Challenges of Testing EB Coated CMC under SPLCF Conditions in Steam Environment at High Temperatures

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Objectives and Approach

Objectives:

Short term:

- **Determine factors controlling premature failure of environmental barrier (EB) coated SiC/SiC composites under sustained peak low cycle fatigue (SPLCF) conditions in steam environment at high temperatures.**
- **Device methods of reducing premature failure and improving durability of EB coated CMCs under above conditions.**

Long term:

- **Determine cyclic durability and failure behavior of EB coated 3-D woven SiC/SiC CMC from 1200 to 1482^{o}C in steam environment. Measure thermally grown oxide (TGO) on CMC.**

Approach:

- **Deposit EB coating on CMC substrates.**
- **Conduct SPLCF tests on EB coated substrates.**
- **Determine microstructural stability and failure mechanism of EBC and CMC.**

Material and Characterization

Materials:
Substrates: Hexoloy and 3D woven SiC/SiC composites with Sylramic-iBN fibers and (CVI+PIP) hybrid matrix
EB coating system: Type-I (Maximum use temperature: 1482^{o}C(2700^{o}F))
Bond Coat: A mixture of (Rare Earth Disilicate (RED) + Mullite) by slurry method
Top Coat: RED by slurry method

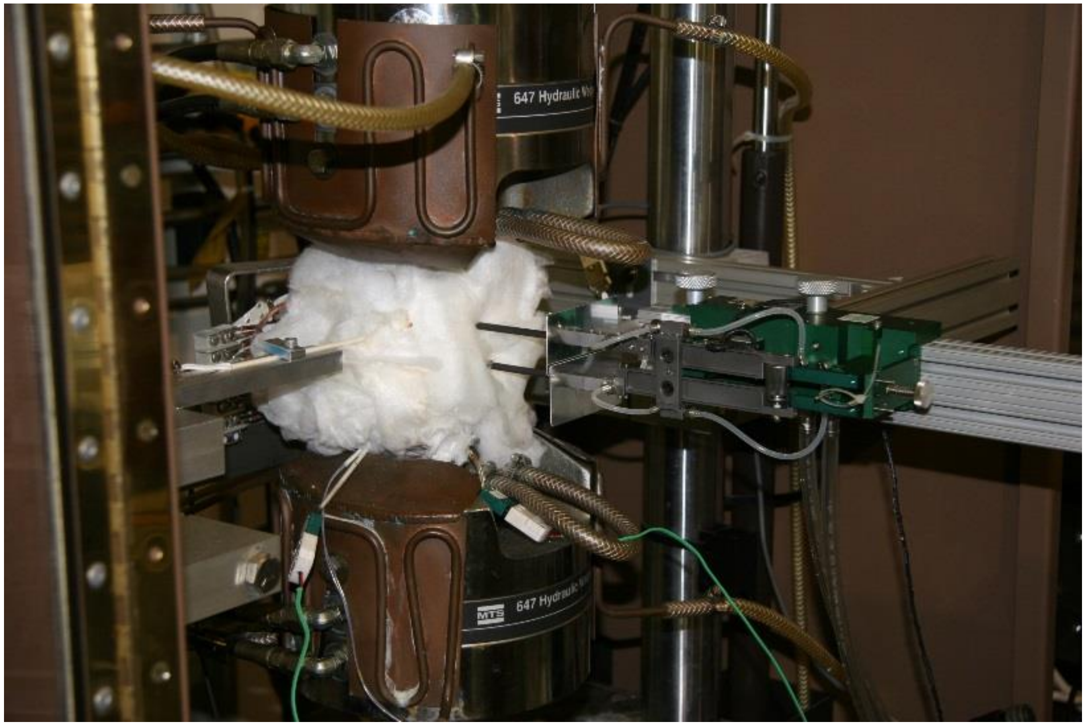
Substrate: 2D woven MI SiC/SiC composites
EB coating system: Type-II (Maximum use temperature: 1400^{o}C(2550^{o}F))
Bond Coat: Silicon by air plasma spray method
Top Coat: RED by air plasma spray method

***Specimen Geometry*:**
Dog-boned specimen EB coated on four sides

***SPLCF Test Conditions*:**
Temperature: 1204^{0}C
R ratio: 0.5
Maximum stress: 69 MPa
Environment: Flowing Steam
Exposure time: Up to 200 hours

***Characterization*:**
Computed tomography (CT), Optical photography and SEM

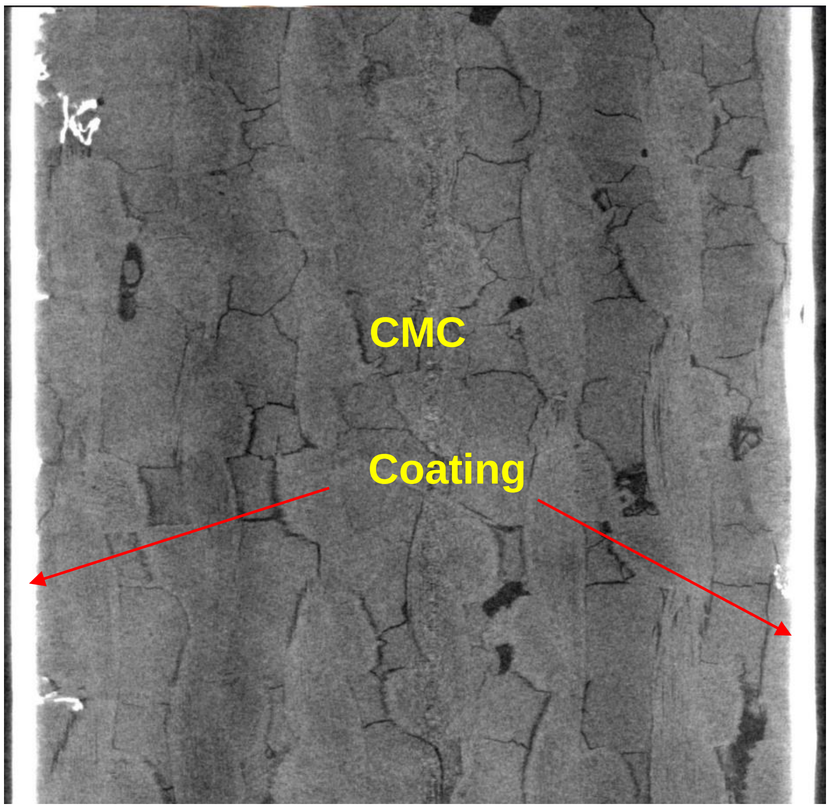
High Temperature Testing of CMCs in Creep, Fatigue and SPLCF in Steam Environment

