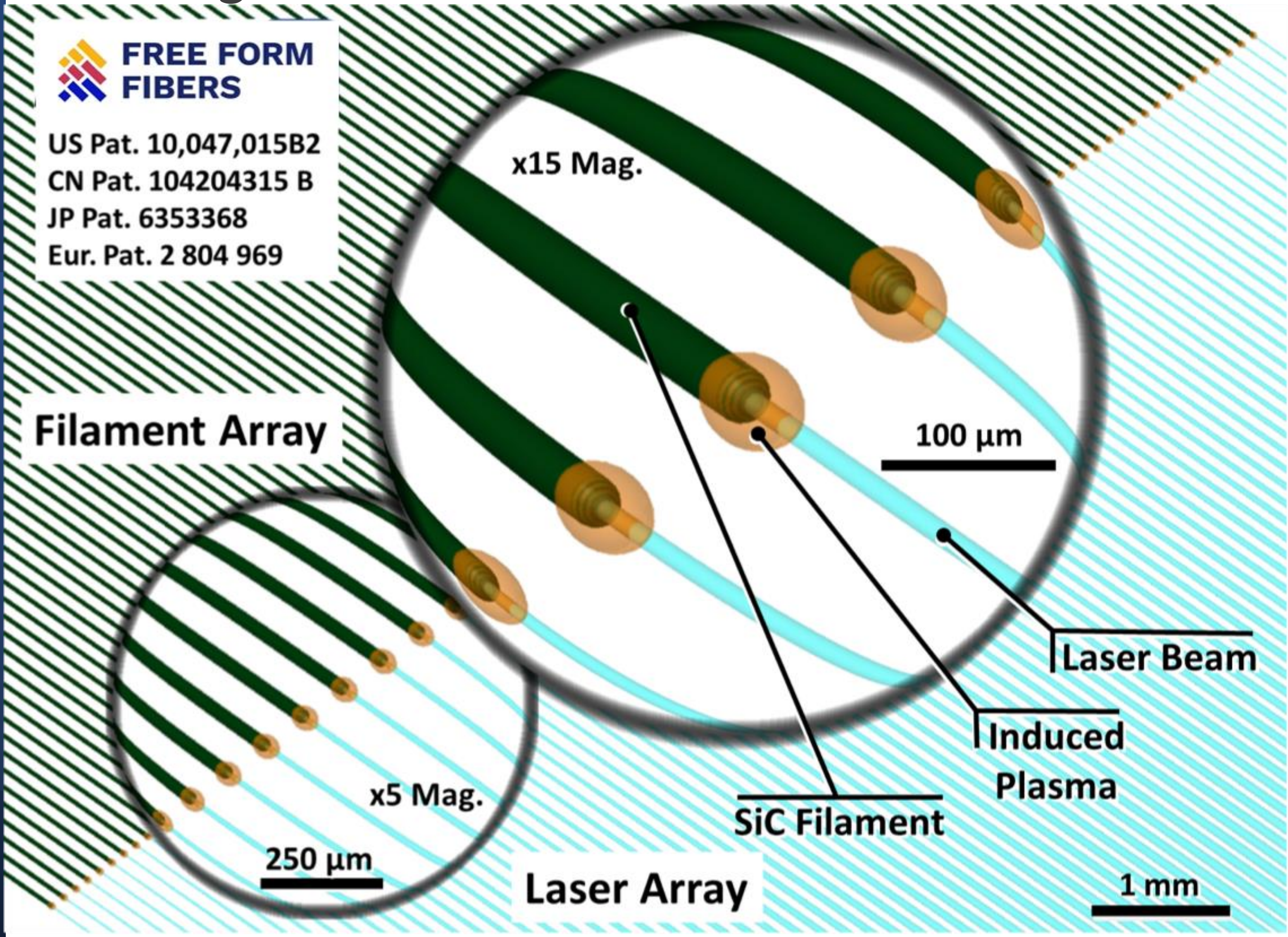
(1): US Pat. No. 11,362,256 B2

(2): US Pat. Application 17/661,059 & PCT Publication No. WO 2023/278905 A1, January 5, 2023

Overview

- Laser-Printed SiC Fibers
- SiC Non-Woven and Micro-Trellis
- Embedded Wire CVD
  - Metal / SiC-SiC joining
  - SiC-SiC Homogeneous Joining
- SC0022704 (Lifing) Applications:
  - Microcomposites
  - Microcomposite testing
  - Commercialization strategy

Laser-Printed Fibers:
Lab stage in 2006



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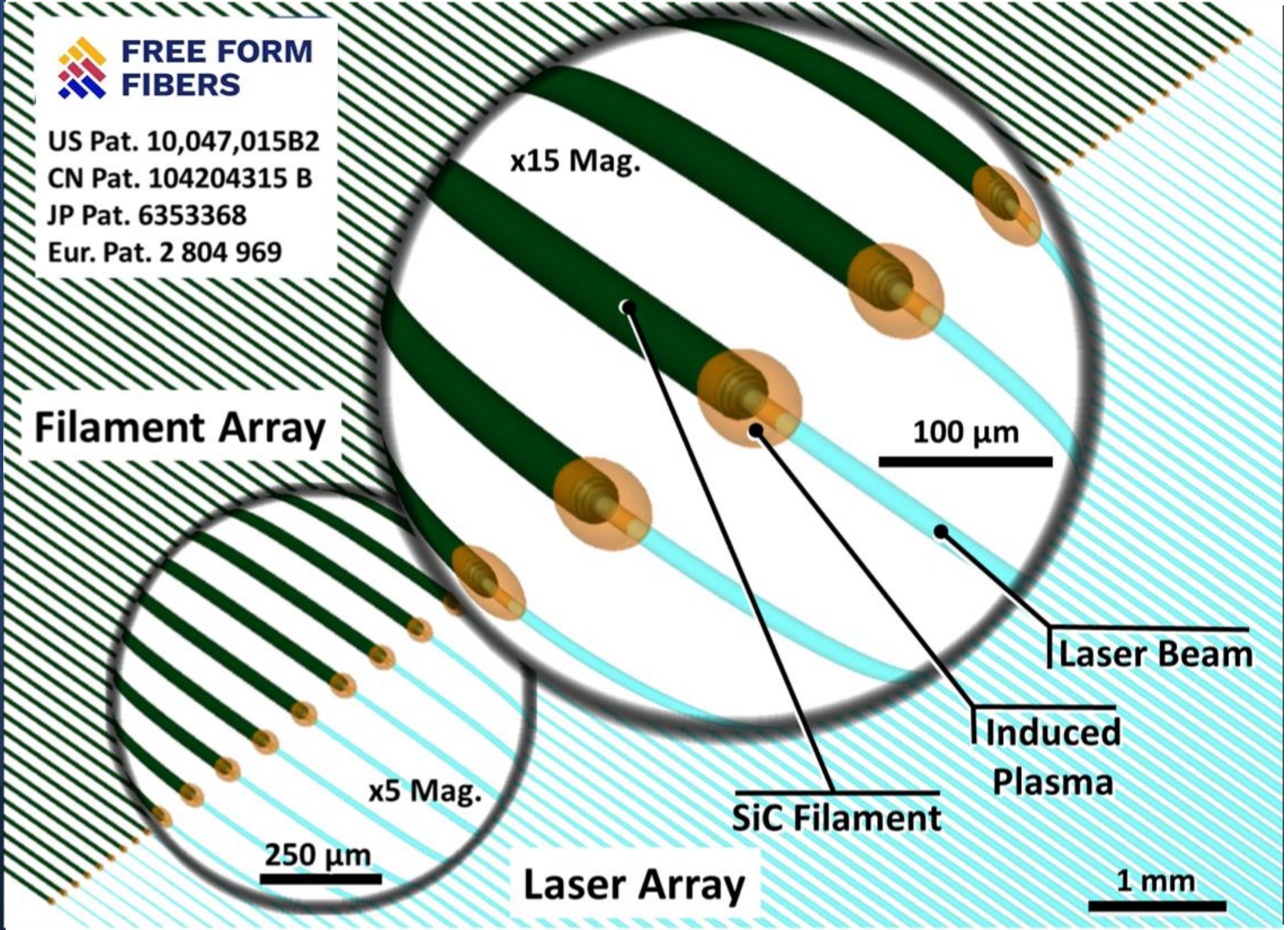
Laser-Printed Fibers:
TRL 4 in 2011



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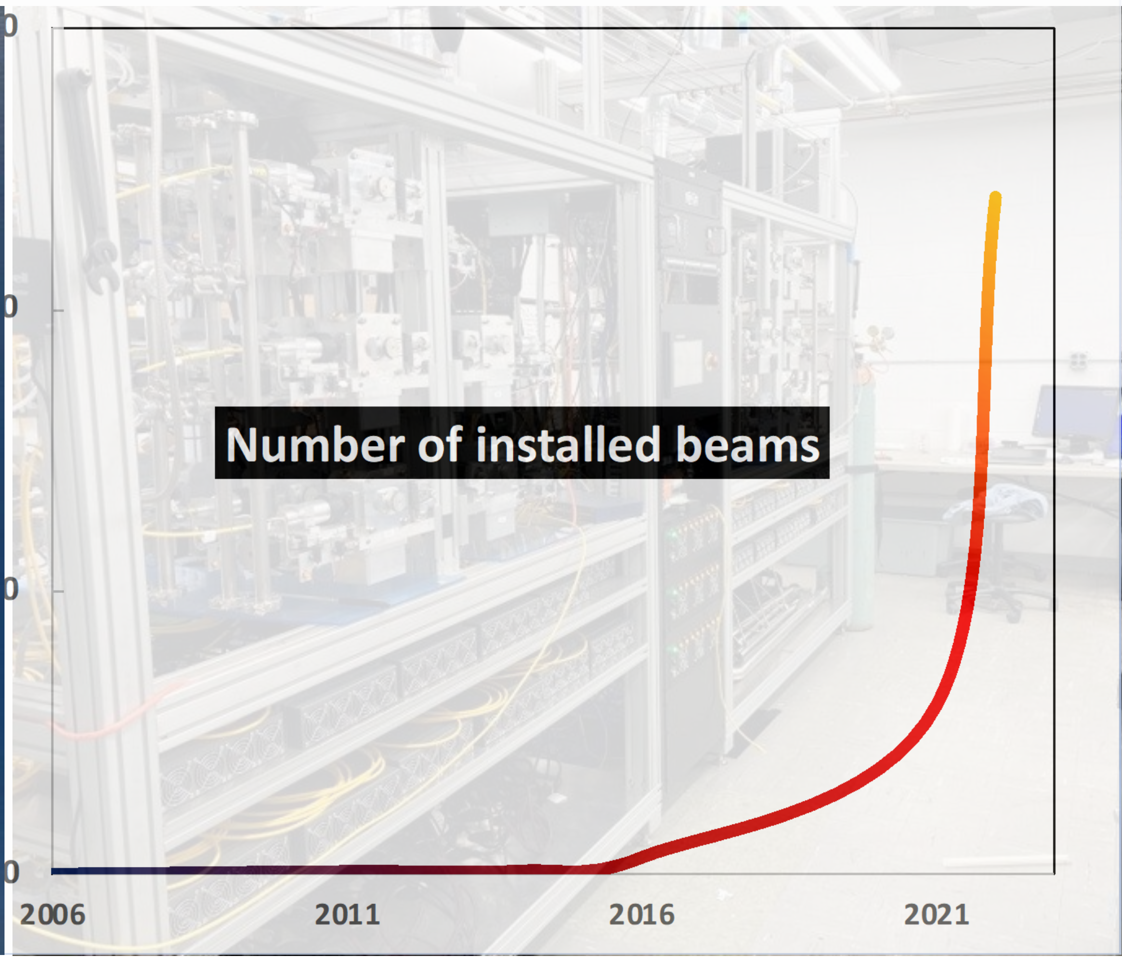
Laser-Printed Fibers:

Prototype Production Starting in 2021



c. 2023: 10,000+ beams

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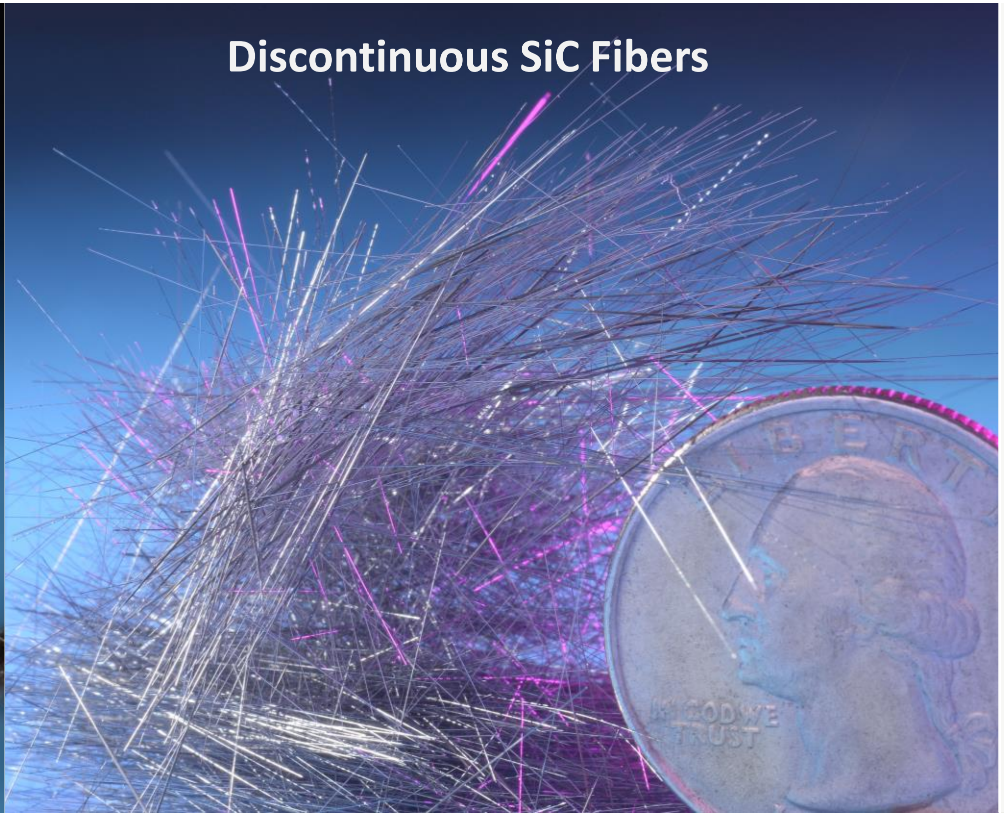
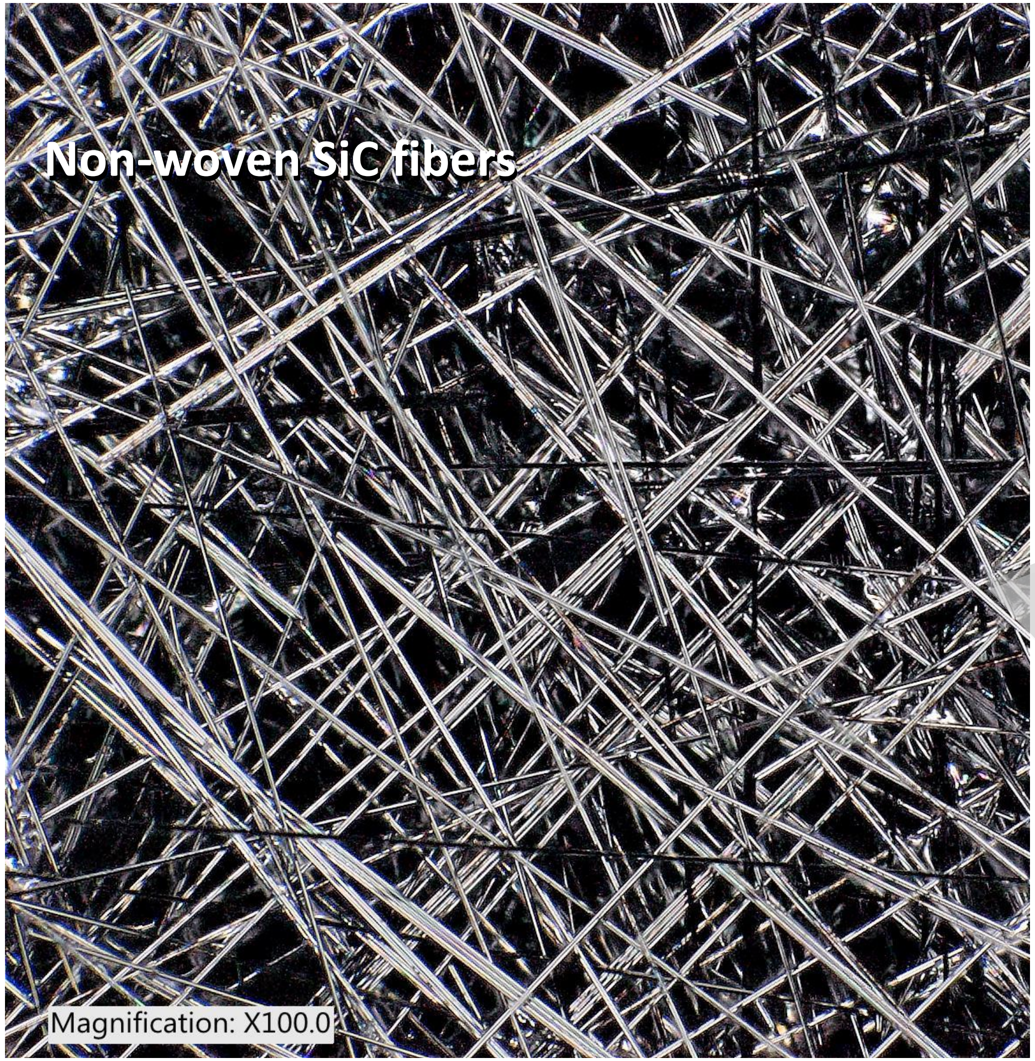
Laser-Printed Fibers:
Line of Products

