



# **CMC / EBC Research at NASA Glenn in 2020: Recent Progress and Plans**

Joseph Grady & Craig Robinson

Materials & Structures Division  
NASA Glenn Research Center

[joseph.e.grady@nasa.gov](mailto:joseph.e.grady@nasa.gov)  
[raymond.robinson-1@nasa.gov](mailto:raymond.robinson-1@nasa.gov)

for the  
44th International Conference and Expo on Advanced Ceramics and Composites  
January 26-31, 2020



# CMC Research at NASA Glenn

- CMC Development & Characterization
- Modeling & Validation
- Additive Manufacturing



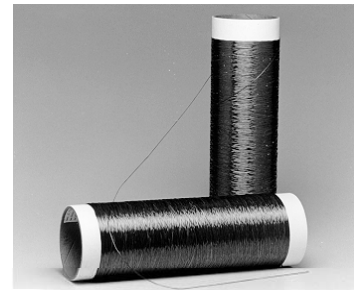
# Material Development and Characterization

- Demonstrated a durable 2700°F CMC / EBC system in a turbine environment
- Established a facility for long-term fatigue testing of CMC's in a steam environment at turbine temperatures
- Implemented Digital Image Correlation capability for full-field strain characterization showing failure progression in cooled CMC
- Measured effect of through-thickness thermal gradient on CMC deformation in creep and fatigue at 2700°F
- Characterized CMAS infiltration of advanced EBC materials

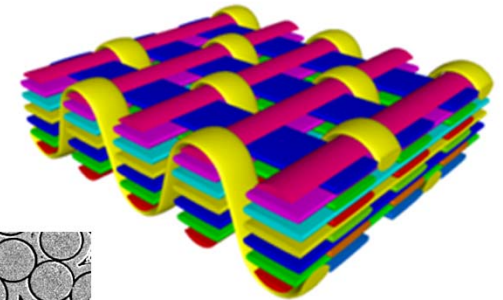


# NASA 2700°F CMC combines three technology advancements

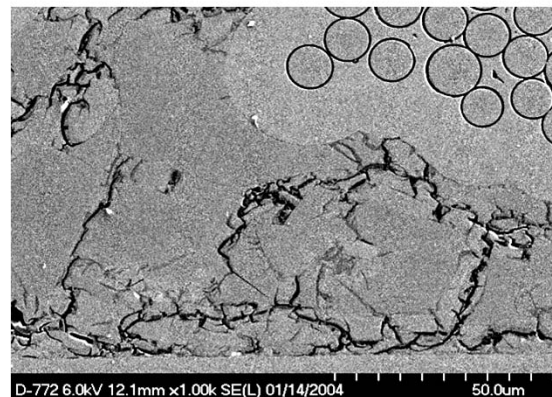
- Creep-resistant Syramic-iBN fiber



- Advanced 3D fiber architecture



- Hybrid CVI-PIP SiC matrix

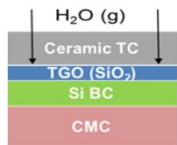


Contact: [Ramakrishna.T.Bhatt@nasa.gov](mailto:Ramakrishna.T.Bhatt@nasa.gov)

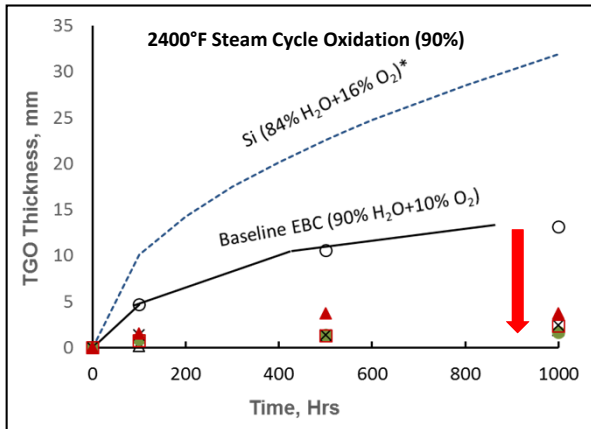
# Recent progress toward a durable 2700°F CMC / EBC