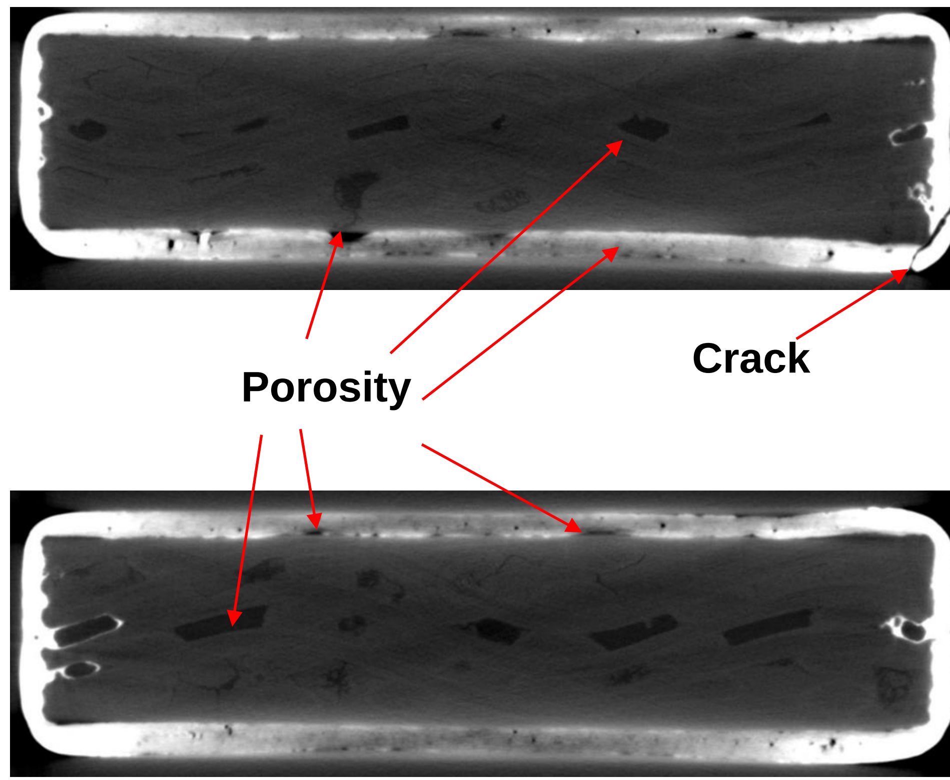


- Electromechanical, 50 kN Load Cell
- Three Zone Igniter Element Furnace
- 1.0 in. Gage Length, Water-Cooled Extensometer
- Frequencies up to 1 Hz
- Temperature capability: 1315^{o}C (Current)
  1482^{o}C (Developmental)

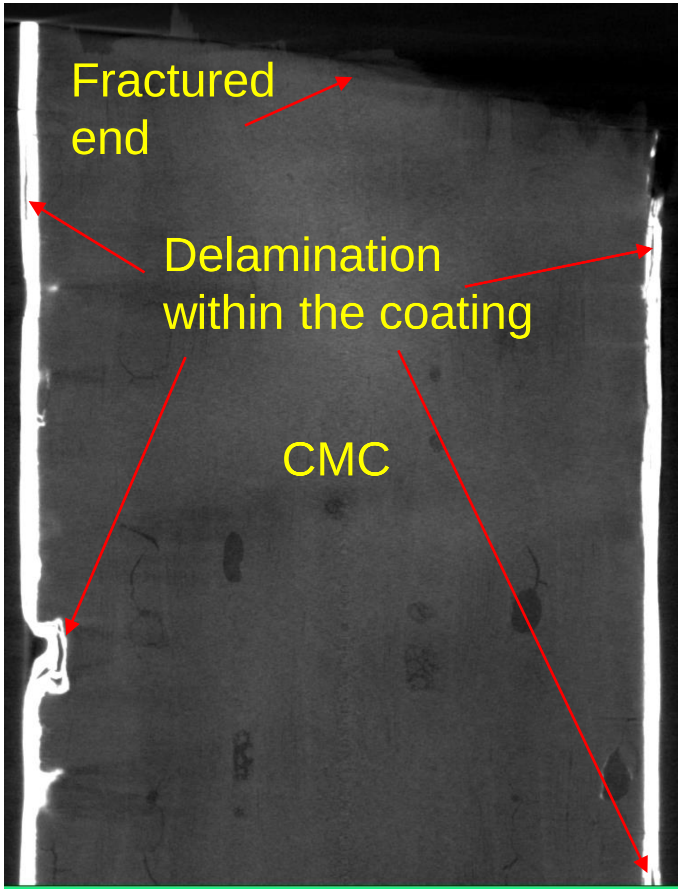
CT Images of Environmental Barrier(EB) Coated 3D Woven CMC before SPLCF testing

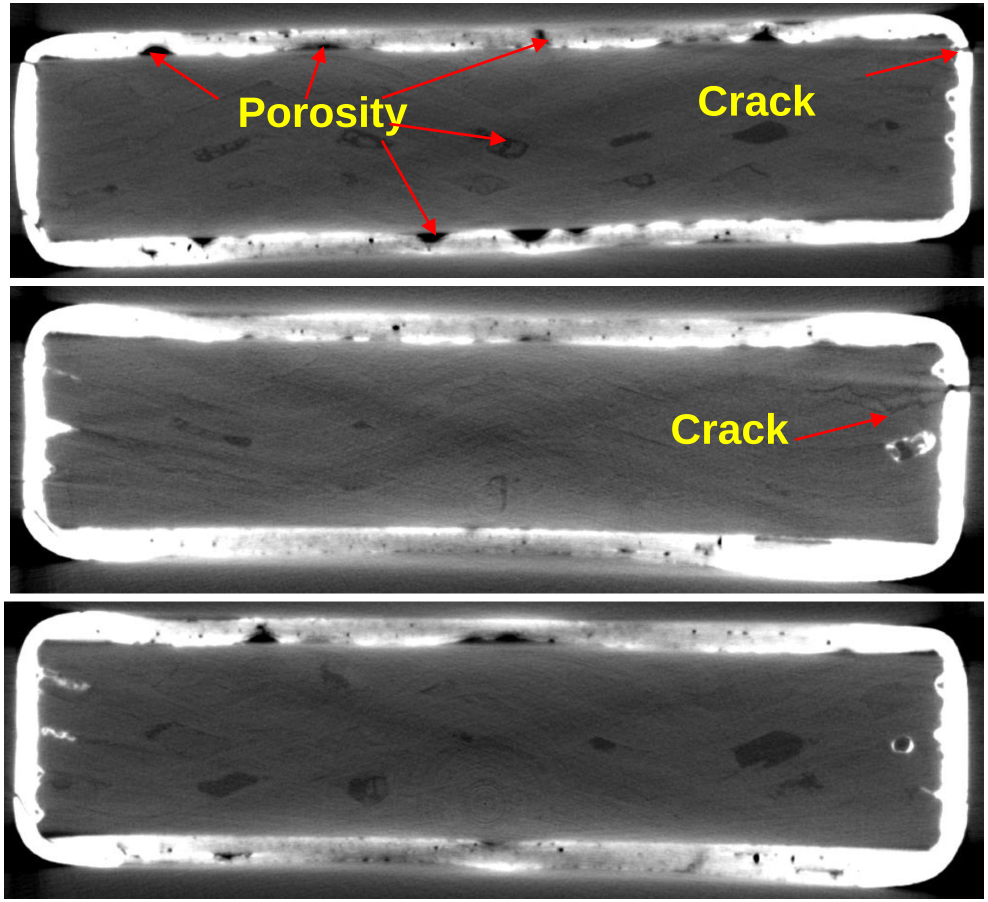
EB Coating System: Type-I
Bond Coat: Rare Earth Disilicate (RED) + Mullite
Top Coat: RED