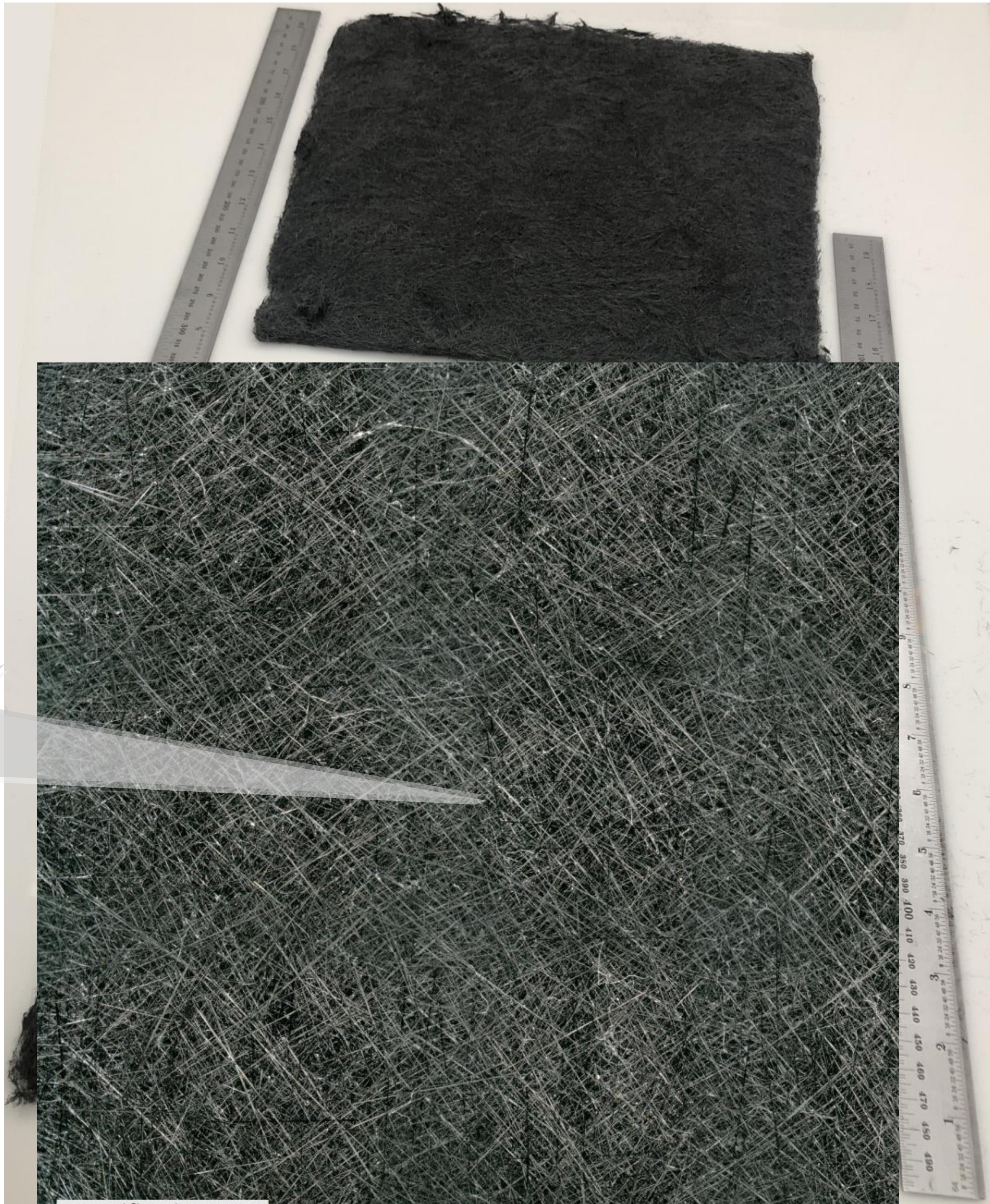

High-Purity SiC Powders


Discontinuous SiC Fibers


Continuous SiC Fiber Ribbons

Laser-Printed Fibers:
Line of Products





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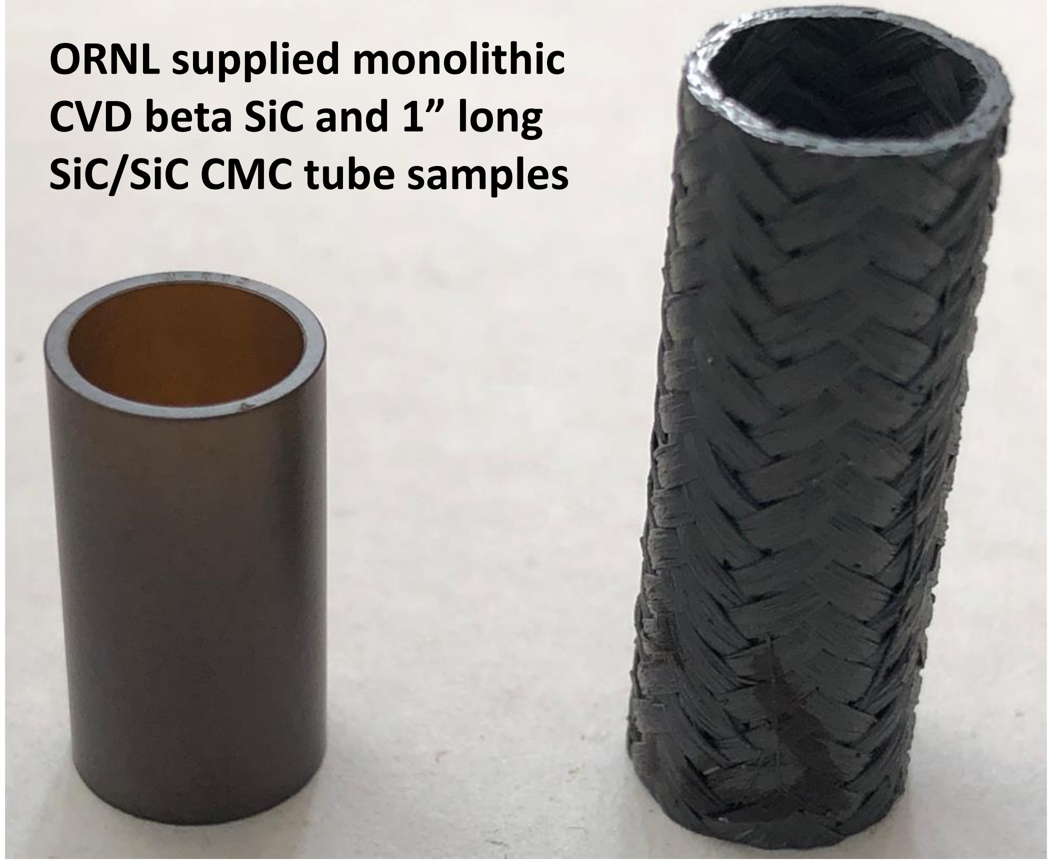
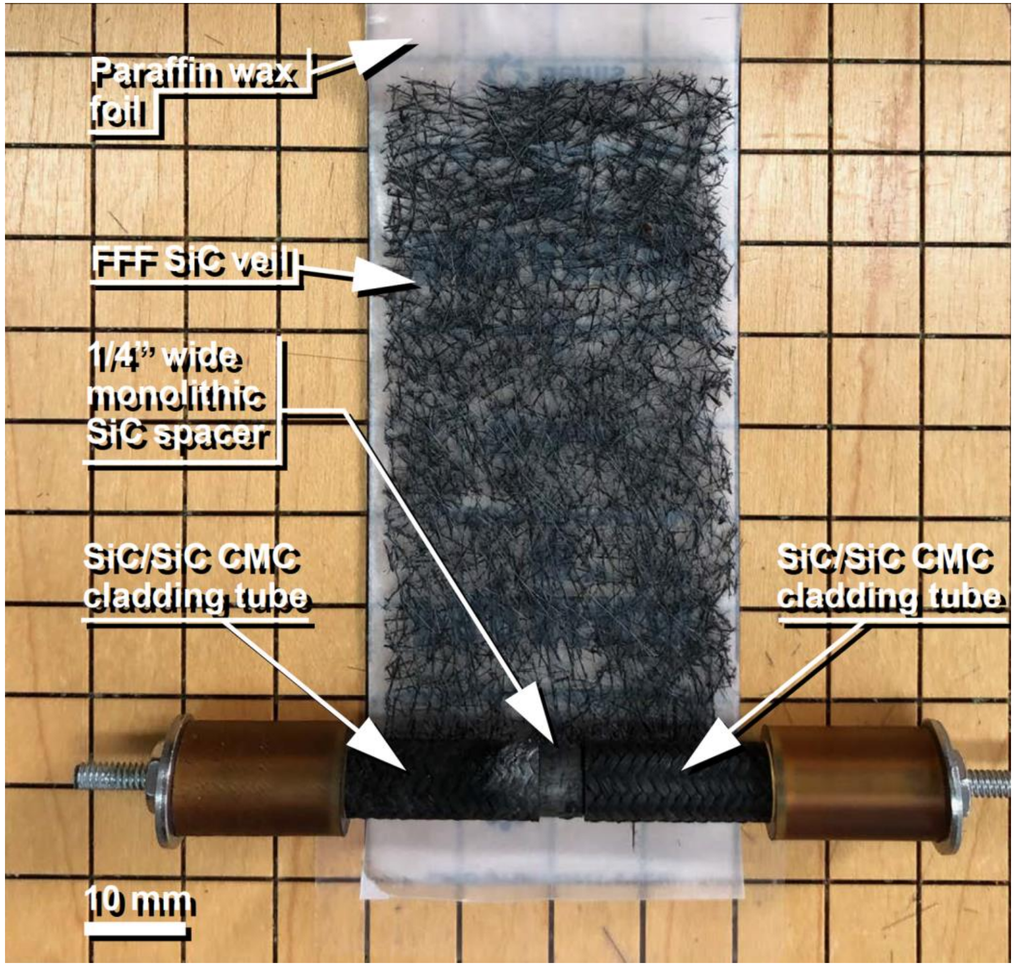
Homogeneous Joining of SiC-SiC Composites (DE-SC0021665)

Sample Preparation



Diamond-sawed tube sections



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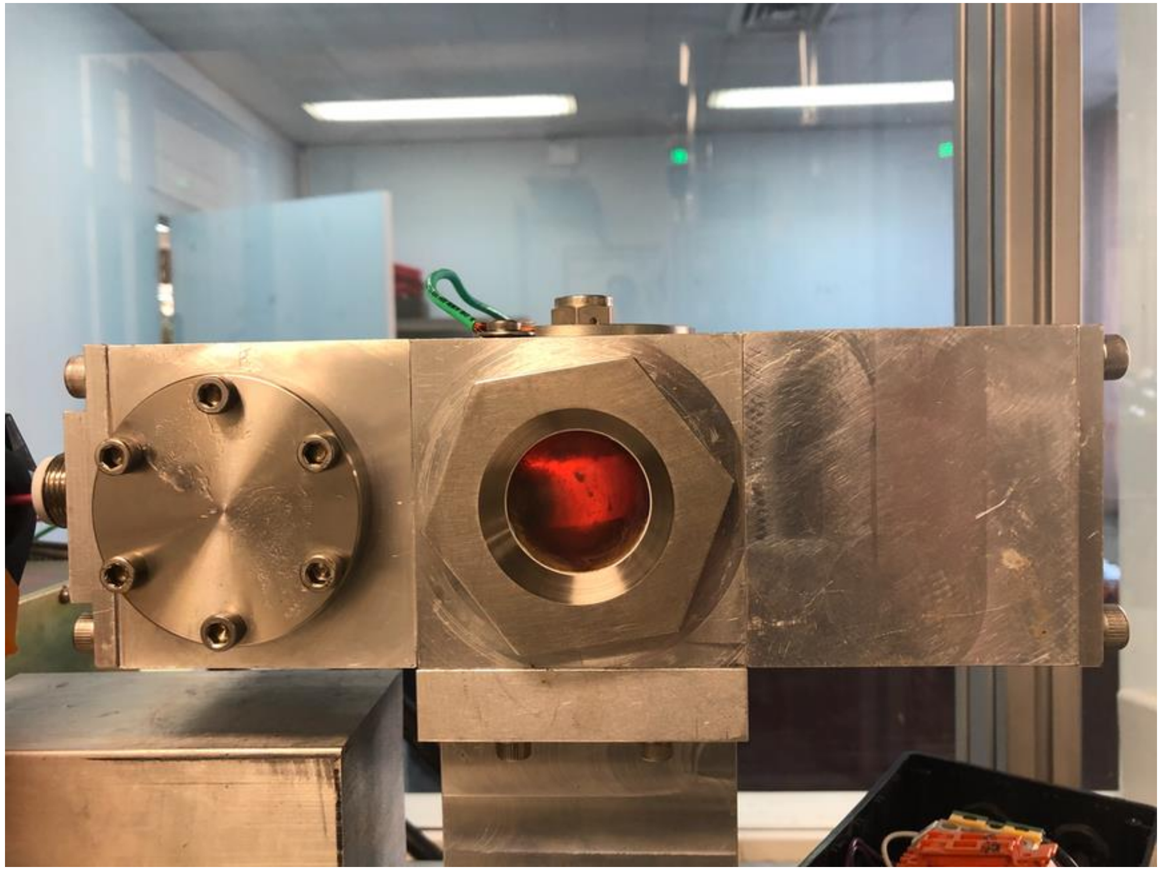
Homogeneous Joining of SiC-SiC Composites