## APS $\text{Yb}_2\text{Si}_2\text{O}_7$ 2400°F EBC Modified for Long Life

- TGO is life-limiting failure mechanism for SOA 2400°F EBC

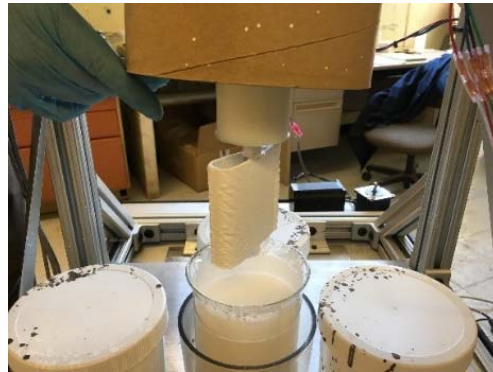


- Certain oxides known to reduce diffusivity in  $\text{SiO}_2$



- Modified EBCs reduced TGO by 80% (~20x life improvement)
- Hypothesis: modifiers dissolve in  $\text{SiO}_2$ , modify structure, slow TGO

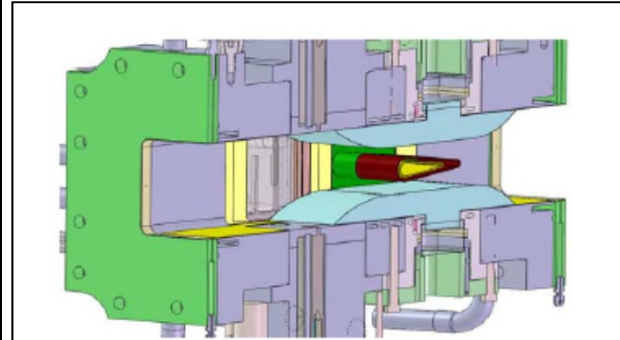
## PS-PVD & Slurry Coat Process Development & Optimization



- Slurry provides economical, non-line of sight, and chemistry friendliness.
- PS-PVD is a hybrid process (plasma and/or vapor) that provides variable microstructure along with non-LOS.
- Both methods demonstrating 2700°F capable coatings.



## Durable 2700°F CMC / EBC material demonstrated



### Cooled CMC / EBC Airfoils Evaluated in Turbine Rig Tests

- Synergy of failure mechanisms
- (3) Test Articles, 45 hrs total
- Compared in-house against commercial EBCs
- 2500-2700°F

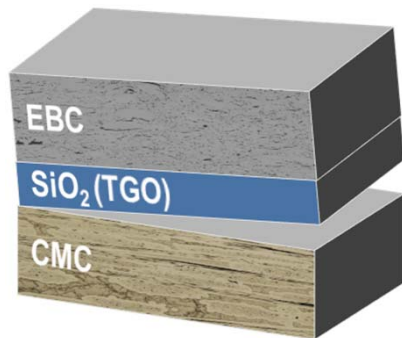


Contact: [Raymond.Robinson-1@nasa.gov](mailto:Raymond.Robinson-1@nasa.gov)

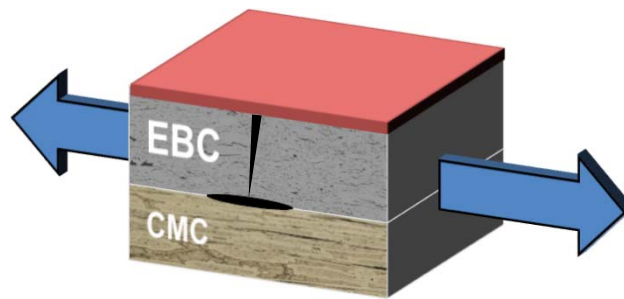
# Fundamental tests characterize CMC/EBC failure modes