CT Images of EB Coated 3D Woven CMC after SPLCF Testing at 1204ºC at 69 MPa for 48hrs in Steam





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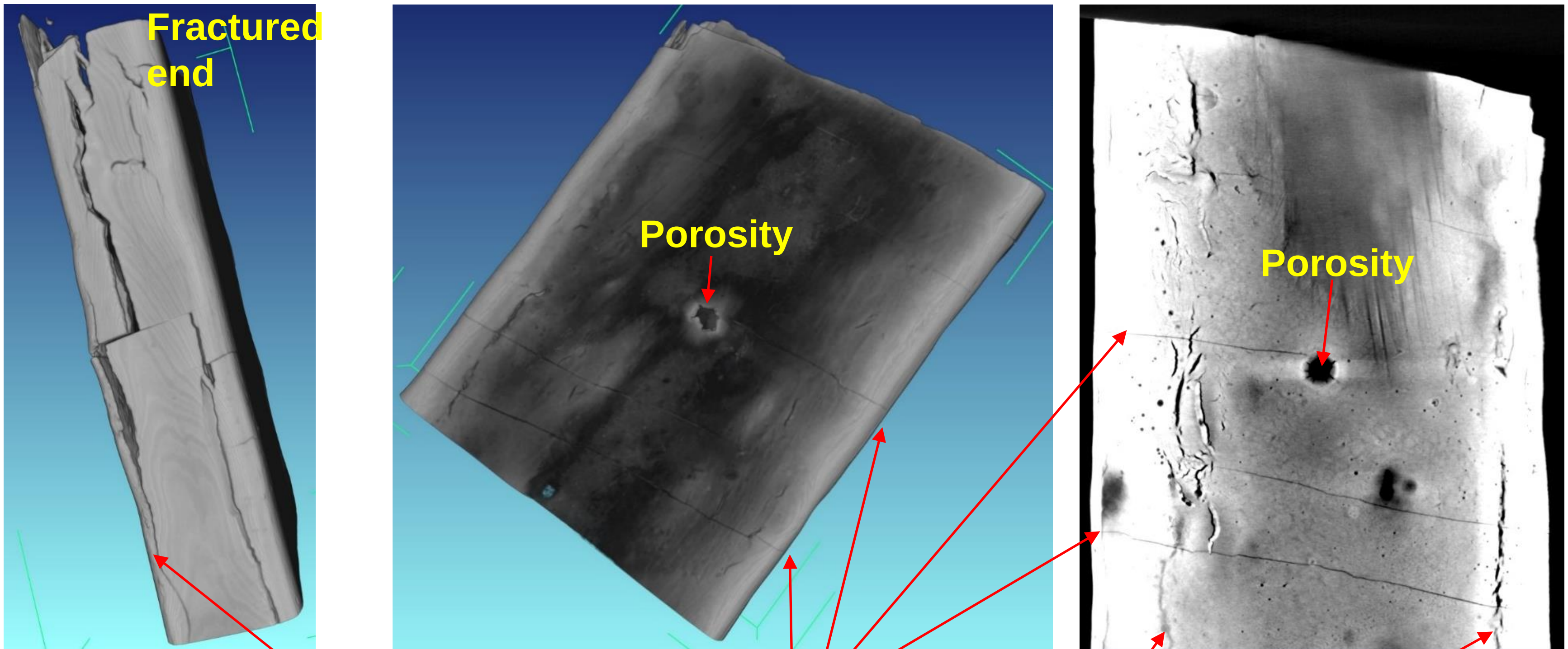
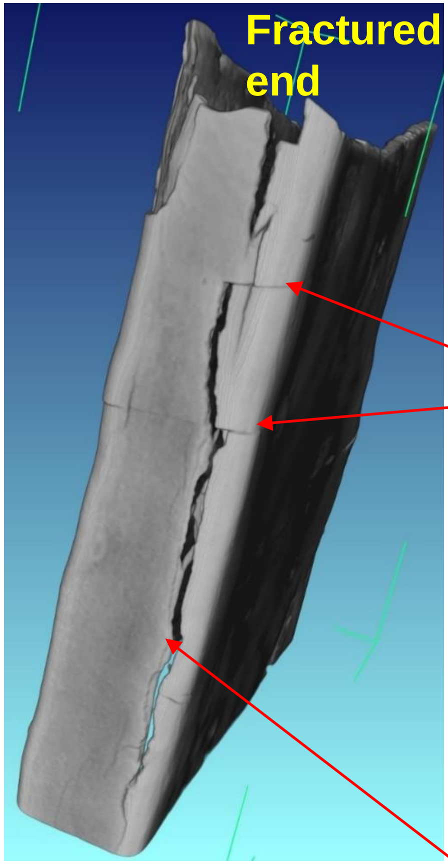
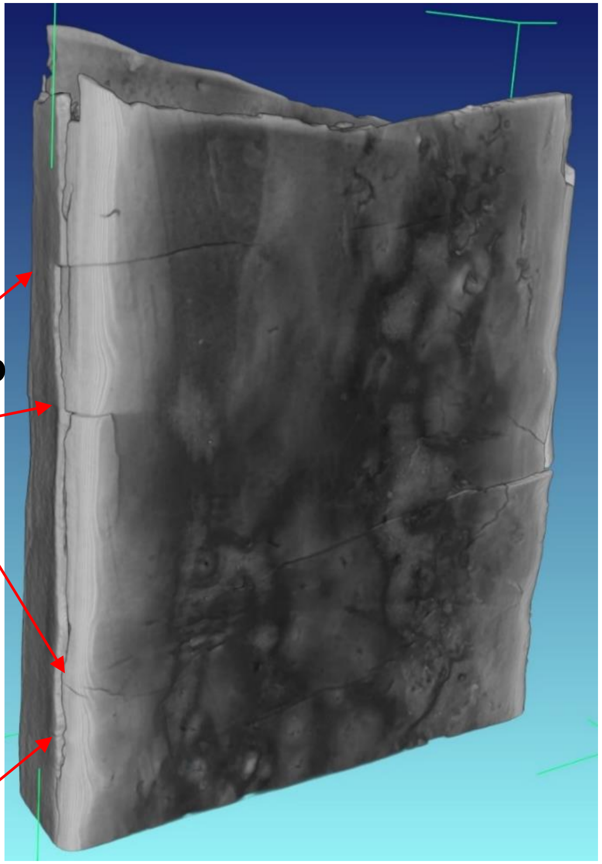