Objectives:
- **Lap Joint**
- **Butt Joint**

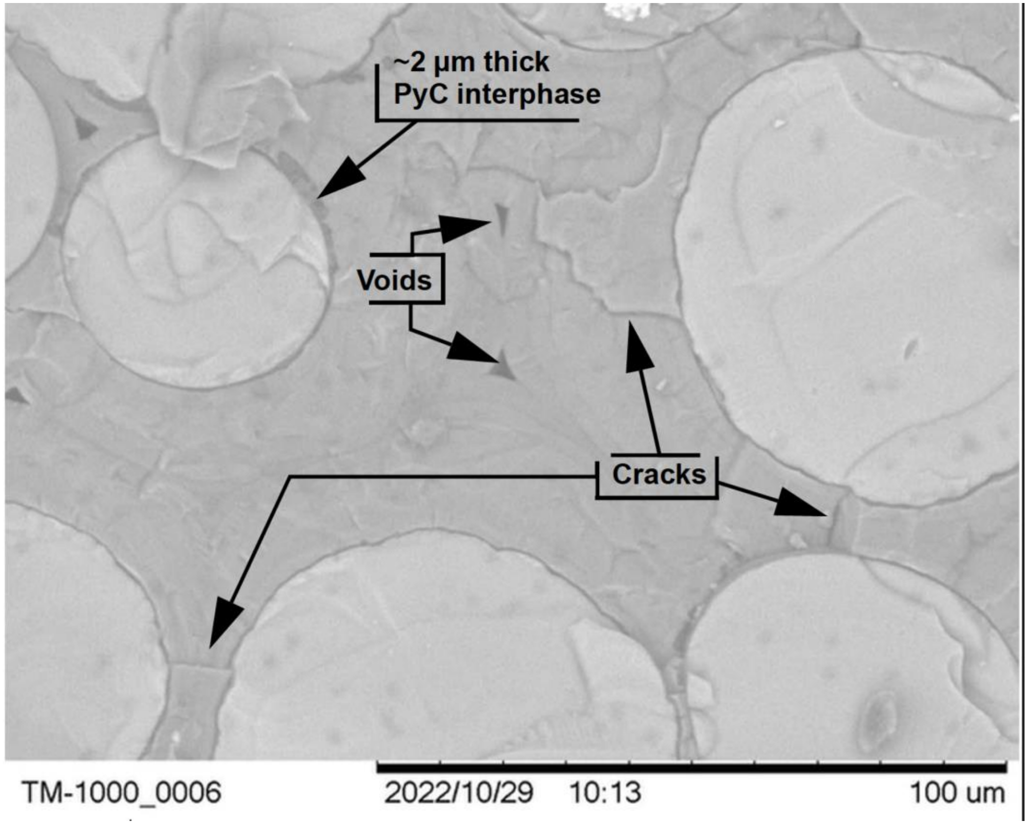
Lap

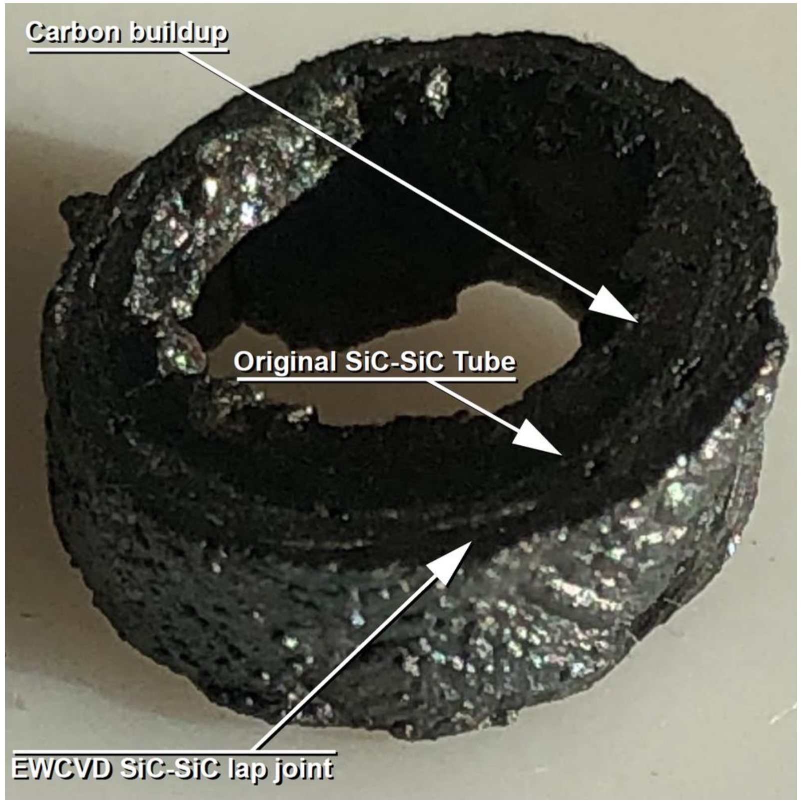
Butt



Homogeneous Joining of SiC-SiC Composites

Destructive SEM Inspection





Flexural failure surface inspection in butt-joint region

Diamond-sawed slice in the lap joint region

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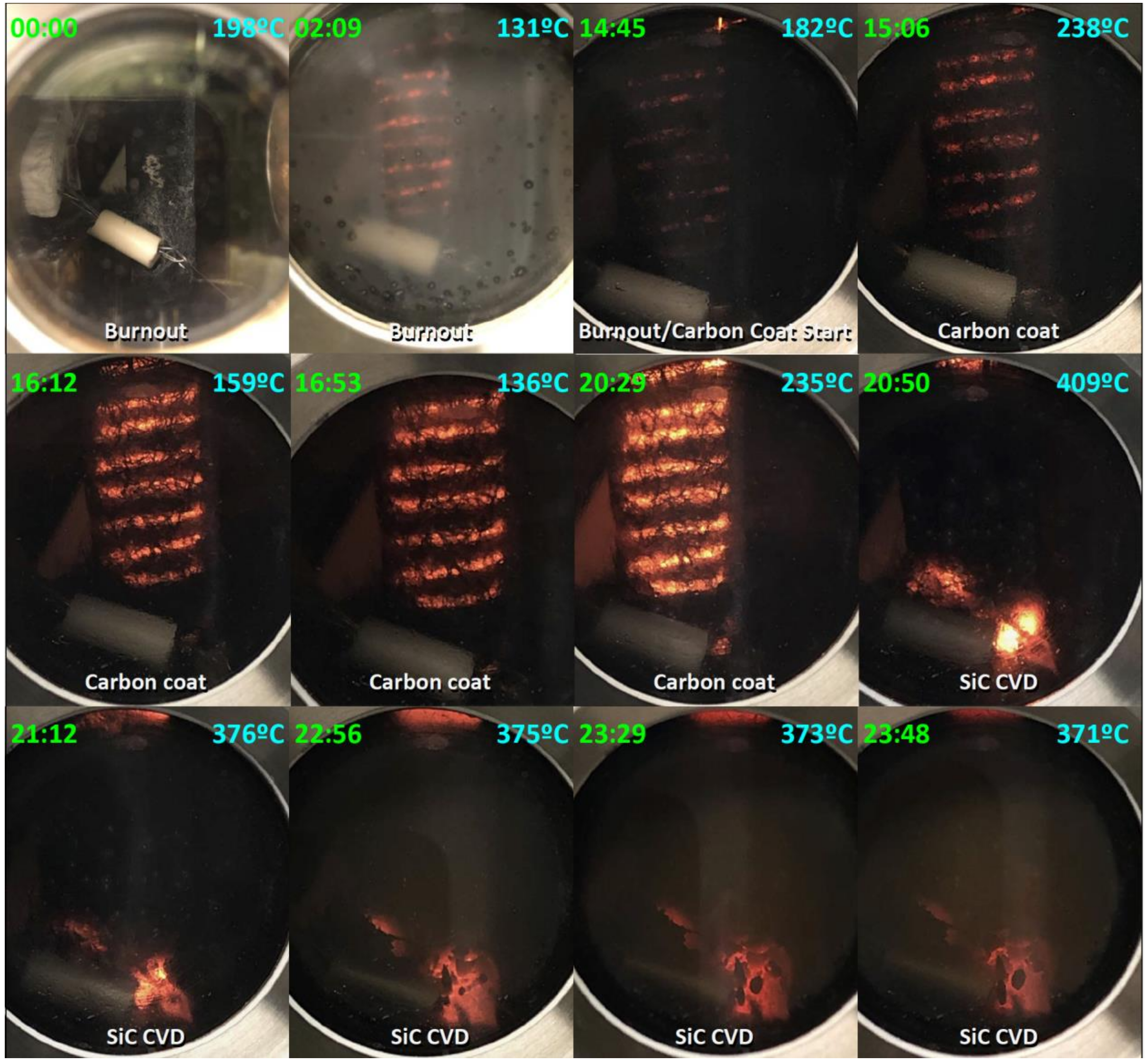
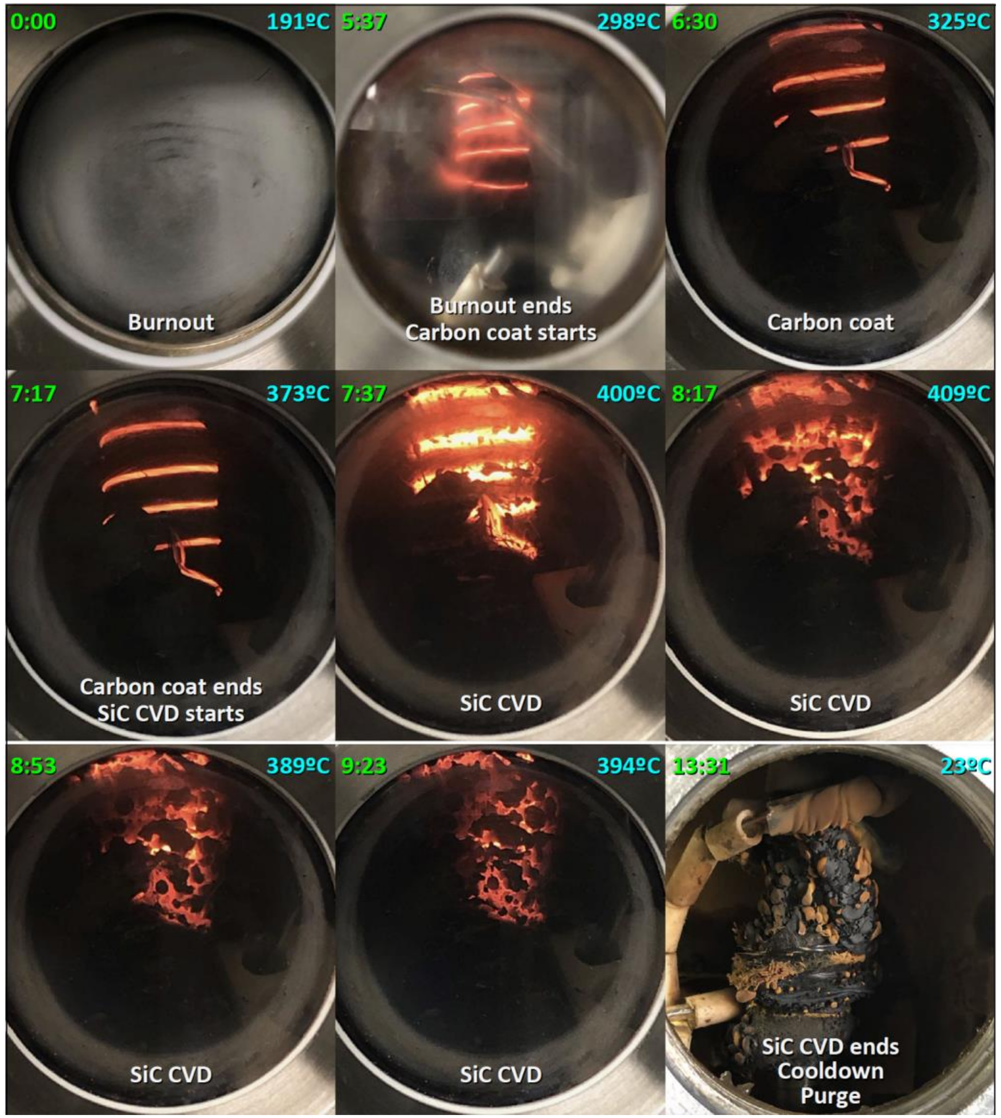
Embedded Wire CVD (EWCVD -- DE-SC0019912): *Processing*



Zone 1 time lapse: Total cycle time 23h48 min, Max Temp. 409ºC



Zone 2 time lapse: Total cycle time 13h31 min, Max Temp. 409ºC

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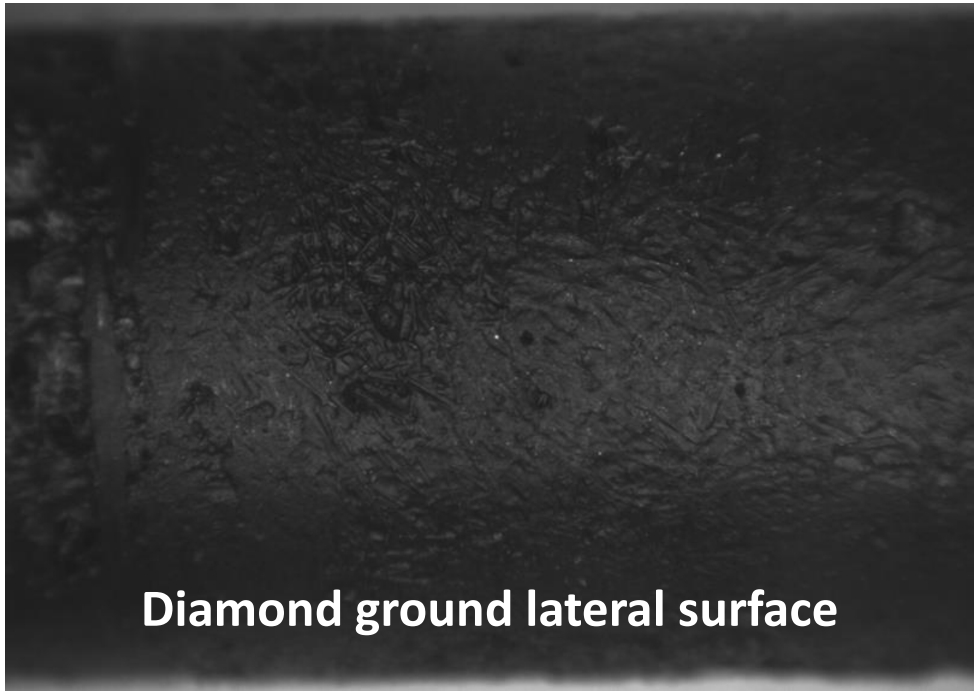
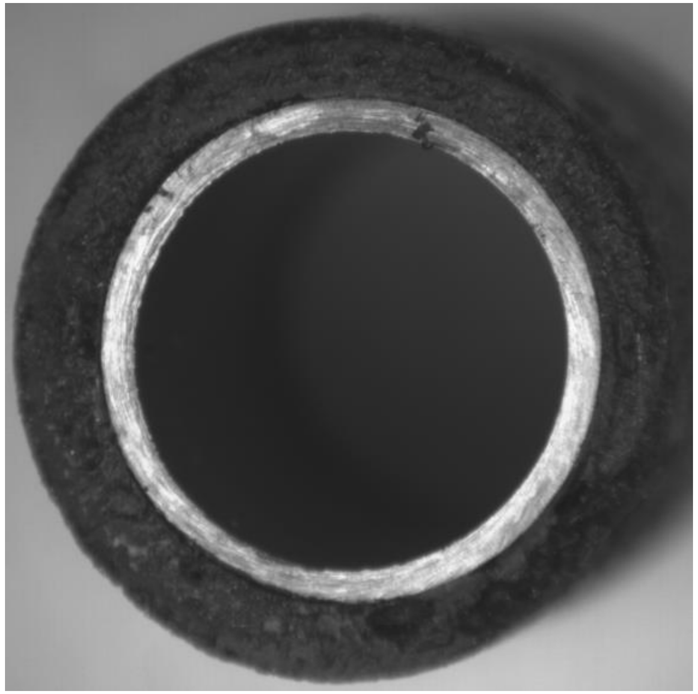
Embedded Wire CVD (EWCVD): *Finishing & Inspection*

Zone 1 Diamond sawed



Diamond ground lateral surface



Zone 2 Diamond sawed

Optical Microscope Views:

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Award: SC0022704: *Design, Modeling, and Experimental Validation for life-optimization of Hydrogen Turbine CMC Components*

Topic 21.e mandate:

Improved Lifing Models for CMC. *This research topic would develop models of environmental degradation of CMC materials in the hot section of the main gas path of a hydrogen turbine.*

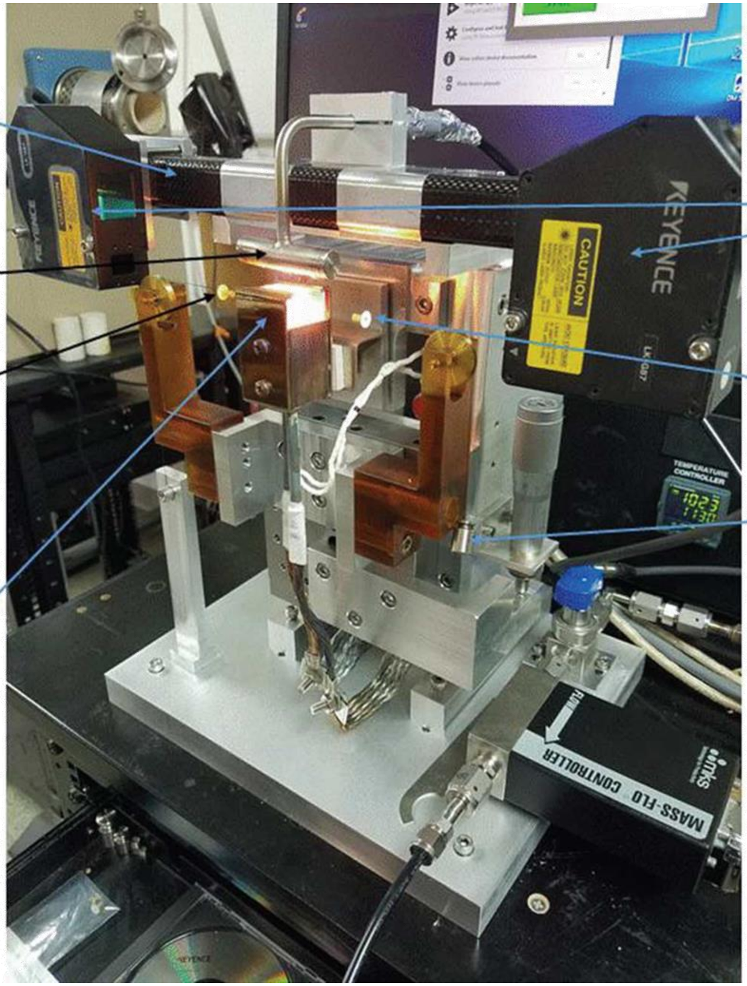
Technical:

- **Reduce size and variability of testing coupons**
- **Standardize coupon & testing procedures**
- **Use results to calibrate CIME models**
- **Build a material database for later AI/ML**

Commercial:

- **No H_2 gas turbines in the world**
- **How does one commercialize a model?**

SC0022704 -- Technical



High-strength fishing lines glued to either end of test fiber near position flags. Lines go around low friction pulleys. Left line is fixed, right line is weighted. Test fiber can be lowered into micro-heater gap via vernier micrometer

Materials Performance and Characterization

doi:10.1520/MPC20200131 / Vol. 10 / No. 2 / 2021 / available online at www.astm.org

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High-Temperature Performance of Next-Generation Silicon Carbide Fibers for CMCs

Reference

S. Harrison, J. Schneiter, J. Pegna, E. Vaaler, R. Goduguchinta, and K. Williams, "High-Temperature Performance of Next-Generation Silicon Carbide Fibers for CMCs," *Materials Performance and Characterization* 10, no. 2 (2021): 207–223. https://doi.org/10.1520/MPC20200131

ABSTRACT

Single-fiber (monofilament, not twisted tow) high-temperature creep performance of refractory ceramic compositions is a fundamental material property that must be examined and understood for the successful deployment of ceramic matrix composite material systems in demanding applications. A novel creep testing approach was devised utilizing an innovative approach to fiber sample attachment and used to evaluate the creep behavior of Free Form Fibers' LP-30SC and Nippon Carbon-based Hi-Nicalon Type S (HNS) silicon carbide fiber products. The testing apparatus, analytical basis for the creep calculations, including the sample gage length, and collected data are presented. Test conditions include a range of peak hold temperatures, from 1,300°C to 1,500°C, gas environments, and hold time patterns at the peak temperature. Clear differences in resistance to creep degradation were evident between the LP-30SC and HNS fibers, as the LP-30SC demonstrated strong resiliency to high temperature exposure while the HNS showed performance degradation consistent with oxidation attack.

Keywords

silicon carbide fiber, creep behavior, single fiber, elevated temperature performance

Introduction

The creep performance of fibers intended for use as the reinforcement phase of ceramic matrix composites (CMCs) in high-temperature applications (nominally greater than 1,100°C) is an extremely important material property for the successful implementation of CMCs. Evaluation of the creep behavior requires carefully controlled experimentation

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Build upon prior experience

Single filament fiber creep measurement*

One unit sold to University Lab. In 2023

S. Harrison, J. Schneiter, J. Pegna, E. Vaaler, R. Goduguchinta, and K. Williams, "High-Temperature Performance of Next-Generation Silicon Carbide Fibers for CMCs," Materials Performance and Characterization 10, no. 2 (2021): 207–223. https://doi.org/10.1520/MPC20200131

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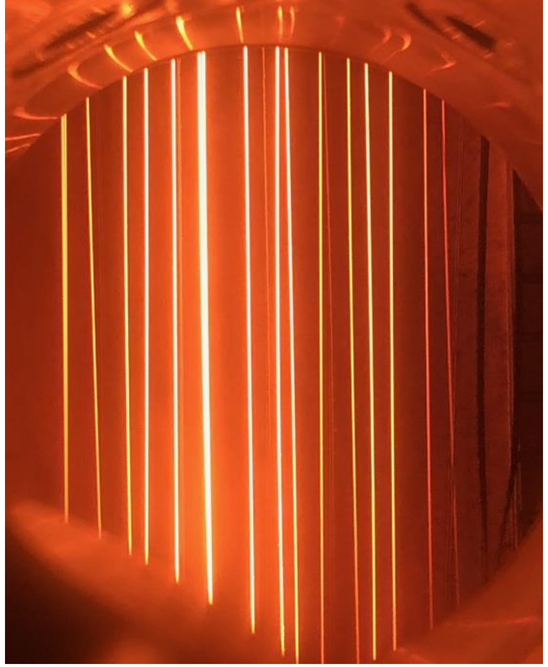
SC0022704 -- Technical

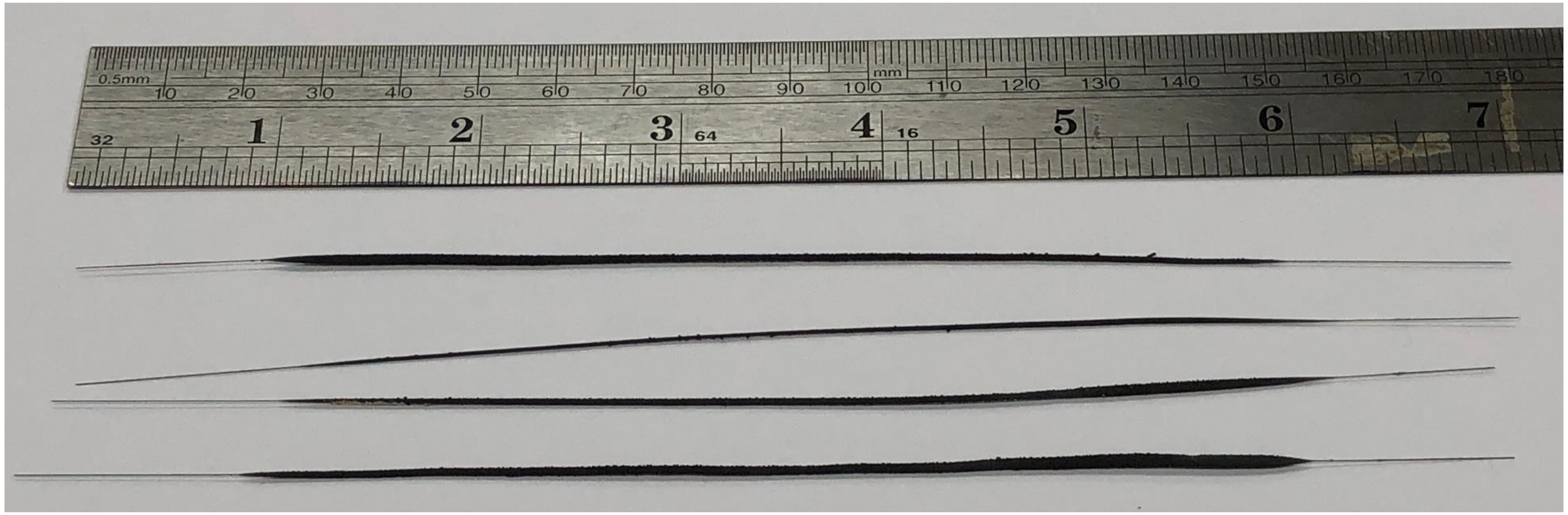
- Reduce size and variability of testing coupons
  - Demonstrated micro-composite (one fiber composite with interphase and matrix shell.)