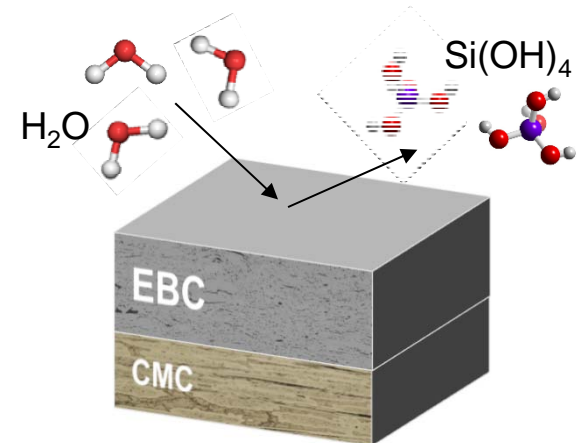
*damage models are incorporated into life prediction codes*



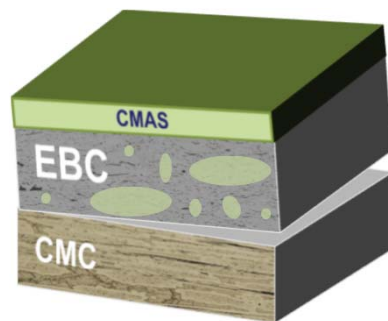
**Steam Oxidation**



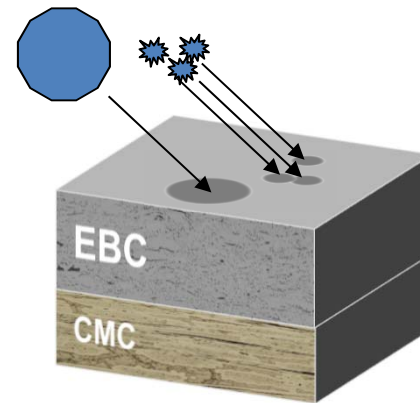
**Thermomechanical Durability**



**Hydroxide Formation/Recession**



**CMAS Attack & Infiltration**



**Erosion and FOD**

**Contact: [Ken.K.Lee@nasa.gov](mailto:Ken.K.Lee@nasa.gov)**

K. N. Lee, "Environmental Barrier Coatings for CMC's"; in *Ceramic Matrix Composites*, Wiley, New York (2015)



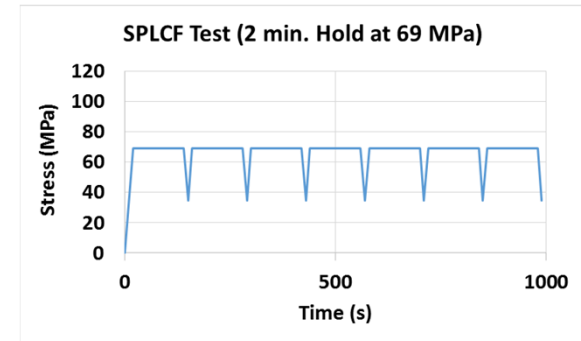
# Capability for 2700 °F fatigue testing in steam environment is being developed

## OBJECTIVE

Characterize fatigue durability of Ceramic Matrix Composites (CMCs) coated with Environmental Barrier Coatings (EBCs) in steam environment up to 2700 °F for future turbine engine components.

## APPROACH

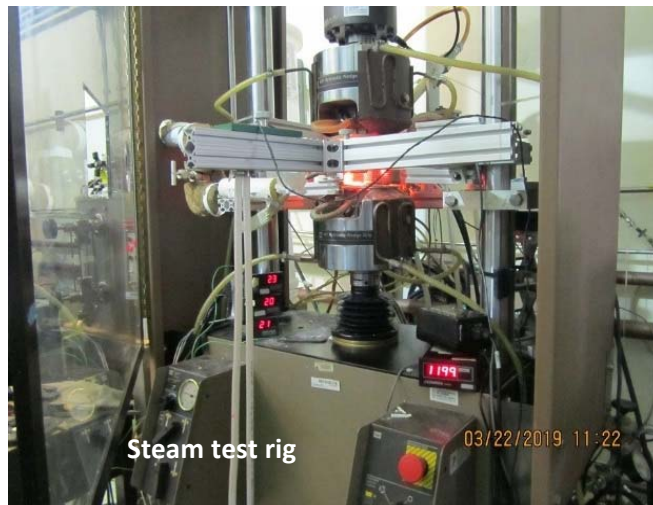
- Initially demonstrate fatigue testing capability at 2200 and 2400 °F in steam environment; eventually develop fatigue testing capability up to 2700 °F in steam.
- Perform sustained peak, low-cycle fatigue (SPLCF) tests on EBC coated MI SiC/SiC composite at 2200 and 2400 °F in steam environment up to 300 hours.
- Develop fatigue testing capability in steam up to 2700°F and perform SPLCF testing on EBC coated CMCs with 3D fiber architectures and hybrid (CVI+PIP) matrices.