Cracks perpendicular to the loading direction

Cracks parallel to the loading direction

CT Images of Coated 3D Woven CMC after SPLCF Testing at 1204^{o}C for 48hrs at 69 MPa in Steam



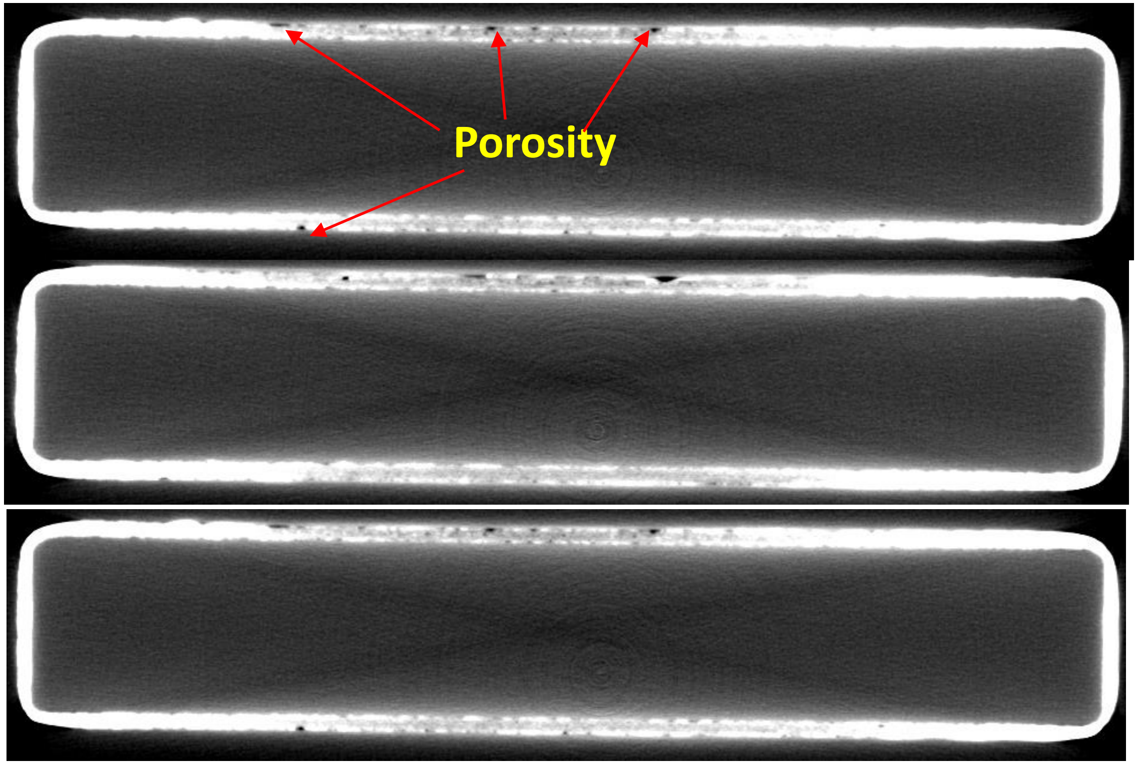


Cracks perpendicular to the loading direction

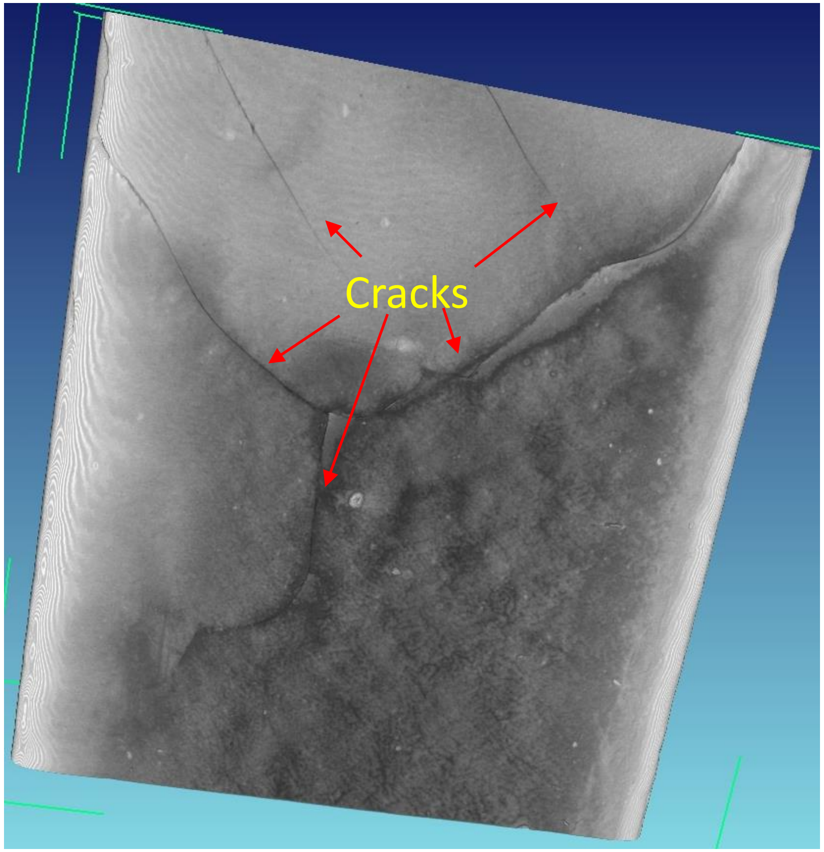
Cracks parallel to the loading direction