Microcomposite fabrication by EWCVD (using carbon coat as resistive element)

Microcomposite coupons as produced.

- Single filament SiC
- Carbon interphase
- SiC matrix shell



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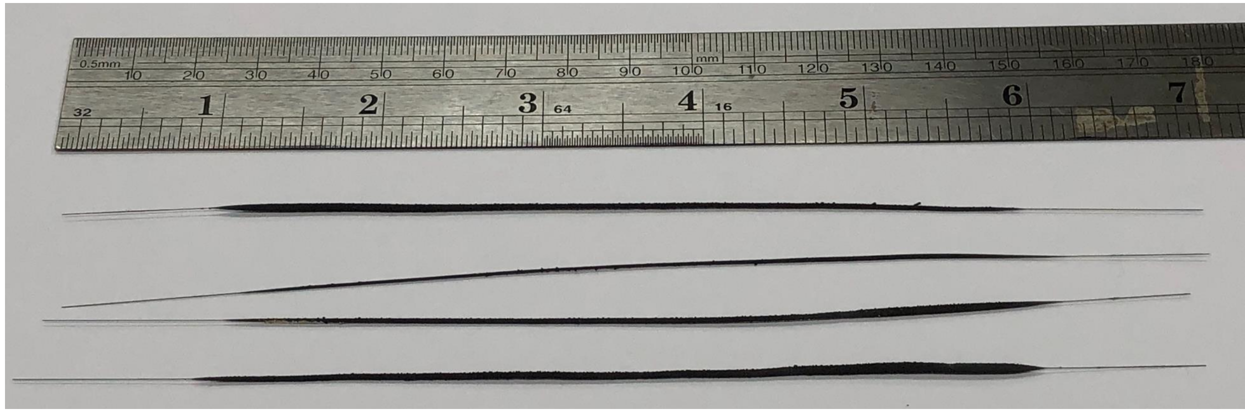
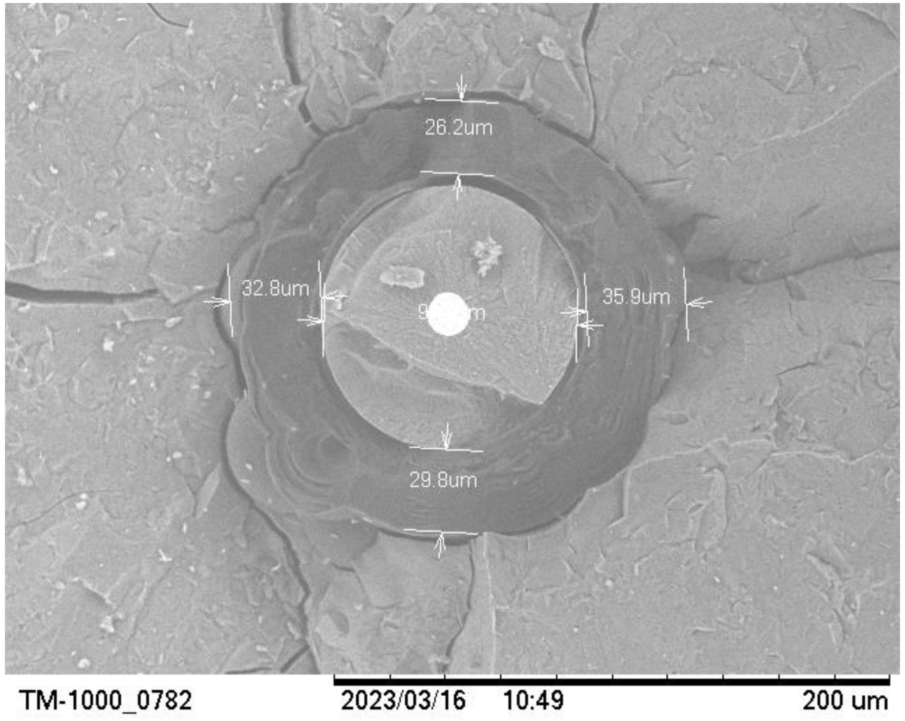
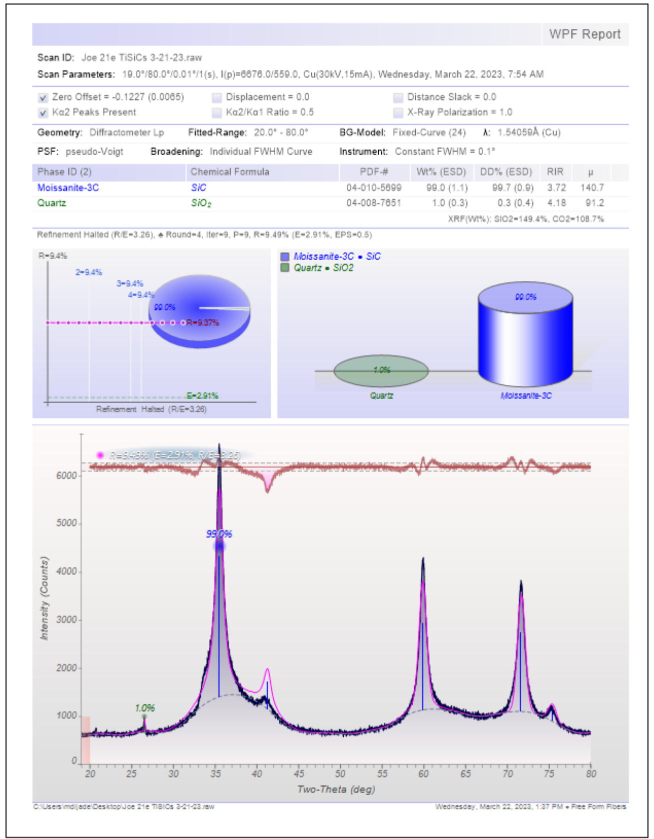
SC0022704 -- Technical



- SEM Inspection
- Materials Analysis





WPF Report

Scan ID: Joe 21e TiSiCs 3-21-23.raw

Scan Parameters: 19.0°/80.0°/0.01°/1(s), I(p)=6676.0/559.0, Cu(30kV,15mA), Wednesday, March 22, 2023, 7:54 AM

Zero Offset = -0.1227 (0.0065) | Displacement = 0.0 | Distance Slack = 0.0

Kα2 Peaks Present | Kα2/Kα1 Ratio = 0.5 | X-Ray Polarization = 1.0

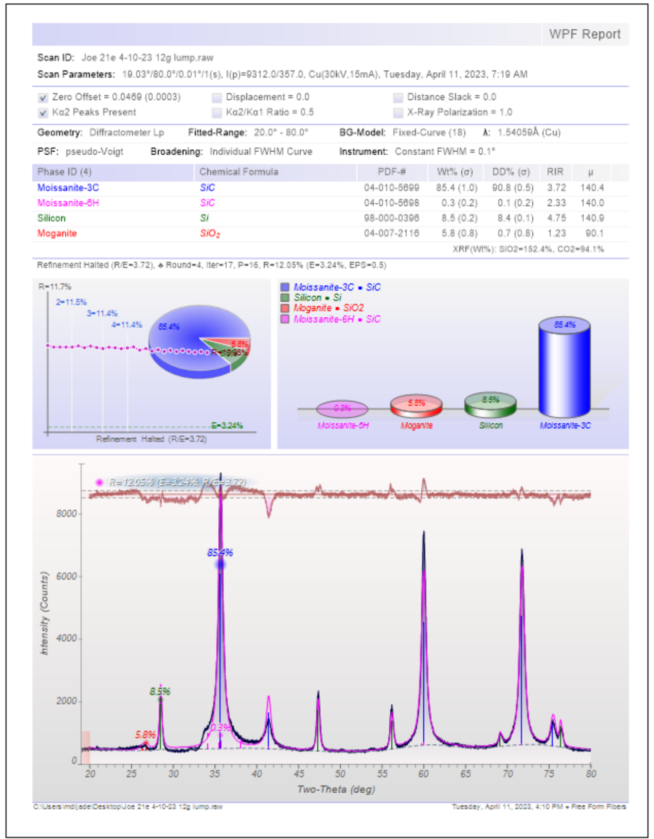
Geometry: Diffractometer Lp **Fitted-Range:** 20.0° - 80.0° **BG-Model:** Fixed-Curve (24) **λ:** 1.54059Å (Cu)

PSF: pseudo-Voigt **Broadening:** Individual FWHM Curve **Instrument:** Constant FWHM = 0.1°

Phase ID (2)	Chemical Formula	PDF-#	Wt% (ESD)	DD% (ESD)	RIR	μ
Moissanite-3C	SiC	04-010-5699	99.0 (1.1)	99.7 (0.9)	3.72	140.7
Quartz	SiO_2	04-008-7651	1.0 (0.3)	0.3 (0.4)	4.18	91.2

XRF(Wt%): SiO2=149.4%, CO2=108.7%

Refinement Halted (R/E=3.26), ♣ Round=4, Iter=9, P=9, R=9.49% (E=2.91%, EPS=0.5)



WPF Report

Scan ID: Joe 21e 4-10-23 12g lump.raw

Scan Parameters: 19.03°/80.0°/0.01°/1(s), I(p)=9312.0/357.0, Cu(30kV,15mA), Tuesday, April 11, 2023, 7:19 AM

Zero Offset = 0.0469 (0.0003) | Displacement = 0.0 | Distance Slack = 0.0

Kα2 Peaks Present | Kα2/Kα1 Ratio = 0.5 | X-Ray Polarization = 1.0

Geometry: Diffractometer Lp **Fitted-Range:** 20.0° - 80.0° **BG-Model:** Fixed-Curve (18) **λ:** 1.54059Å (Cu)

PSF: pseudo-Voigt **Broadening:** Individual FWHM Curve **Instrument:** Constant FWHM = 0.1°

Phase ID (4)	Chemical Formula	PDF-#	Wt% (σ)	DD% (σ)	RIR	μ
Moissanite-3C	SiC	04-010-5699	85.4 (1.0)	90.8 (0.5)	3.72	140.4
Moissanite-6H	SiC	04-010-5698	0.3 (0.2)	0.1 (0.2)	2.33	140.0
Silicon	Si	98-000-0396	8.5 (0.2)	8.4 (0.1)	4.75	140.9
Moganite	SiO_2	04-007-2116	5.8 (0.8)	0.7 (0.8)	1.23	90.1

XRF(Wt%): SiO2=152.4%, CO2=94.1%

Refinement Halted (R/E=3.72), ♣ Round=4, Iter=17, P=16, R=12.05% (E=3.24%, EPS=0.5)

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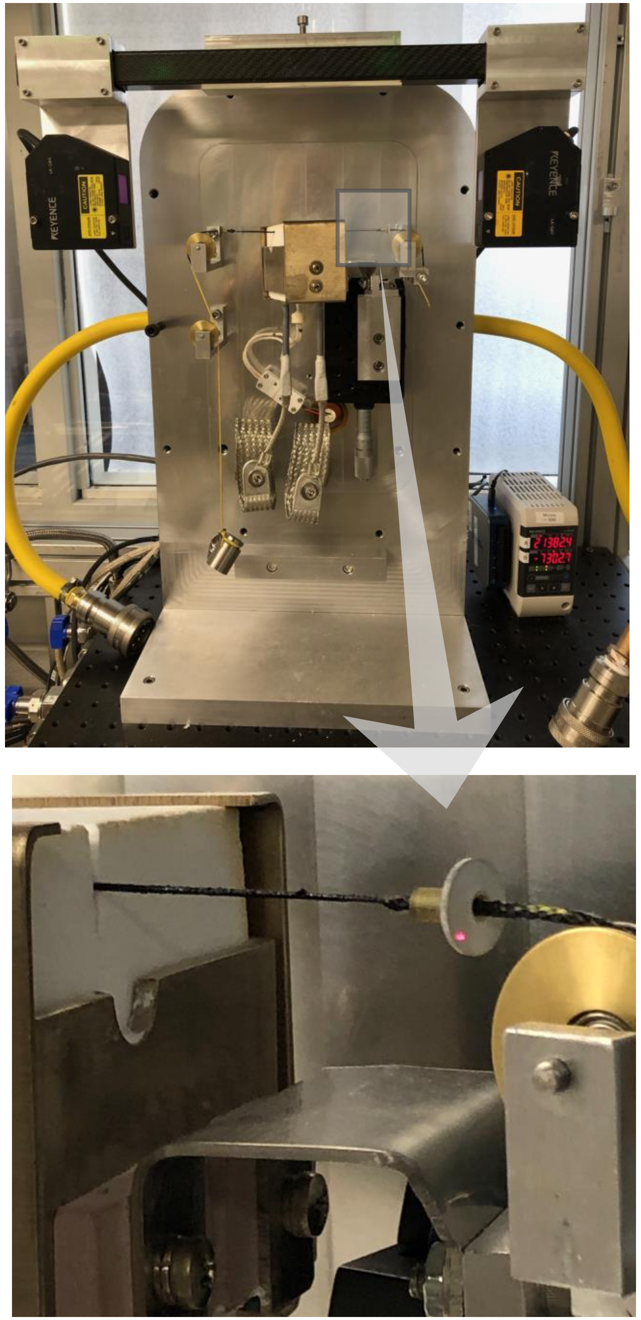
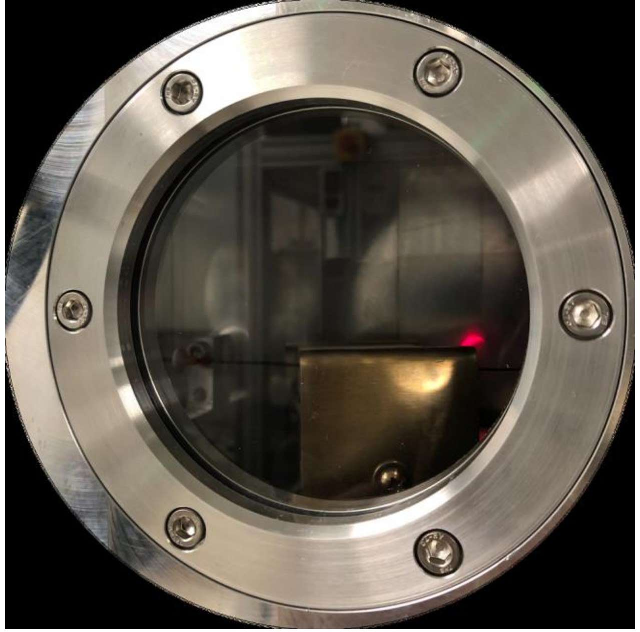
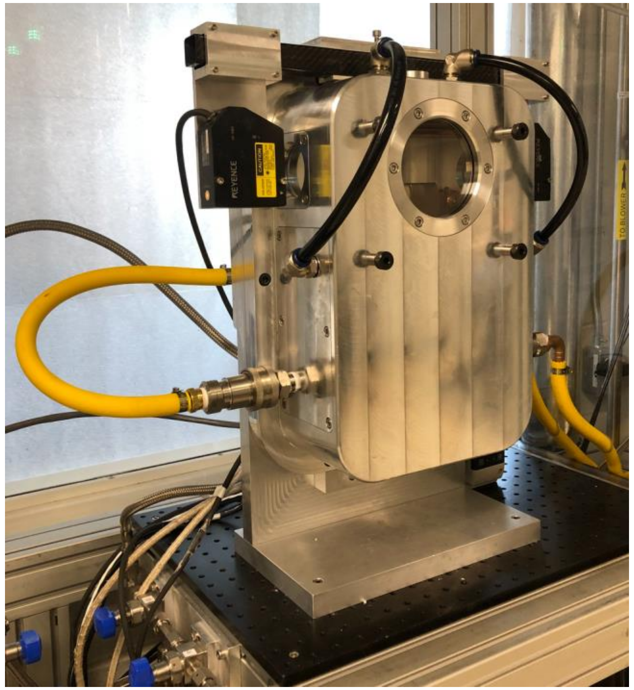
SC0022704 -- Technical

- Standardize coupon & testing procedures
  - Built and fired environmental microcomposite testing apparatus.
  - Temp. Max 1750ºC
  - No combustion
  - Gases: H_2, CH_4, H_2O, CO_2, and other neutrals



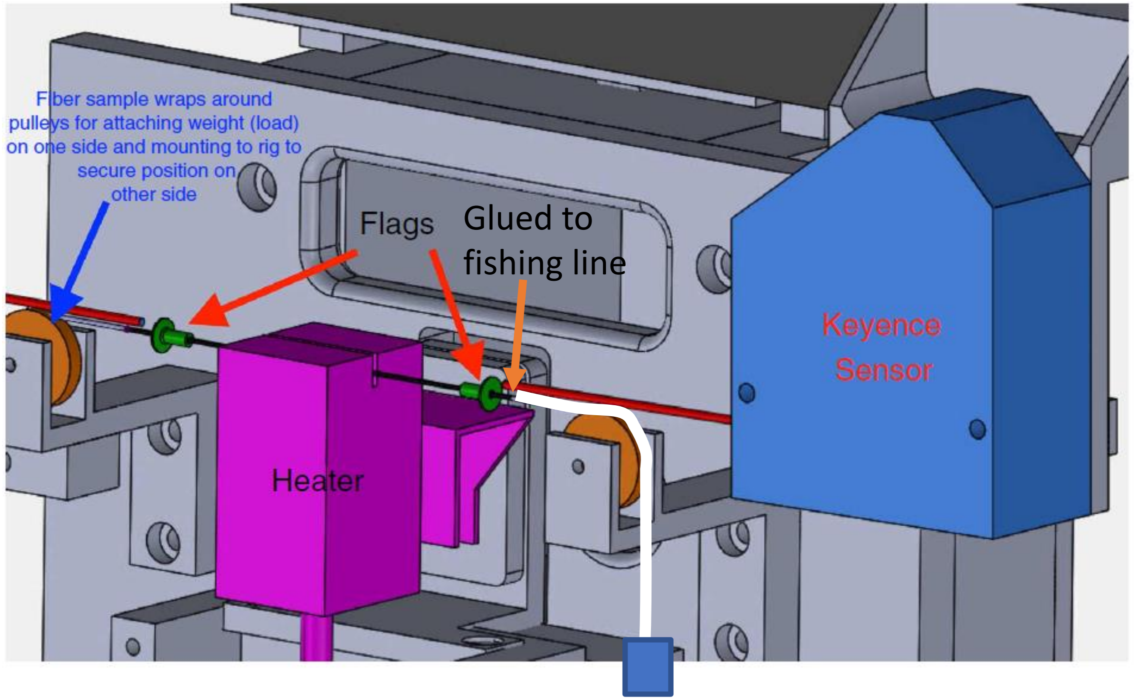


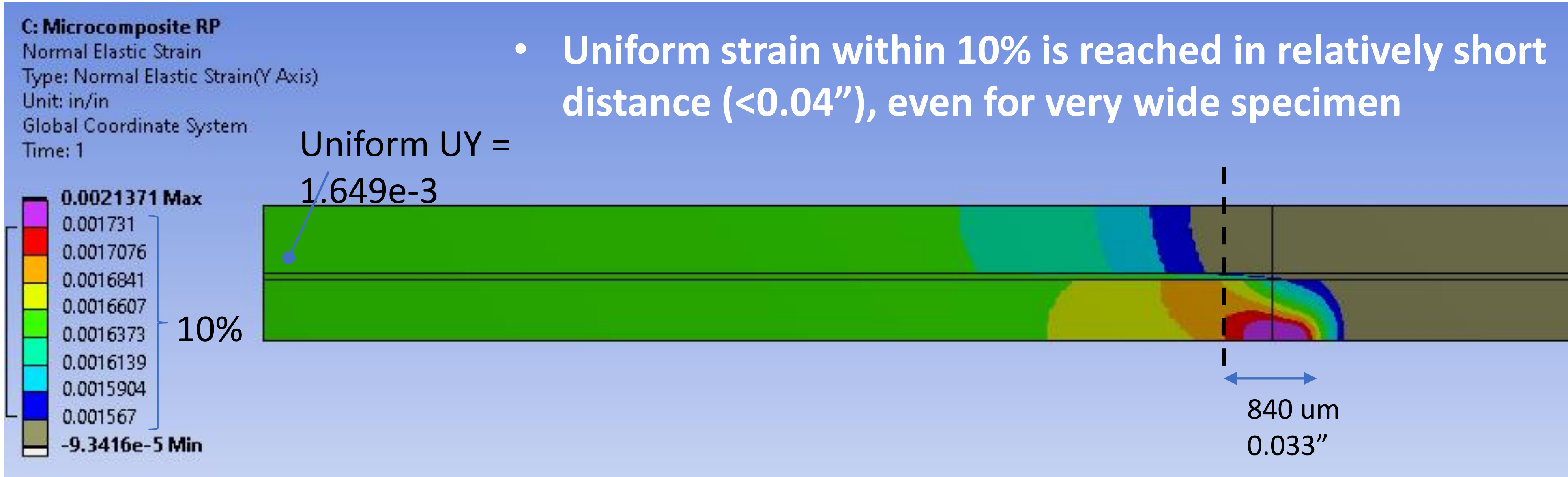


SC0022704 -- Technical

Calibrate CIME models – Worked with MR&D to

- Validate testing methodology
- Thermal design
- Corrosion model (in process).





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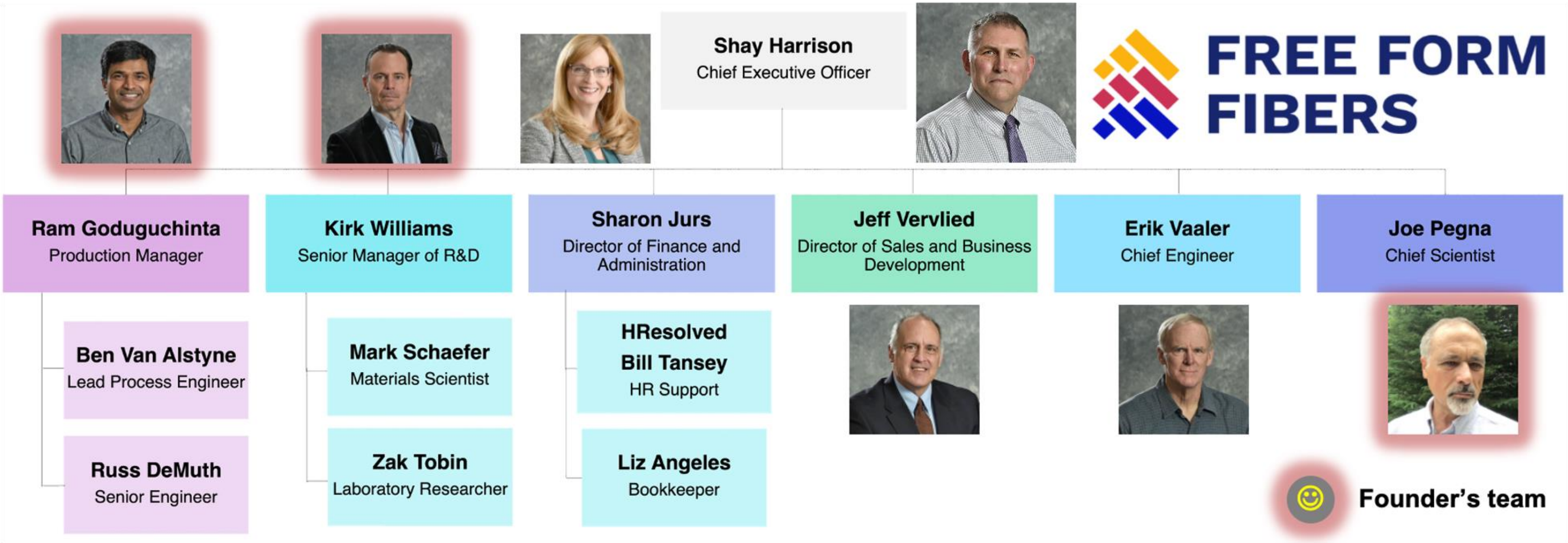
SC0022704 -- Commercial

- Free Form Fibers’ TEAM



BOARD:

- John Schneiter, Chairman & CEO Emeritus, Serial Entrepreneur
- Lt. Gen. John Novalis (ret. Army)
- F. Michael Zovistoski, CPA, CFA, Institutional Investor rep.
- Shay Harrison, Ph.D., CEO.
- Founders.

SC0022704 -- Commercial

- Commercialize a Model ???
  - Resources:
    - Ceramics Expo panel
    - USACA
    - JFCA, ECeRS, ISO
    - ASTM C28, ISO TC206
  - Case studies:
    - Kodak
    - X-Ray Optical Systems

Answer:

- Work through Standards (ASTM & ISO)
- Standardize test coupons & methodology

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Phase II Goals

Technical:

- Implement combustion & probing techniques that could not be done in Phase I:
    - Temperature
    - LIBS
- Continue implementing dual track experiment / modeling to build CIME database.

Commercial:

- Work with International Standard organizations to gain standard approval.
- Establish “Gateway technology”.

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* Flame temperature measured with a Fluke 53 thermometer using a Type B thermocouple: 1550 - 1650ºC

Comparative Burn Tests between Sylramic, HNS, and FFF Type B. Top number in **Minutes***

Comparative Burn Tests between FFF Types A & B. Top number in **Hours***

Conclusion

- **Micro-Trellis:** 3-D non-woven fiber architecture
- **Embedded Wire Chemical Vapor Deposition:** Fast, low-cost, CVI/CVD for SiC-SiC CMC shells
- **Joining:**
  - SiC-SiC / Metal Joining
  - SiC-SiC homogeneous joining
- **SC0022704 (Topic 21.e):**
  - Low-cost standard testing coupons / method
  - Need to become approved standard for viable commercialization.

Acknowledgments:

- Microcomposite testing: Department of Energy SBIR Award DE-SC0022704 (PM: Dr. Adam Payne)
- Micro-trellis: Department of Energy SBIR Award No. DE-SC0019912 (PM: Dr. Frank Goldner)
- Homogeneous joining: Department of Energy SBIR Award No. DE-SC0021665 (PM: Dr. Kenneth Wade)

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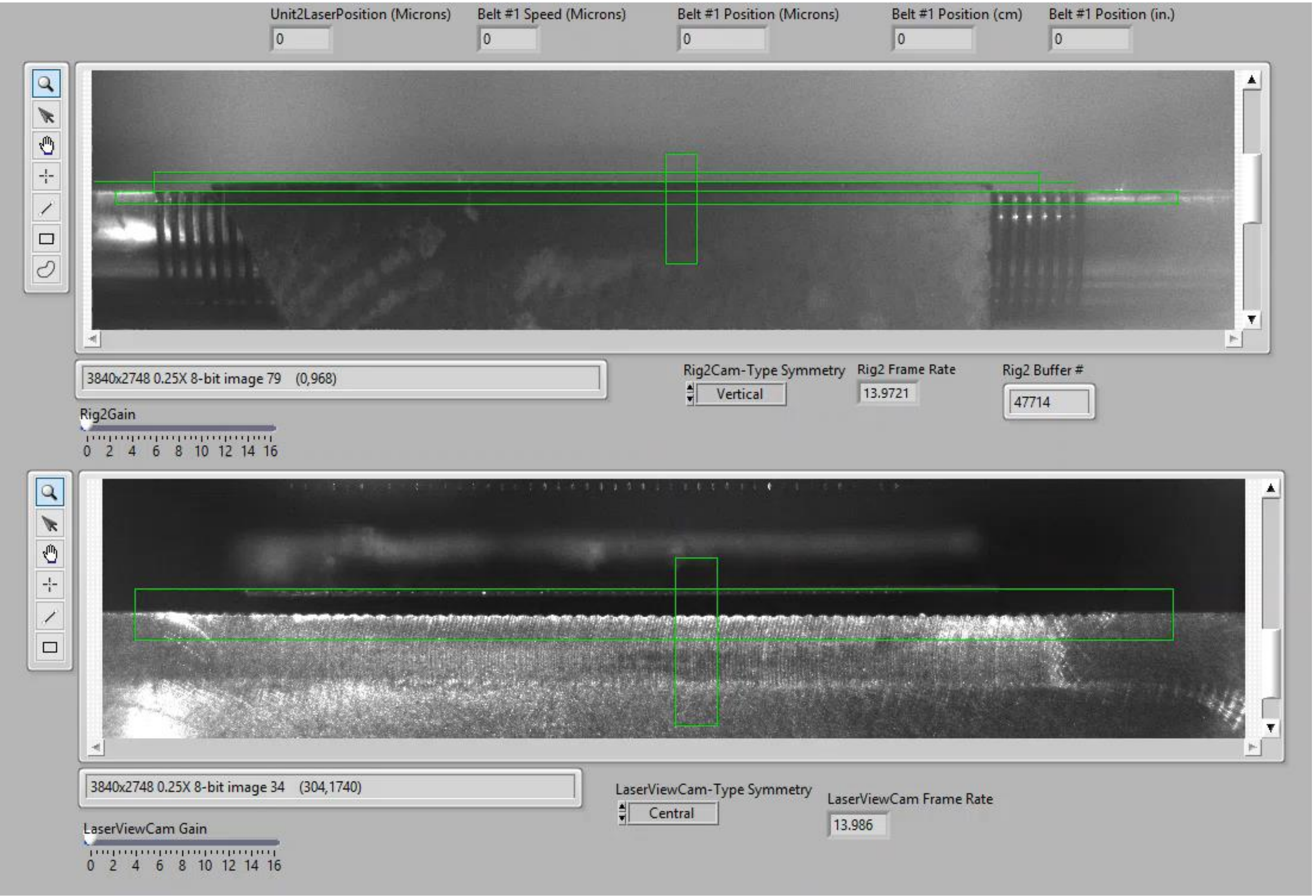
QUESTIONS?



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Low-Cost/High Performance Silicon Carbide Composite Shells for Gas Turbines

2023 FECM/NETL SPRING R&D PROJECT REVIEW MEETING

Track 2: Advanced Energy Materials for Hydrogen Turbines (Advanced Energy Materials)

Moderator:
Heather Hunter
April 18, 2023

Low-Cost/High Performance Silicon Carbide Composite Shells for Gas Turbines

Backup Slides

Laser-Printed SiC Fiber Microstructure Gradient

Journal of Nuclear Materials 548 (2021) 152805

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Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Hydrothermal corrosion of laser printed SiC fibers under extreme environment

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SiC
R-LCVD
gamma irradiation
Nuclear Fuel

ABSTRACT

SiC/SiC fiber composites are proposed cladding materials to improve the accident tolerance of commercial light water nuclear reactor fuel. To evaluate their viability, understanding the kinetics of corrosion under irradiation and high-temperature, high-pressure aqueous environment is critical. In the present work, the microstructure and mechanical properties of novel SiC fibers manufactured by Laser Chemical Vapor Deposition (LCVD) are introduced. The LCVD technology has control over microstructure and stoichiometry while allowing for high purity manufacturing, all key qualities needed in search of an optimum SiC fiber for nuclear energy applications. The isolated hydrothermal corrosion of commercially available carbon-rich Hi-Nicalon Type-S fibers is compared to the LCVD stoichiometric and LCVD silicon-rich fibers. Autoclave testing was carried out at 310°C and 14 MPa in the absence of irradiation. In a separate study, the effect of gamma irradiation on the fibers in the room-temperature environment was analyzed. It was observed from the experiments that the dissolution of the fibers was highly dependent on the stoichiometric ratio, fiber surface morphology and thermal pre-treatment of fibers. Contrary to previous findings, fiber dissolution was also visible under low-temperature gamma irradiation in aqueous environment with LCVD fibers showing lower dissolution.

1. Introduction

Silicon Carbide (SiC) has a rich history of applications in nuclear energy. Over the past decades, silicon carbide has been demonstrated to serve as an effective coating layer in Tristructural-isotropic (TRISO) fuel because of its high-temperature strength retention and irradiation performance [1-4]. These properties, in addition to being a low-Z material, are also the reason SiC has been considered for structural materials and plasma-facing components in fusion reactors [5-8]. Though several investigations were conducted in the past on SiC for non-Light Water Reactor (LWR) reactors, after the Fukushima Daiichi accident, the focus has been on SiC as a potential accident tolerant fuel (ATF) cladding [9,10] for LWR.

Several concepts incorporating SiC are proposed as ATF candidates. The prominent one being the SiC/SiC composite cladding [11,12]. Fully Ceramic Microencapsulated Fuel (FCM) [13,14] is another concept considered for a later deployment, which is comprised of TRISO particles in a SiC matrix with a SiC/SiC composite cladding. Fuel-in-Fiber [15-17] is a similar concept developed by *Free Form Fibers*, which leverages Laser Chemical Vapor Deposition (LCVD) additive manufacturing to allow higher fuel packing fractions and lower enrichment compared to FCM. This is achieved by laser deposition from the gas phase of layers analog to TRISO. The resulting Fuel-in-Fiber thus presents a multifunctional architecture. When embedded in a SiC matrix, fuel fibers serve both as structural reinforcements, and a vessel containing TRISO-like fue. Whether it is for SiC/SiC cladding or Fuel-in-Fibers, SiC plays a central role as constitutive material. When in fiber form, SiC plays a vital structural role, which warrants the focus in this paper on nuclear-grade SiC fibers.

Recently, several works were carried out to understand the mechanism of corrosion in CVD SiC and SiC/SiC composite relevant to the operating conditions of a pressurized water reactor (PWR) and boiling water reactor (BWR). It is clear from the experiments conducted in the past decade [18,19], including the latest results by Terrani et al. [20], that in the absence of irradiation and under PWR pressure and temperature, silica forms and dissolves as a result of its reaction with the high-temperature coolant. All things being equal, the formation and subsequent dissolution

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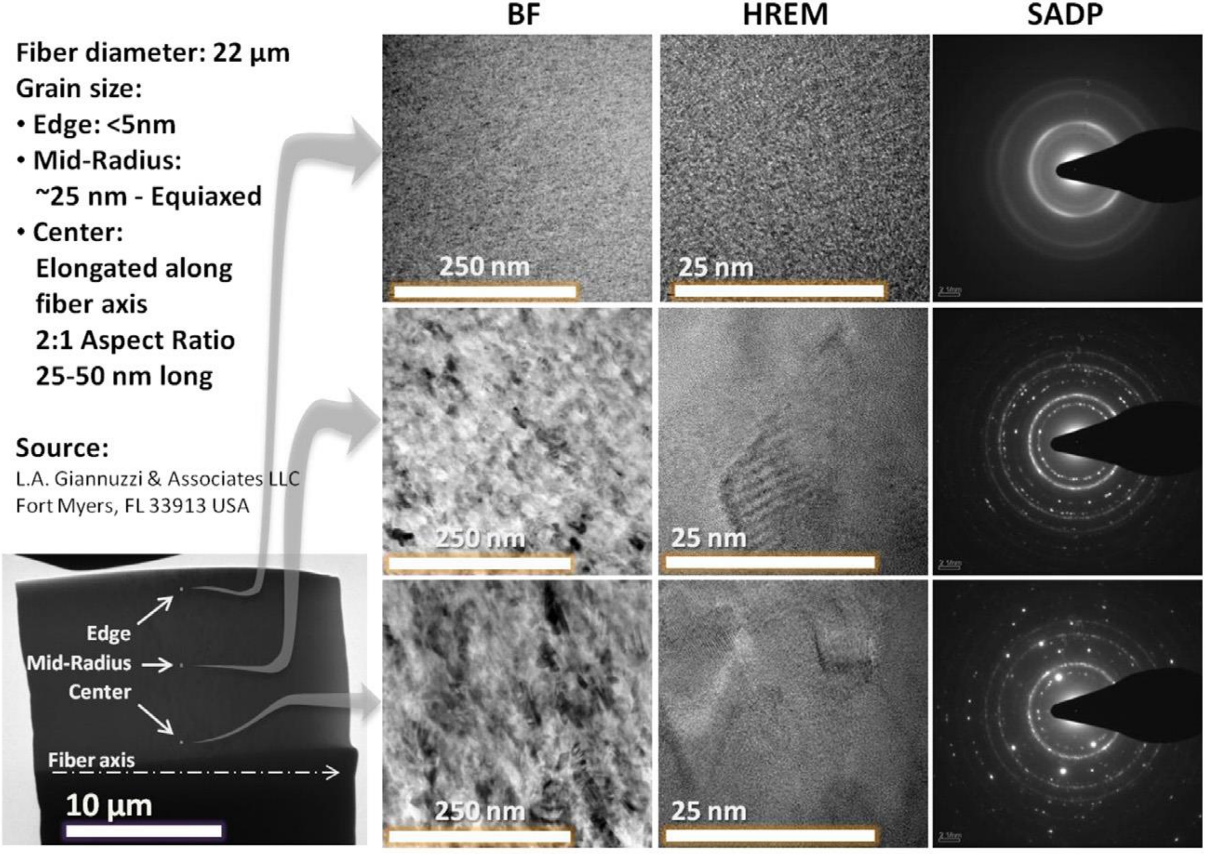
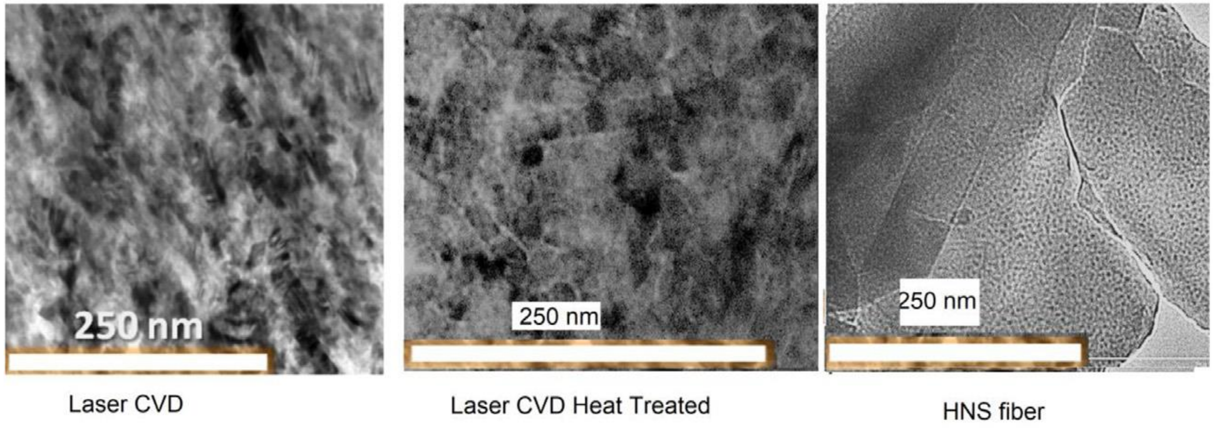


(a)



(b)

Laser-Printed SiC Fiber Microstructure Gradient


Journal of Nuclear Materials 548 (2021) 152805

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Hydrothermal corrosion of laser printed SiC fibers under extreme environment

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ABSTRACT

SiC/SiC fiber composites are proposed cladding materials to improve the accident tolerance of commercial light water nuclear reactor fuel. To evaluate their viability, understanding the kinetics of corrosion under irradiation and high-temperature, high-pressure aqueous environment is critical. In the present work, the microstructure and mechanical properties of novel SiC fibers manufactured by Laser Chemical Vapor Deposition (LCVD) are introduced. The LCVD technology has control over microstructure and stoichiometry while allowing for high purity manufacturing, all key qualities needed in search of an optimum SiC fiber for nuclear energy applications. The isolated hydrothermal corrosion of commercially available carbon-rich Hi-Nicalon Type-S fibers is compared to the LCVD stoichiometric and LCVD silicon-rich fibers. Autoclave testing was carried out at 310°C and 14 MPa in the absence of irradiation. In a separate study, the effect of gamma irradiation on the fibers in the room-temperature environment was analyzed. It was observed from the experiments that the dissolution of the fibers was highly dependent on the stoichiometric ratio, fiber surface morphology and thermal pre-treatment of fibers. Contrary to previous findings, fiber dissolution was also visible under low-temperature gamma irradiation in aqueous environment with LCVD fibers showing lower dissolution.