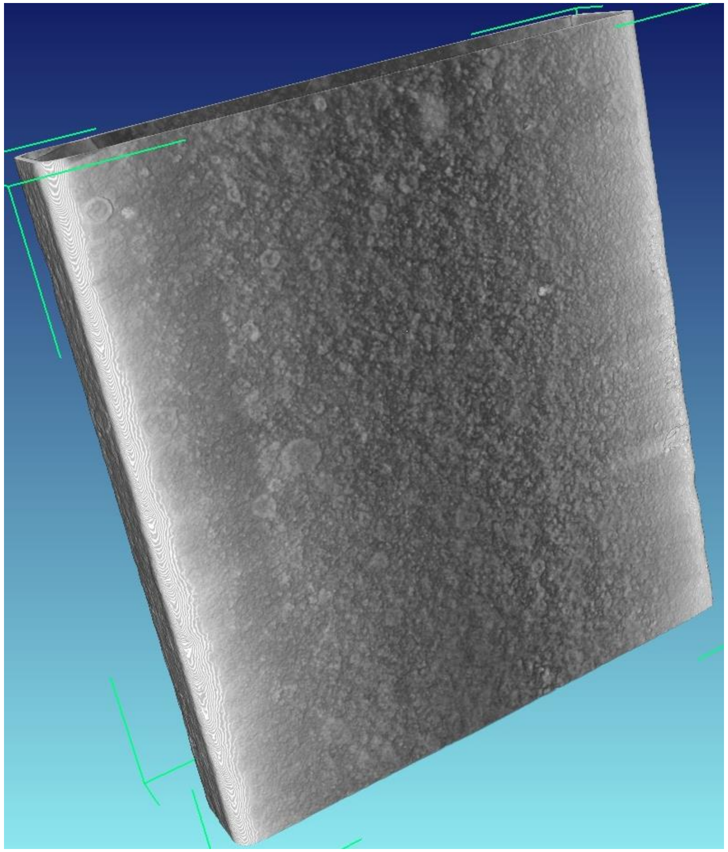
CT Images of EB Coated Hexoloy before SPLCF Testing

EB Coating System: Type-I
Bond Coat: Rare Earth Disilicate(RED)+ Mullite
Top Coat: RED



CT Images of Coated Hexoloy after SPLCF Testing at 69 MPa at 1204°C for 200hrs in Steam