1. Introduction

Silicon Carbide (SiC) has a rich history of applications in nuclear energy. Over the past decades, silicon carbide has been demonstrated to serve as an effective coating layer in Tristructural-isotropic (TRISO) fuel because of its high-temperature strength retention and irradiation performance [1-4]. These properties, in addition to being a low-Z material, are also the reason SiC has been considered for structural materials and plasma-facing components in fusion reactors [5-8]. Though several investigations were conducted in the past on SiC for non-Light Water Reactor (LWR) reactors, after the Fukushima Daiichi accident, the focus has been on SiC as a potential accident tolerant fuel (ATF) cladding [9,10] for LWR.

Several concepts incorporating SiC are proposed as ATF candidates. The prominent one being the SiC/SiC composite cladding [11,12]. Fully Ceramic Microencapsulated Fuel (FCM) [13,14] is another concept considered for a later deployment, which is comprised of TRISO particles in a SiC matrix with a SiC/SiC composite cladding. Fuel-in-Fiber [15-17] is a similar concept developed by *Free Form Fibers*, which leverages Laser Chemical Vapor Deposition (LCVD) additive manufacturing to allow higher fuel packing fractions and lower enrichment compared to FCM. This is achieved by laser deposition from the gas phase of layers analog to TRISO. The resulting Fuel-in-Fiber thus presents a multifunctional architecture. When embedded in a SiC matrix, fuel fibers serve both as structural reinforcements, and a vessel containing TRISO-like fue. Whether it is for SiC/SiC cladding or Fuel-in-Fibers, SiC plays a central role as constitutive material. When in fiber form, SiC plays a vital structural role, which warrants the focus in this paper on nuclear-grade SiC fibers.

Recently, several works were carried out to understand the mechanism of corrosion in CVD SiC and SiC/SiC composite relevant to the operating conditions of a pressurized water reactor (PWR) and boiling water reactor (BWR). It is clear from the experiments conducted in the past decade [18,19], including the latest results by Terrani et al. [20], that in the absence of irradiation and under PWR pressure and temperature, silica forms and dissolves as a result of its reaction with the high-temperature coolant. All things being equal, the formation and subsequent dissolution

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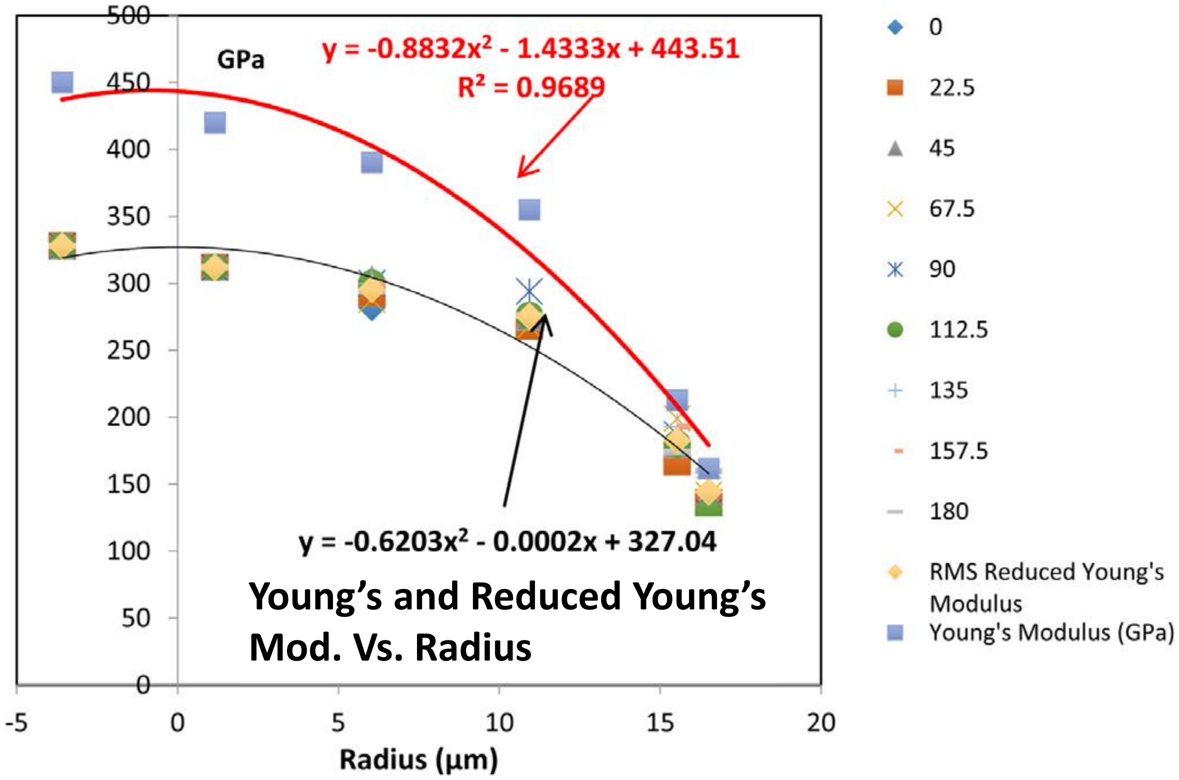
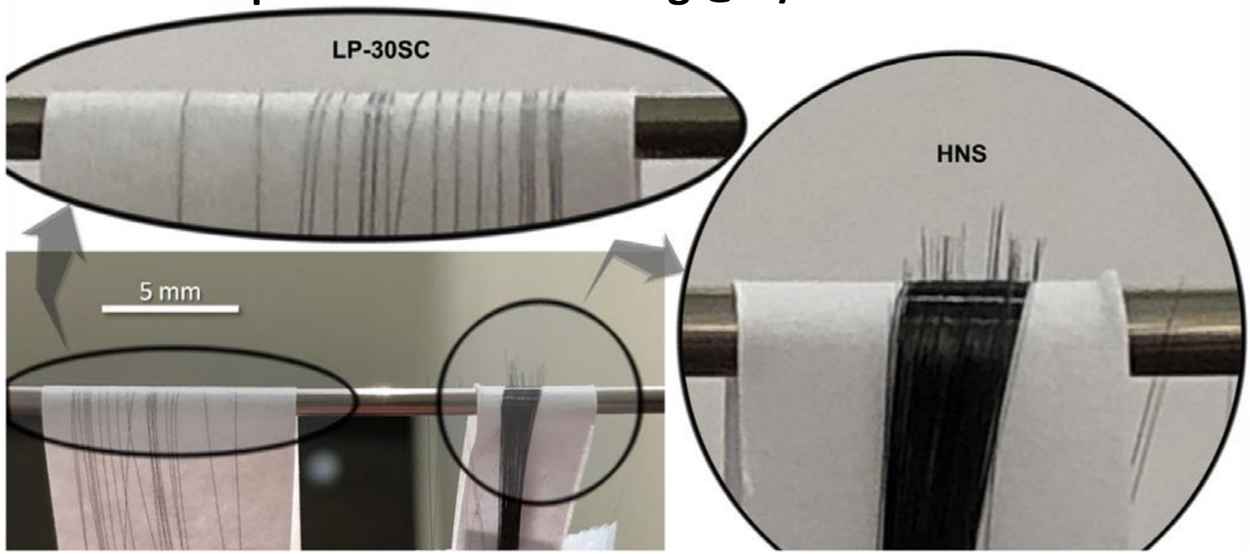


Comparative Pure Bending @ 1/16th " Radius



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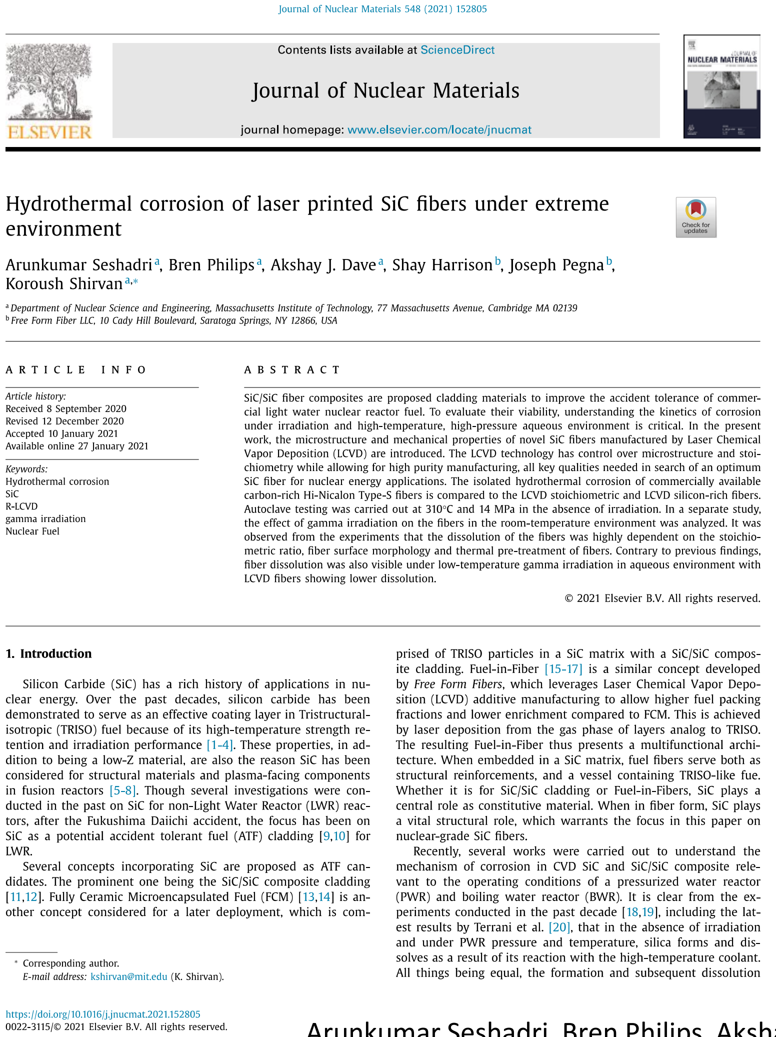

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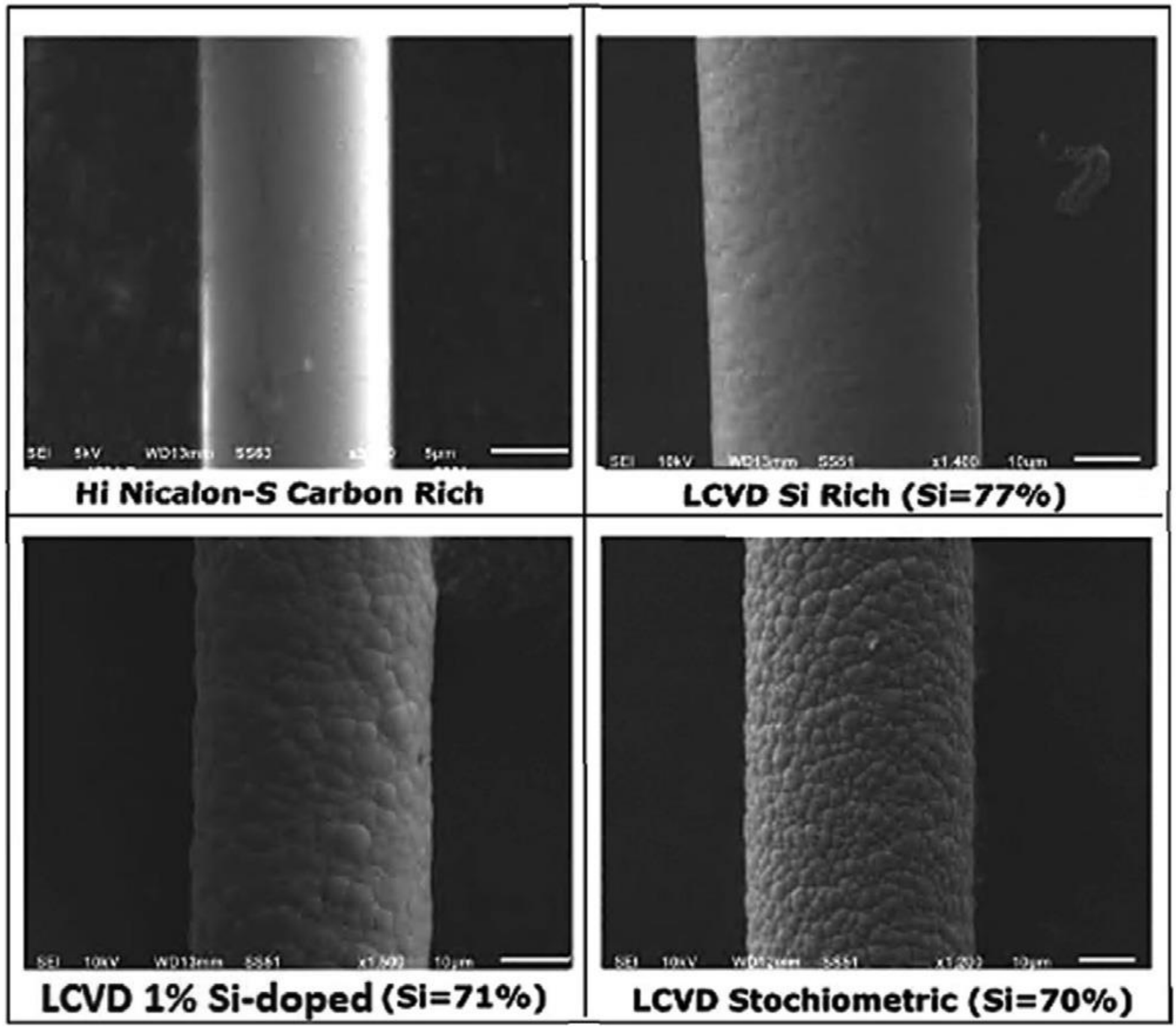
Comparative Morphologies

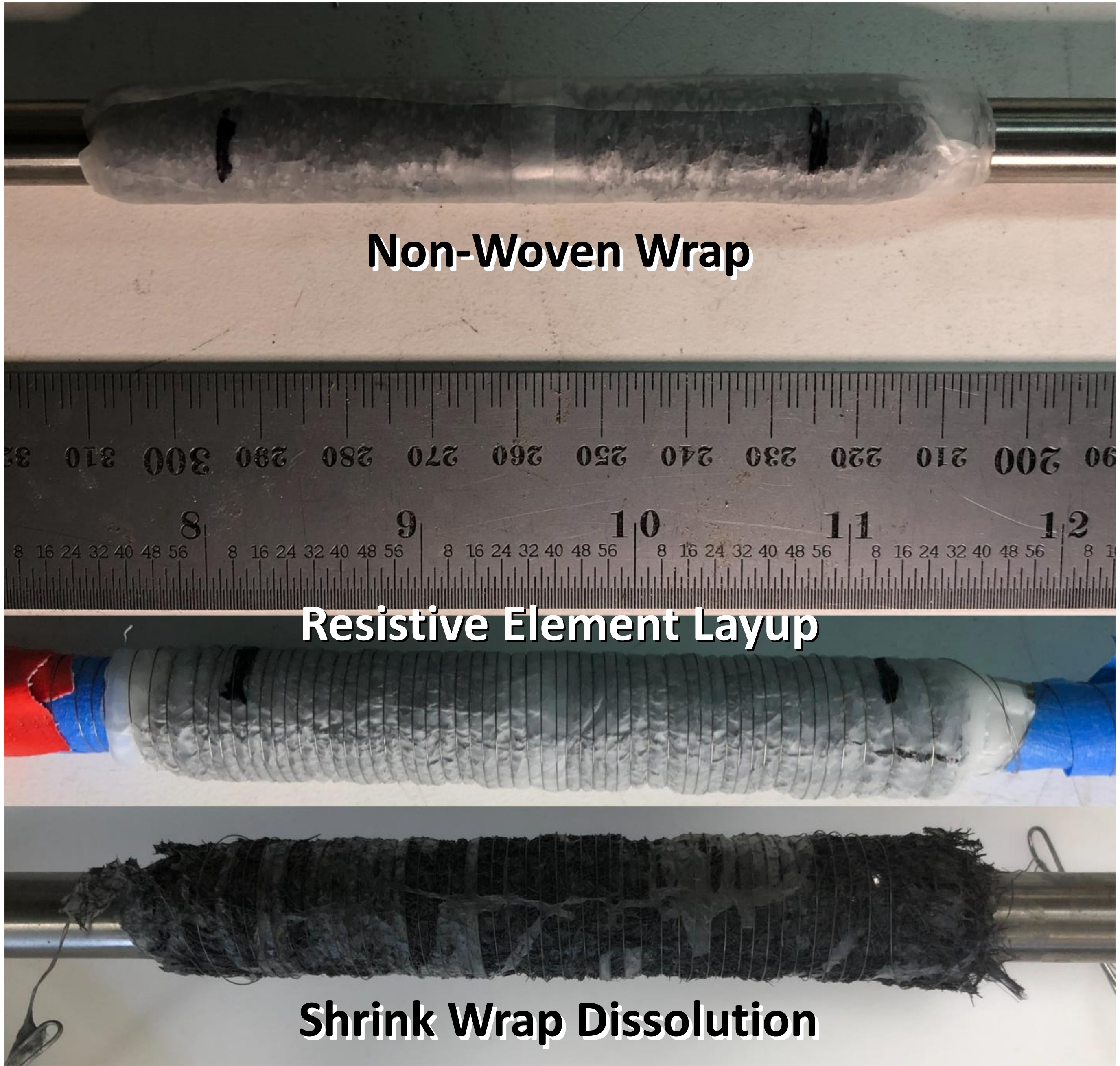


DELPOUVE, E. et al., INFLUENCE OF THE THICKNESS AND CRYSTALLINITY OF A BN INTERPHASE ON THE MECHANICAL PROPERTIES OF A MODEL CERAMIC MATRIX COMPOSITE, Proceedings of HT-CMC 10 (Bordeaux, France, Oct. 2019