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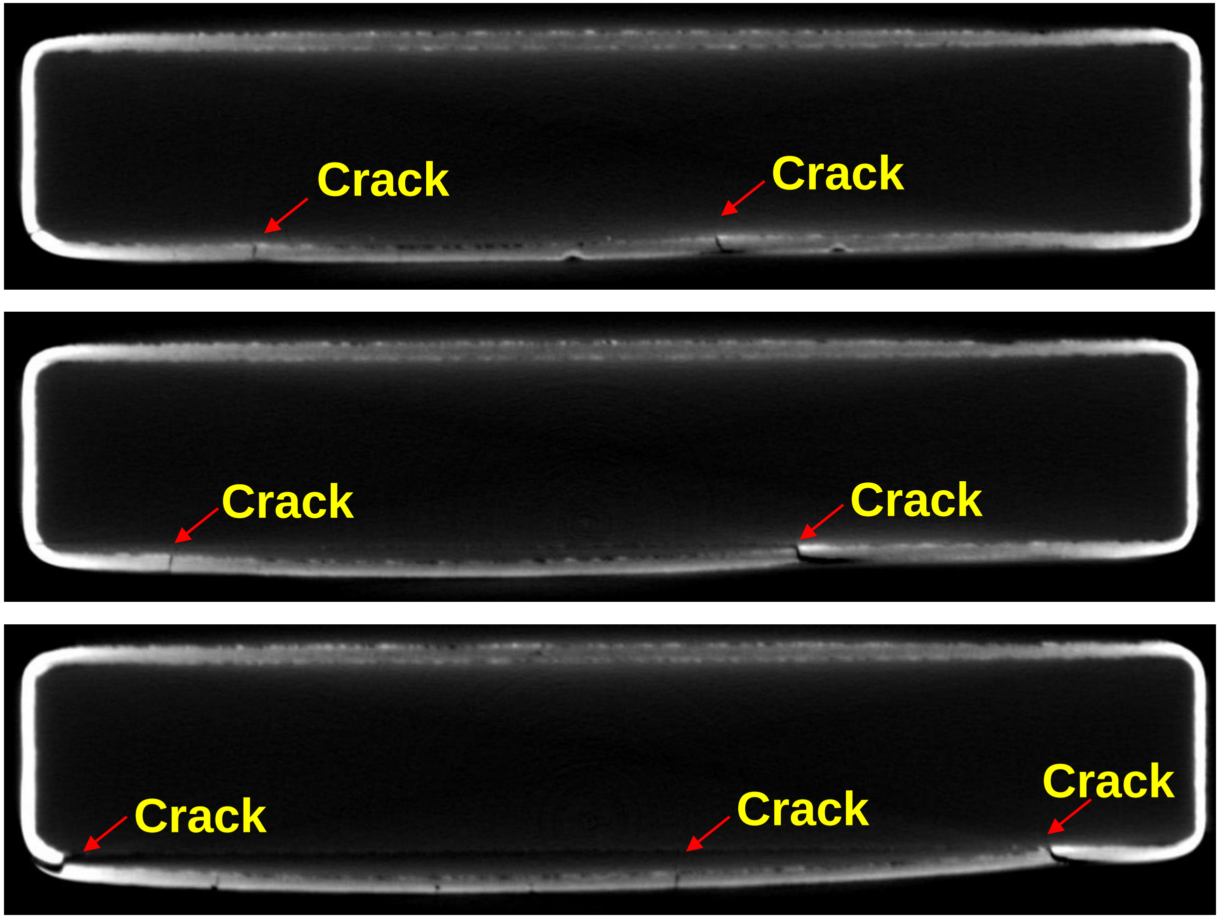
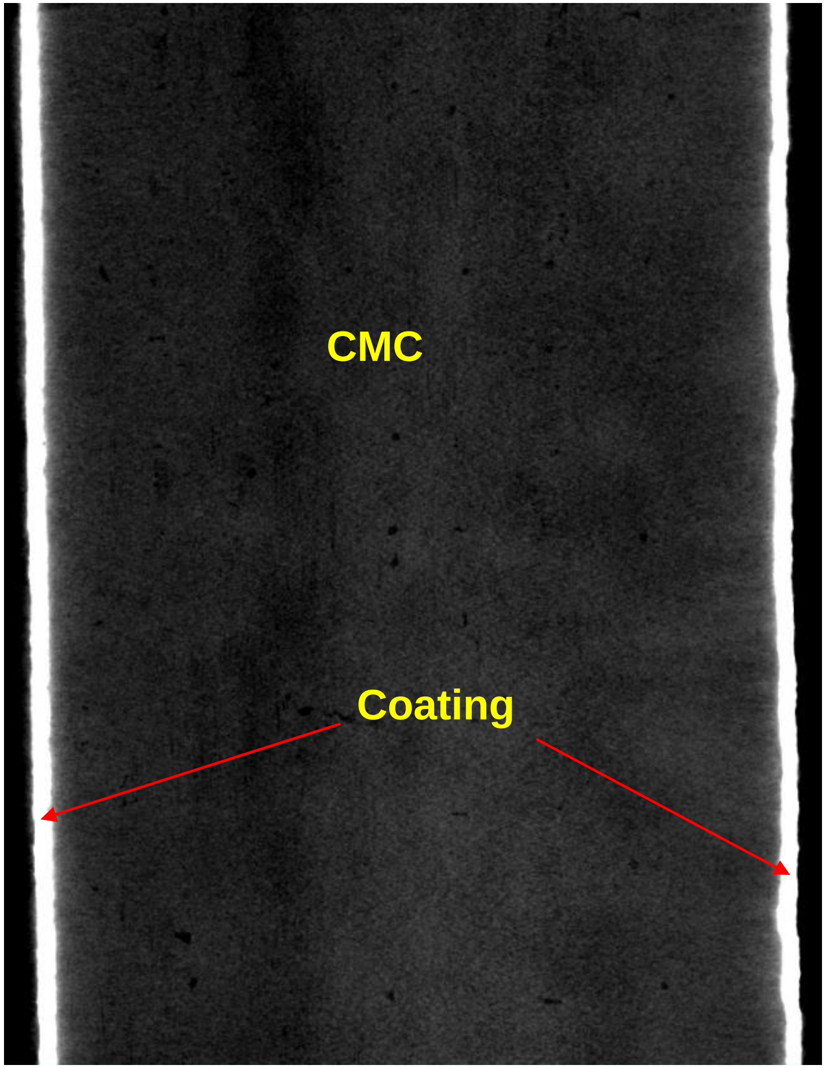
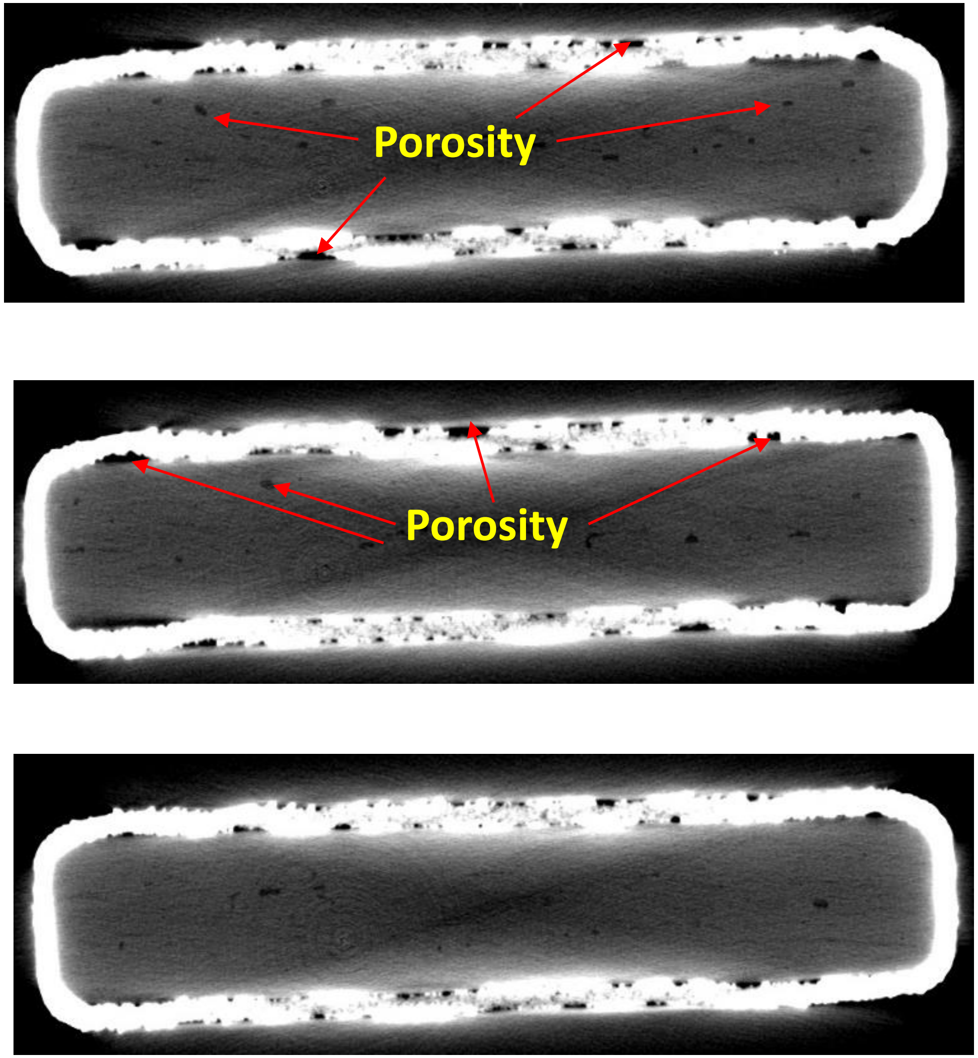


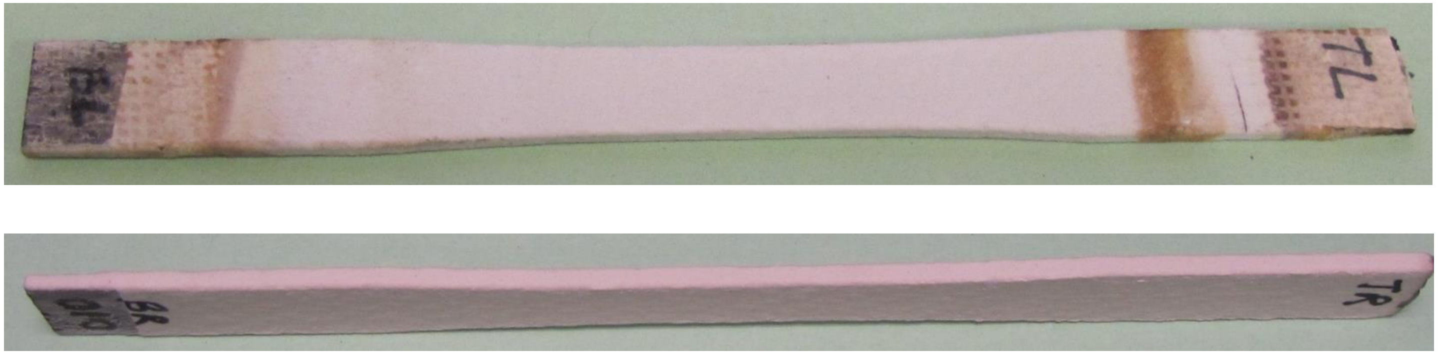
CT Images of EB Coated MI SiC/SiC Composite before SPLCF Testing

EB Coating System: Type-II
Bond Coat: Silicon
Top Coat: Rare Earth Disilicate (RED)

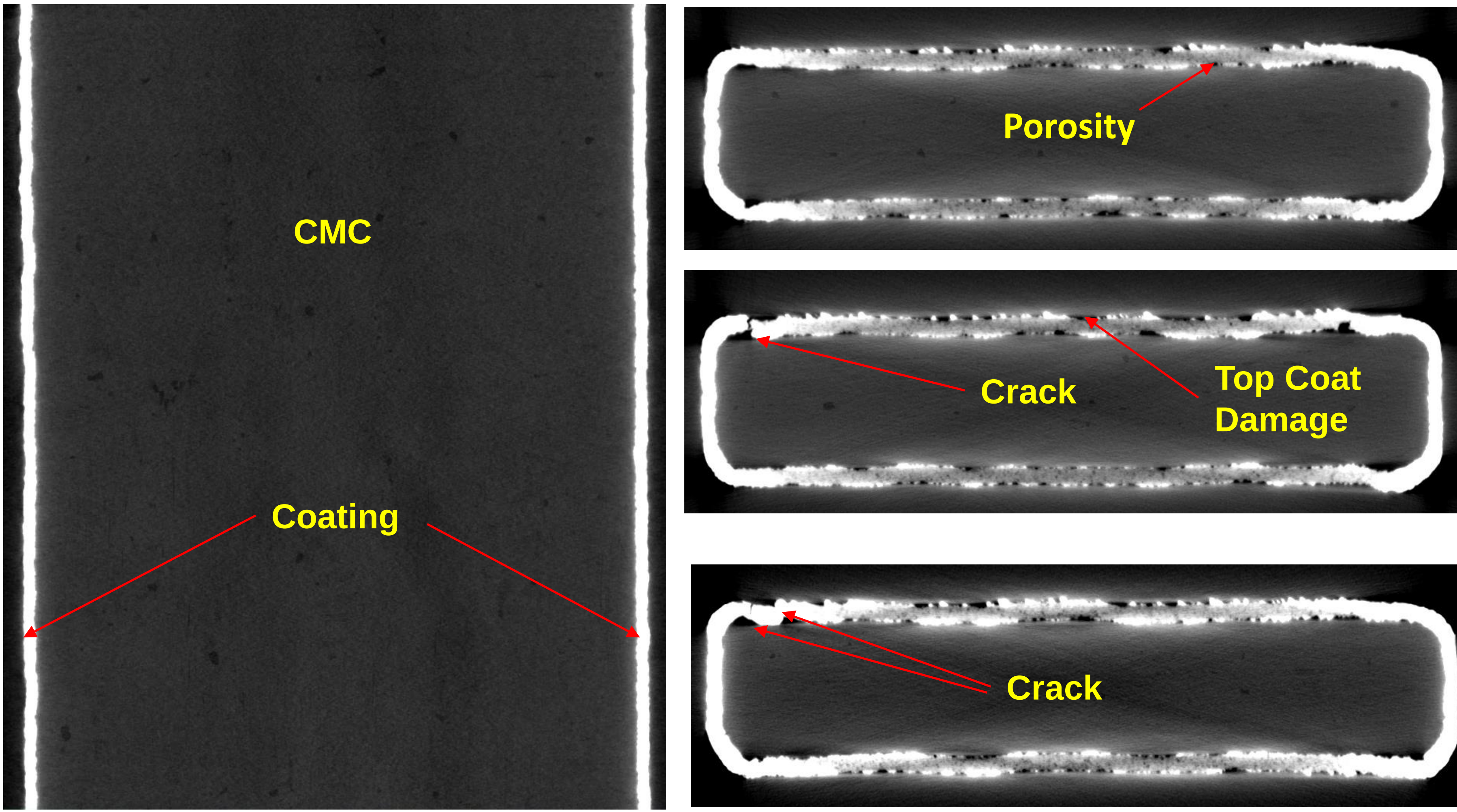




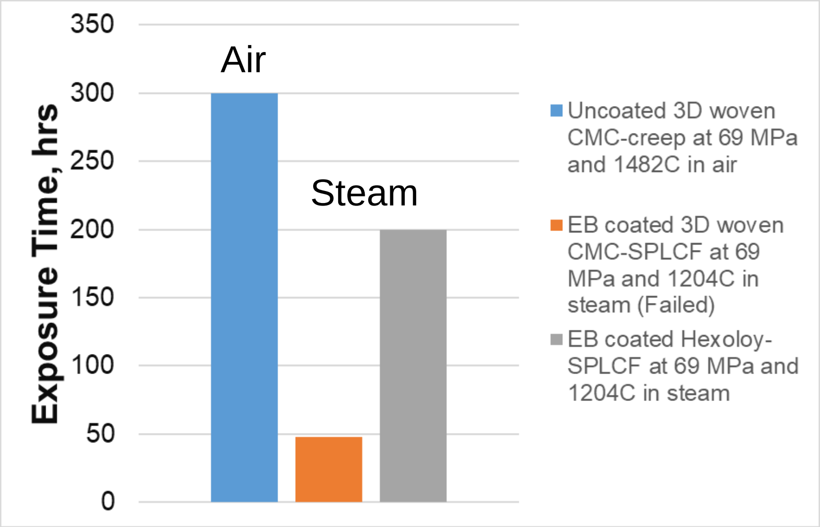
Optical Photographs of EB Coated MI SiC/SiC Composite after SPLCF Testing at 69 MPa at 1204ºC for 200hrs in Steam

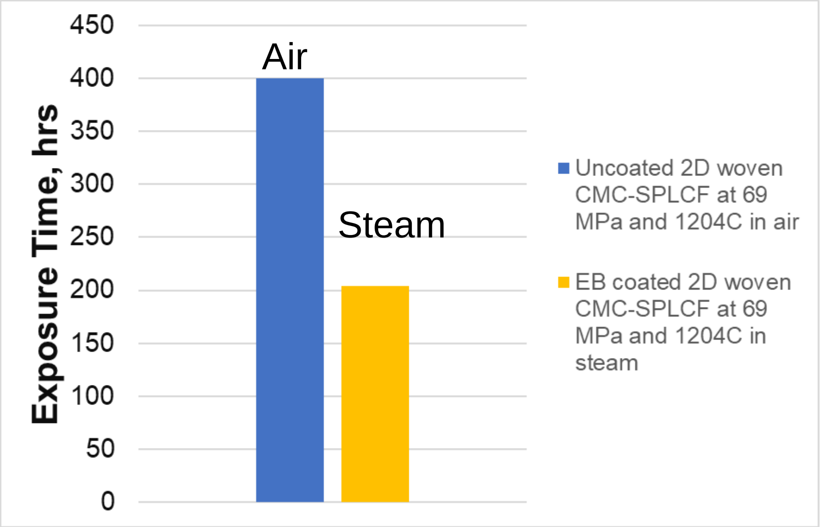


CT Images of EB Coated MI SiC/SiC Composite after SPLCF Testing at 69 MPa at 1204ºC for 200hrs in Steam