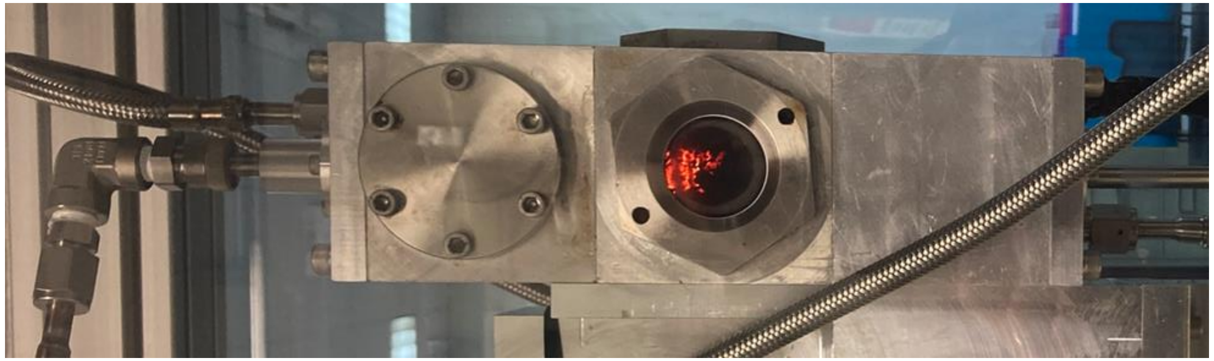
CALLAWAY, E. et al., Effects of fiber and interfacial properties on fiber fragmentation and pullout in SiC-BN-SiC minicomposites, Proceedings of HT-CMC 10 (Bordeaux, France, Oct. 2019

Embedded Wire CVD (EWCVD – Award DE-SC0019912):

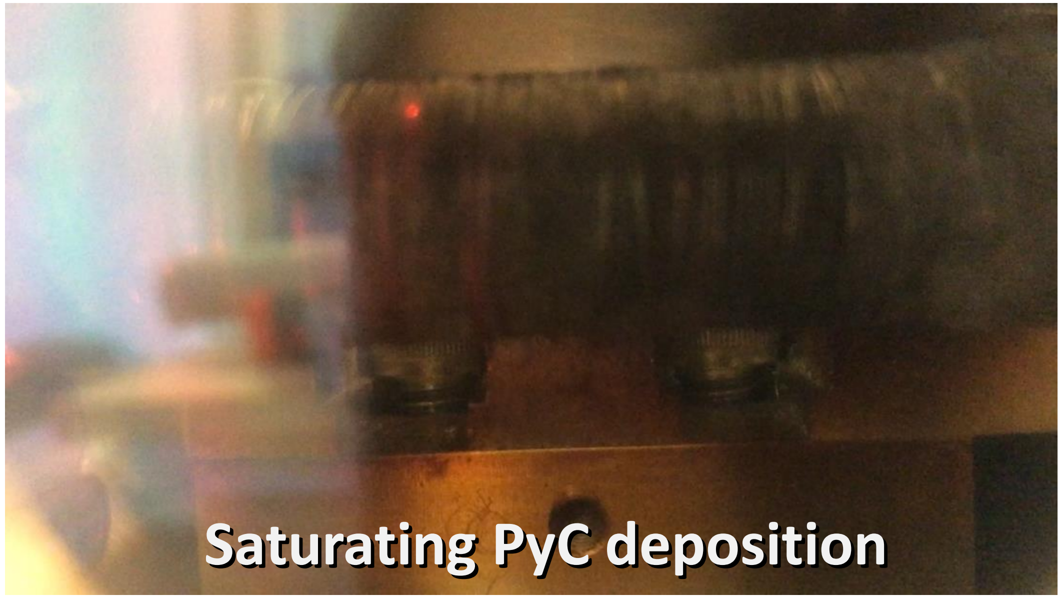
Sample preparation on Zr cladding tube



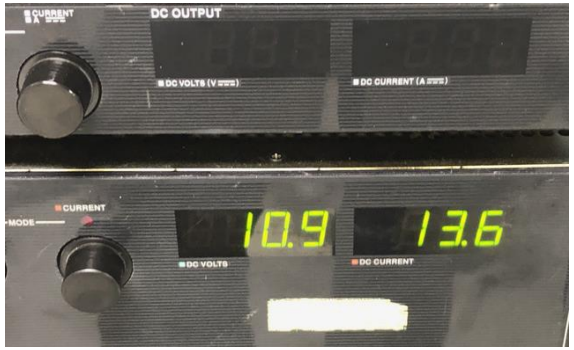
Embedded Wire CVD (EWCVD)



Sample Process Chamber

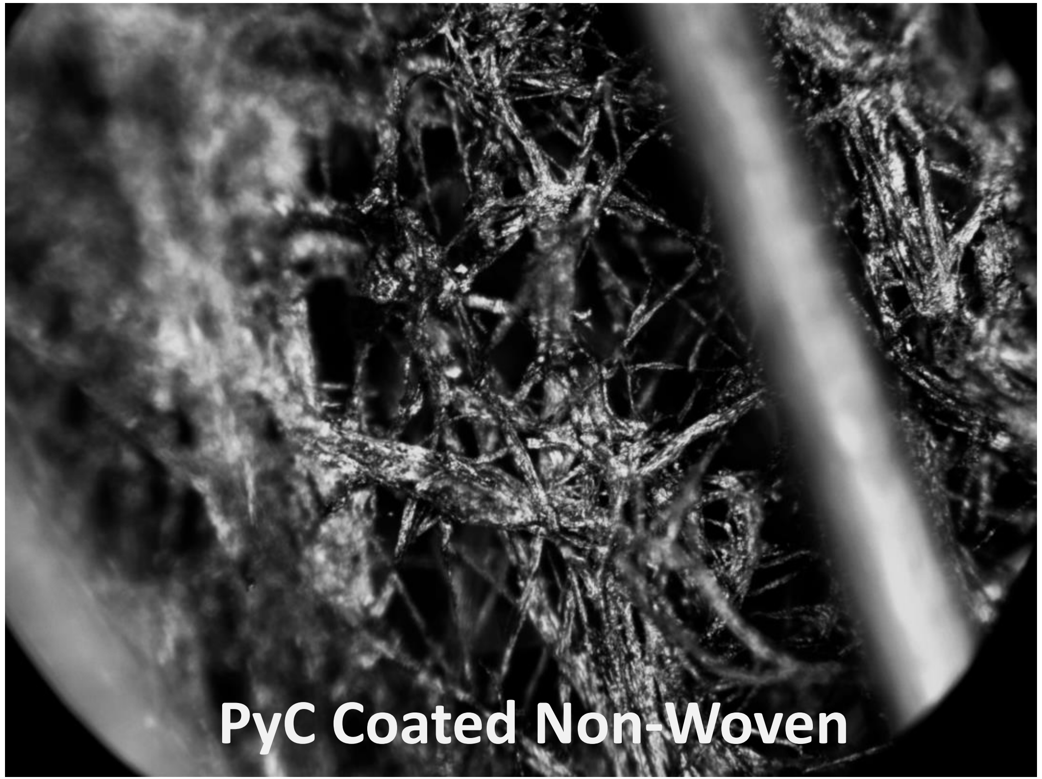


Saturating PyC deposition



Process Power: ~150W/in.

Temperature Controller



PyC Coated Non-Woven

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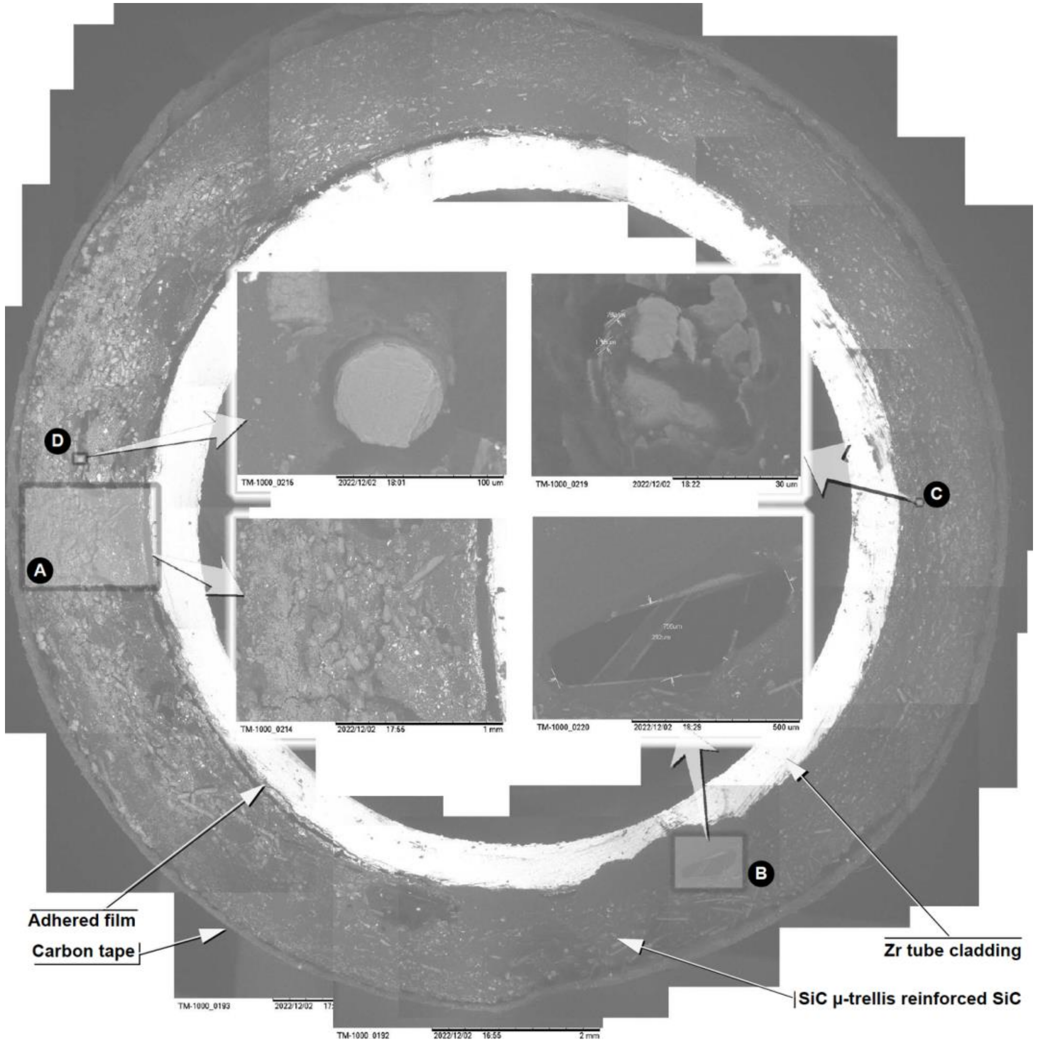
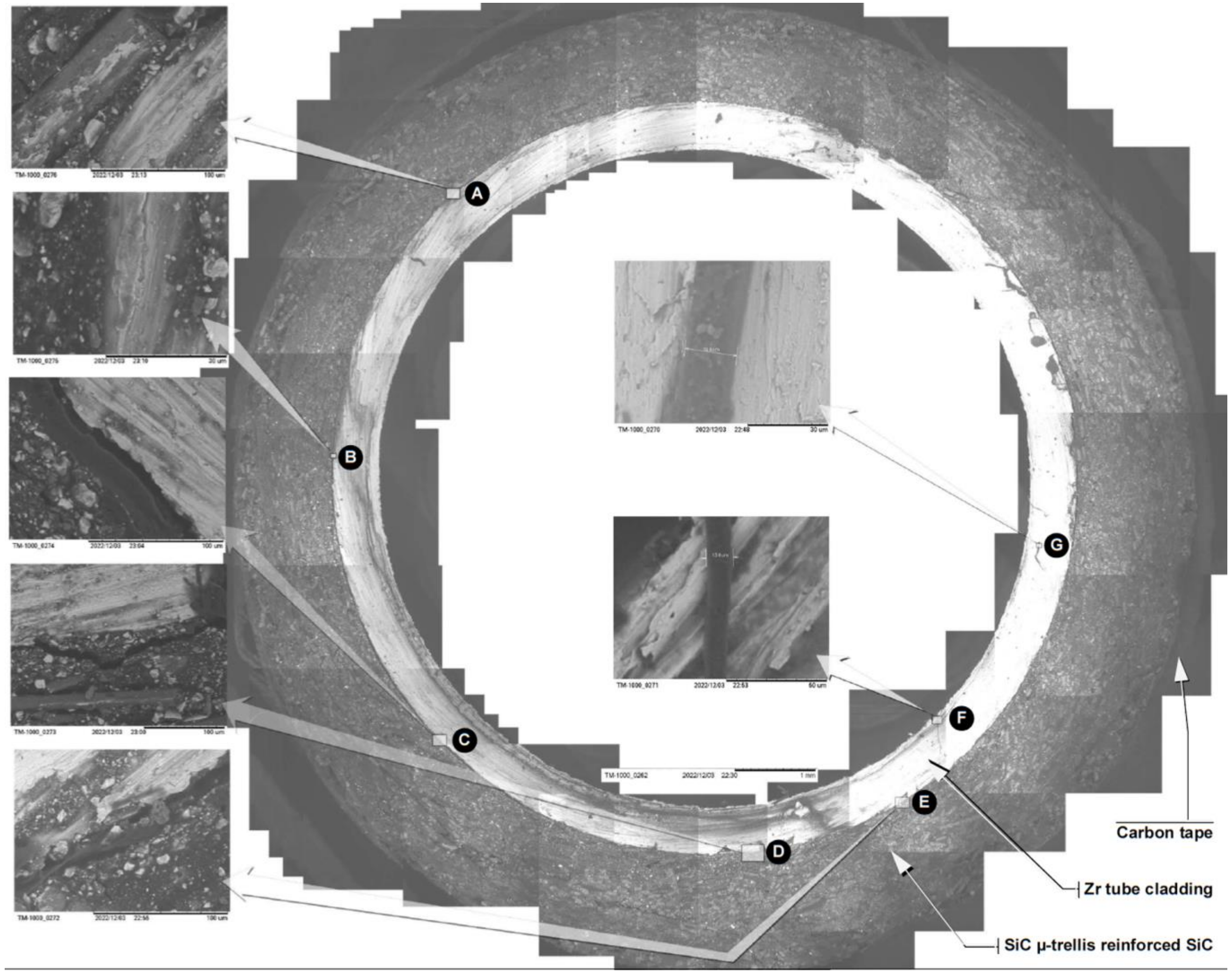
Embedded Wire CVD (EWCVD)

Non-Destructive Radial Cross-Section Composite SEMs



Zone 1



Zone 2

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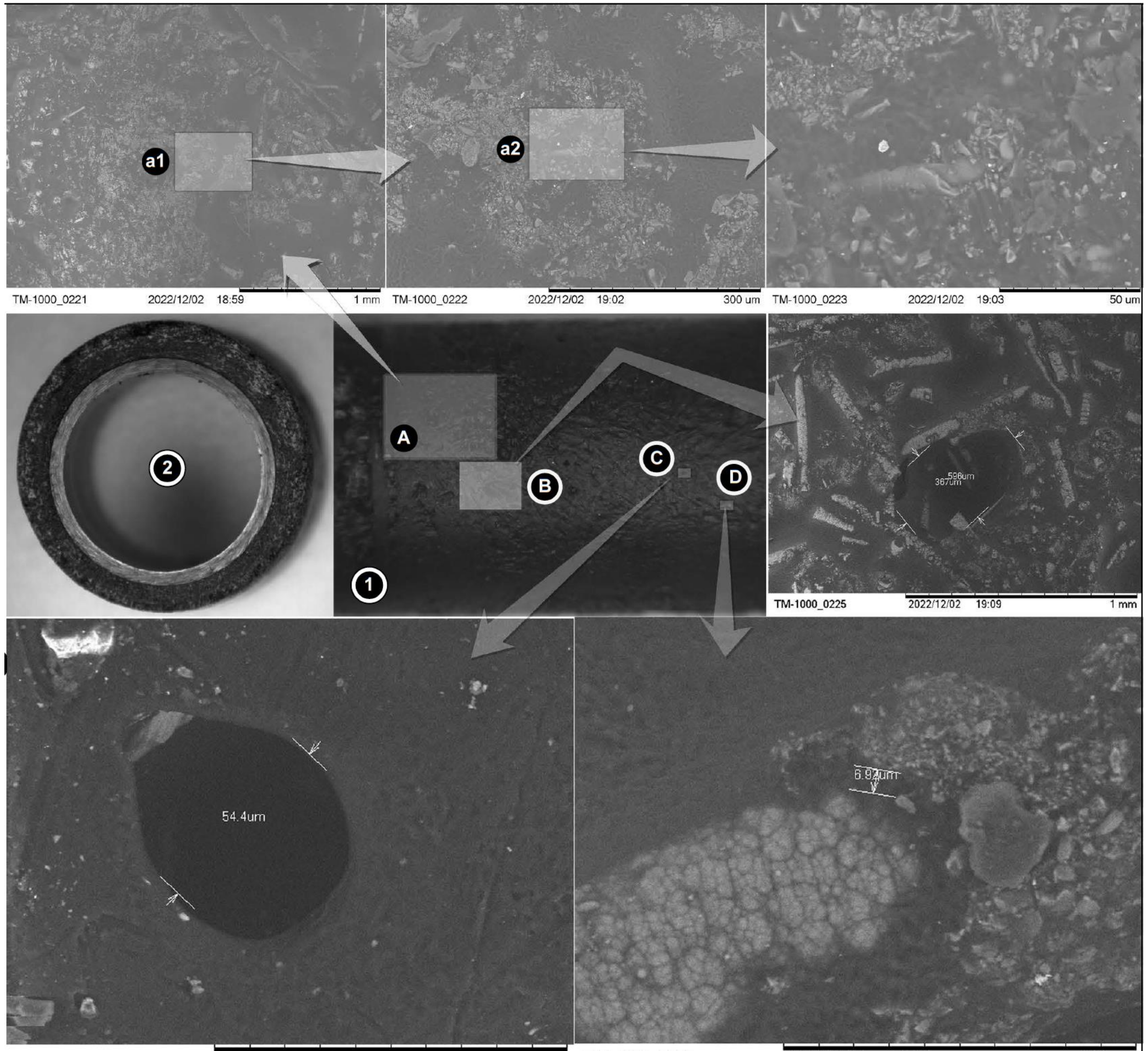
Embedded Wire CVD (EWCVD)

Non-Destructive SEM Inspection of Lateral Surface



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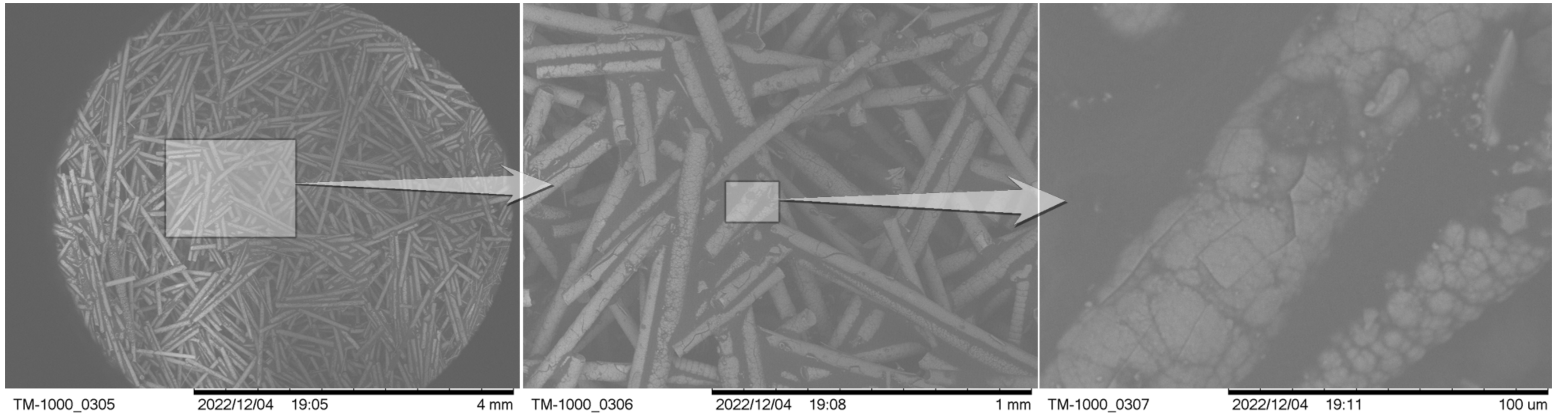
Embedded Wire CVD (EWCVD)

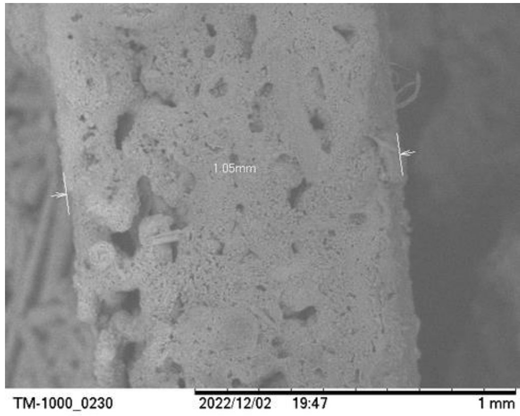
Destructive SEM Inspection of SiC-SiC

Ripped SiC-SiC shell
(in Machining Wax)

Zone 1

Sheared SiC-SiC

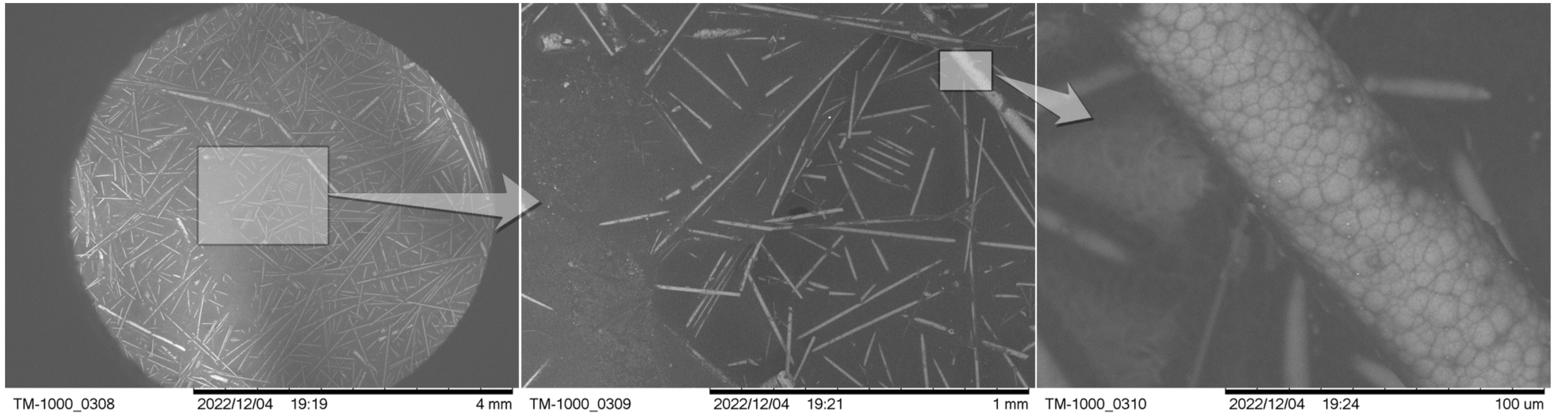


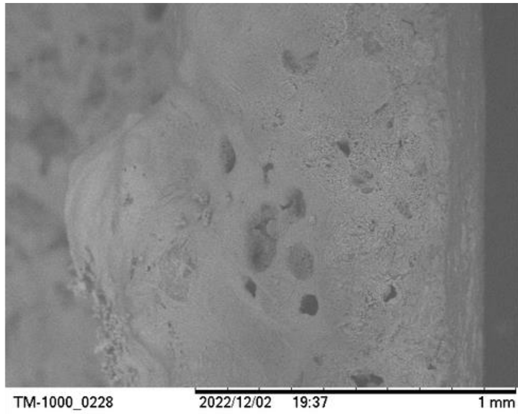


Lateral ID surface

Edge

Sheared SiC-SiC

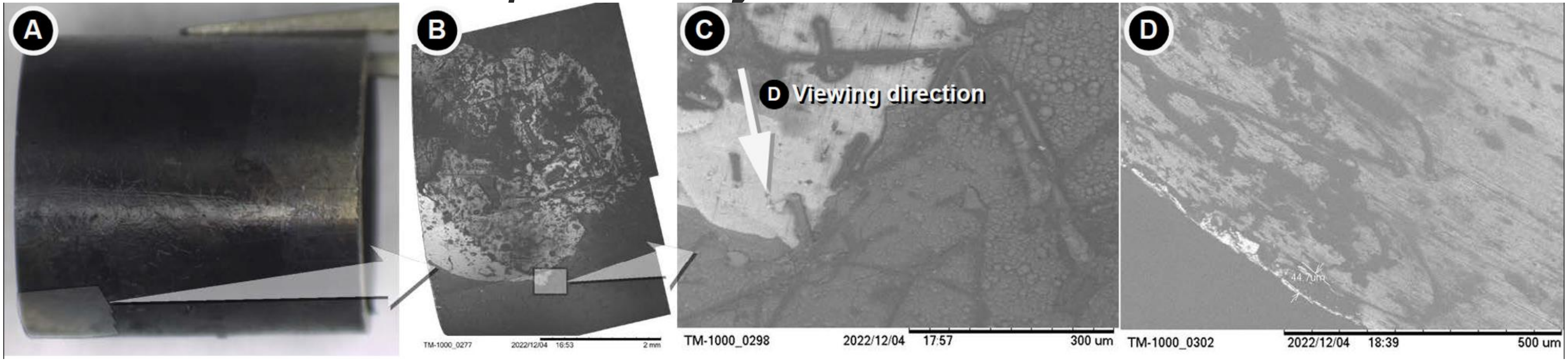


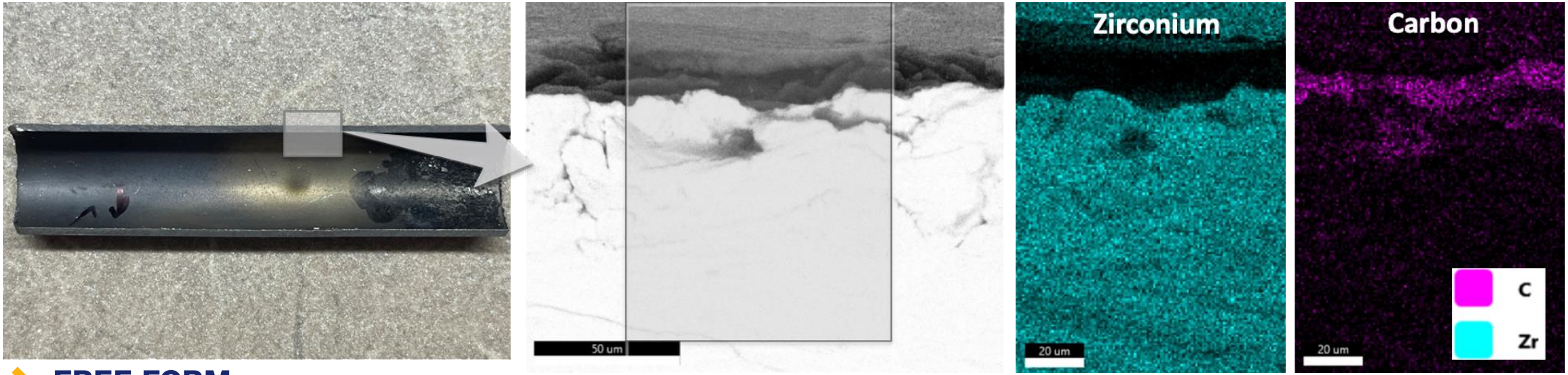


Zone 2

Embedded Wire CVD (EWCVD)

Destructive SEM Inspection of Zr tube





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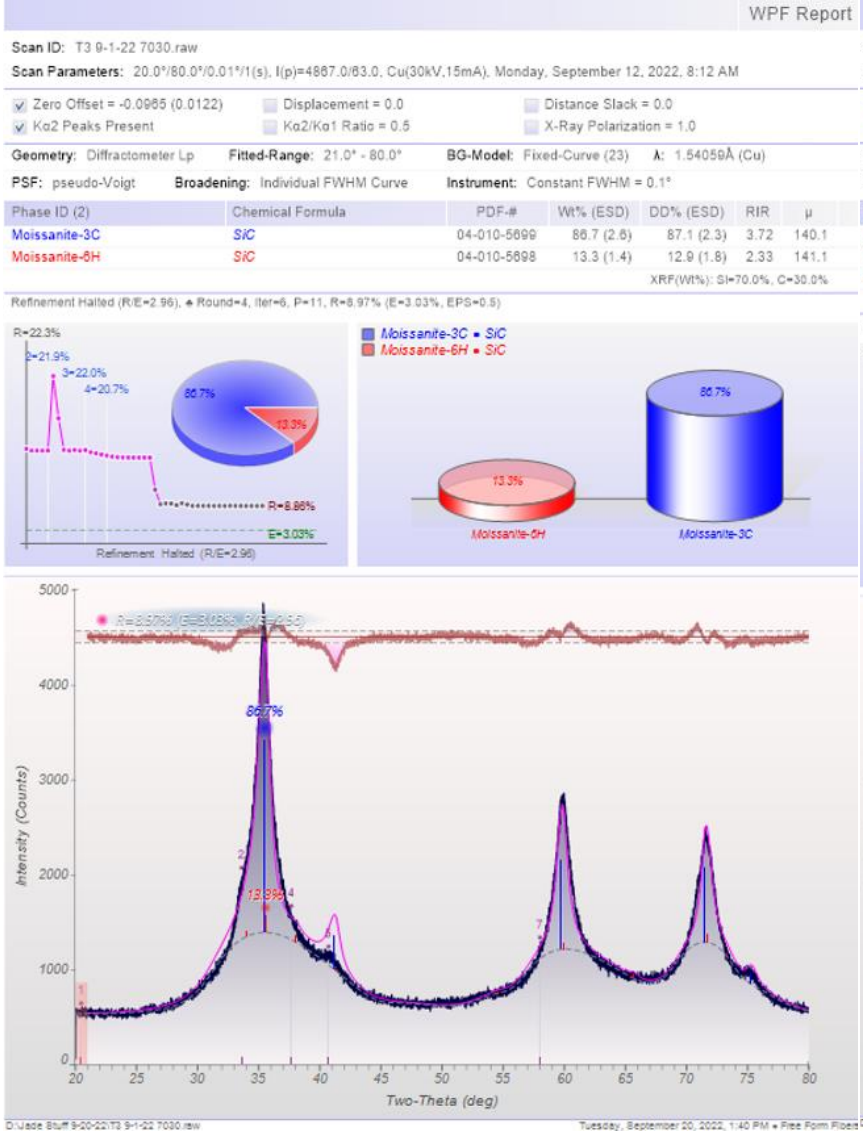
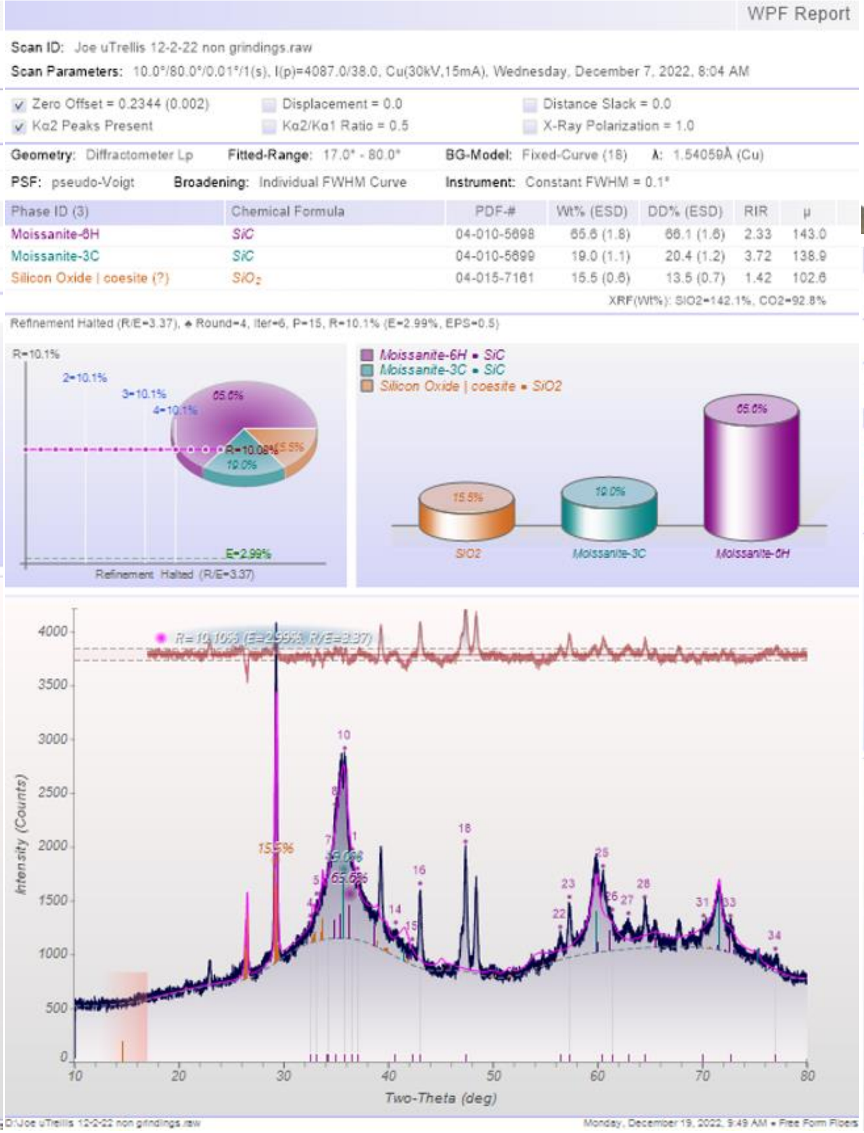
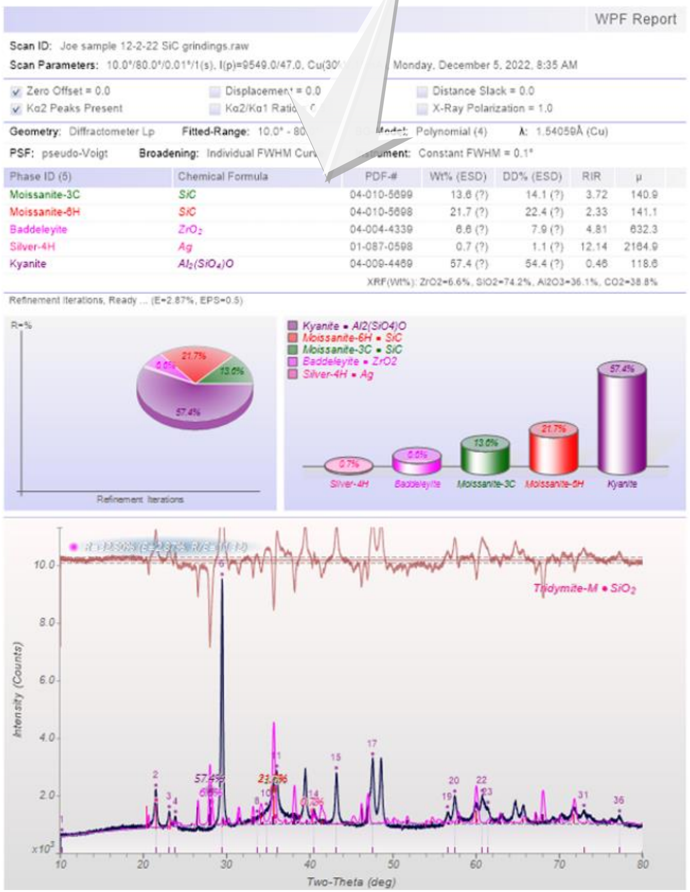
Embedded Wire CVD (EWCVD)

SiC-SiC Materials Analysis







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SiC fibers alone | **SiC-SiC CMC** | **Dust from Diamond Grinding**