History of Uncoated and EB Coated CMCs and Hexoloy Specimens Tested in Air and Steam





Note: In steam environment, Type-I EB coating system does not protect 3D woven SiC/SiC CMC from degradation, but Type-II EB coating does protect 2D woven SiC/SiC CMC.

Room Temperature Tensile Properties of 2D Woven MI, 3D Woven (CVI+PIP) SiC/SiC Composites and Hexoloy

	MI SiC/SiC Composites	(CVI+PIP) SiC/SiC Composites	Hexoloy
Fiber type	Sylramic-iBN	Sylramic-iBN	-
Fiber architecture	2D woven	3D woven	-
Fiber layup	0/90, Balanced	Biased	-
# of plies	8	4	-
Physical density, gm/cc	2.81$\pm$0.06	2.56$\pm$0.11	3.1
Fiber fraction in the loading direction	0.17$\pm$0.01	0.15$\pm$0.01	-
Proportional limit stress(PLS), MPa	128$\pm$31	94	-
Proportional limit strain, %	0.05$\pm$0.01	0.085	-
Primary elastic modulus (E), GPa	240$\pm$25	110	410
Ultimate tensile strength (UTS), MPa	333$\pm$90	250	-
Ultimate tensile strain, %	0.31$\pm$0.12	0.48	-

Room temperature Bend Properties of Plasma Sprayed Stand alone EB Coating Layers

	Mullite	Silicon	Ytterbium Monosilicate
Physical density, gm/cc	2.58$\pm$0.04	2.09$\pm$0.6	7.54$\pm$0.05
Elastic modulus, GPa	109	97	91
Ultimate strength, MPa	28	40	57
Ultimate failure strain, %	0.03	0.04	0.06

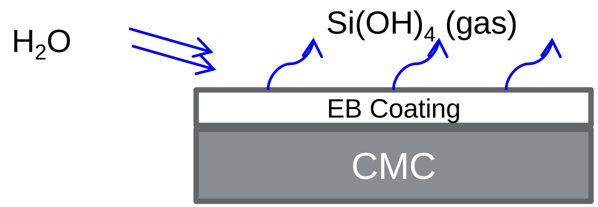
Initial Elastic Strain at Maximum Stress of 69 MPa during SPLCF Test

MI SiC/SiC Composites	(CVI+PIP) SiC/SiC Composites	Hexoloy
0.03	0.06	0.02

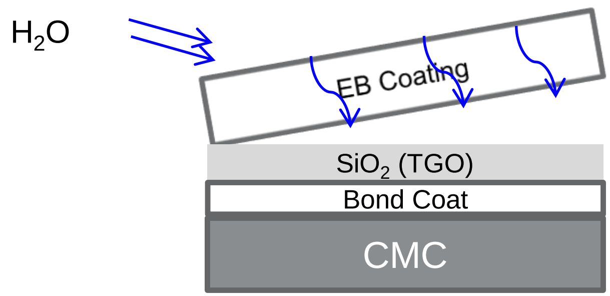
Note: Failure strains of EB coating layers are low. Depending on elastic modulus of CMC substrate and applied load, EB coating layer may crack during initial loading or during the test

EB Coating Failure Modes in Steam Environment Under Thermo-Mechanical Stress

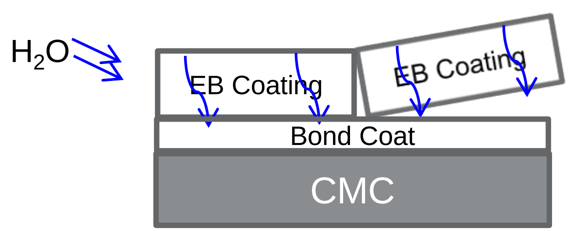
Recession of EBC by water vapor
(Temperature and velocity dependent)



Compliant Bond Coat
Delamination of EB coating due to growth related stresses in TGO



Non Compliant Bond Coat
Cracking and delamination of EB coating, and steam degradation of CMC substrate



K. N. Lee, J. Am. Ceram. Soc., 102(3) 1507-1521 (2019).

Summary of Results and Conclusions

- **EB coated monolithic SiC (Hexoloy), 2D woven MI SiC/SiC and 3D woven SiC/SiC Composites with (CVI+PIP) hybrid matrix were SPLCF tested at 69 MPa at 1204^{o}C in steam environment for up to 200hrs. The 3D woven SiC/SiC composite and Hexoloy specimens were coated with Type-I EB coating and the 2D woven MI SiC/SiC composite specimens were coated with Type-II EB coating.The results indicate the following.**
- **Both types of coating showed coating damage under SPLCF conditions, but the coating damage in 3D woven SiC/SiC composites is extensive compared to that in Hexoloy and 2D MI SiC/SiC composites. In EB coated 3D woven SiC composites, premature failure of the coating starts from the sharp corners and machined surfaces of the specimen, and then moisture ingresses into the CMC via cracked coating, causing CMC ultimate failure. In this composite system, EB coating cracking is due to non-compliant, low toughness bond coat. In EB coated 2D MI SiC/SiC composites, local cracking of the coating was observed, but the composite survived 200hr SPLCF testing. In this composite system, silicon bond coat is compliant. Therefore durability of this composite system depends on growth related stresses in TGO layer and recession of silicon.**

Planned Studies

***For Type-I EB coated 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix*:**

- **Bevel and apply seal coat on CMC specimens. Apply Type-I EB coating. Conduct SPLCF tests at 1200 and 1315^{o}C at 69 to 103 MPa in steam for up to 500hrs. Measure in-plane residual tensile properties and TGO layer thickness.**
- **Develop compliant ceramic bond with improved fracture toughness**

***For Type-II EB coated 2D woven MI SiC/SiC composites*:**

- **Conduct SPLCF tests at 1200 and 1315^{o}C from 69 to 138 MPa in steam for up to 500hrs. Measure in-plane residual tensile properties and TGO layer thickness.**