**Tensile SPLCF at 2200 °F in Steam**  
Two minute hold at, 69 MPa max. stress; R = 0.5  
Time to failure = 48 hours; ~ 1,200 cycles



Failed in gage section after 48 hours (1,200 cycles)



## SIGNIFICANCE

Assessment of long-term fatigue durability of EBC coated CMCs in steam environment up to 2700°F will enable development of future aero-propulsion engines with greatly improved performance metrics.

## STATUS & ACCOMPLISHMENTS

### SPLCF loading at 2200°F

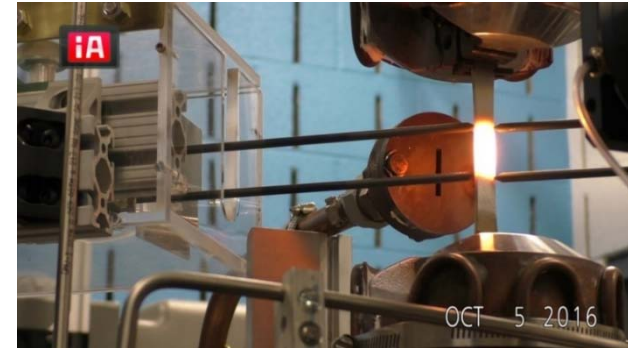
- 3D hybrid CMC/EBC in steam at lasted 160 hours
- Hexoloy with EBC did not fail after 200 hours
- 3D CMC / EBC failed at 48 hours
- Test of MI SiC/SiC with Gen 2 EBC is underway

Contact: Sreeramesh.Kalluri-1@nasa.gov

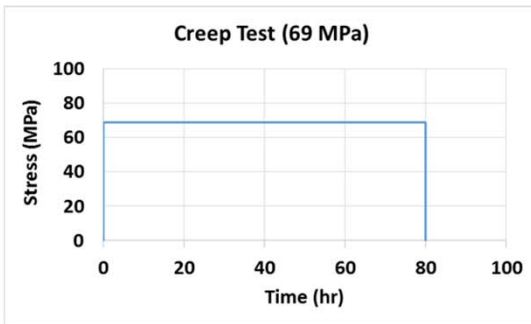
# Effect of thermal gradients on sequential tensile creep and SPLCF testing on SiC/SiC CMCs at 2700 °F

CMCs with 2D & 3D fiber architectures and CVI (2D only), PIP (2D only), and hybrid (CVI+PIP) matrices tested for 80 hr. in creep at 10 ksi [69 MPa] followed by 80 hr. in SPLCF at 10 ksi (8 hr. hold at max stress/cycle) under isothermal (Iso.) & thermal gradient (TG) conditions (2700 °F hot side and 2400 °F cold side)

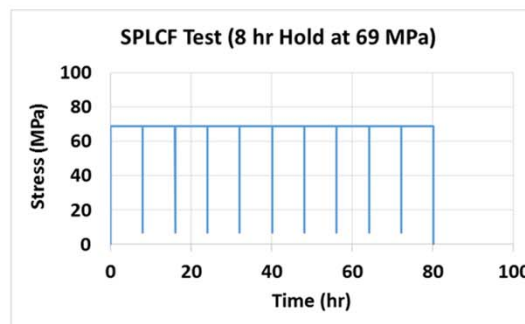
Through-thickness TGs generated in uncoated SiC/SiC CMCs with laser heating and backside air cooling. Front and back side temp. measured with pyrometers and IR camera



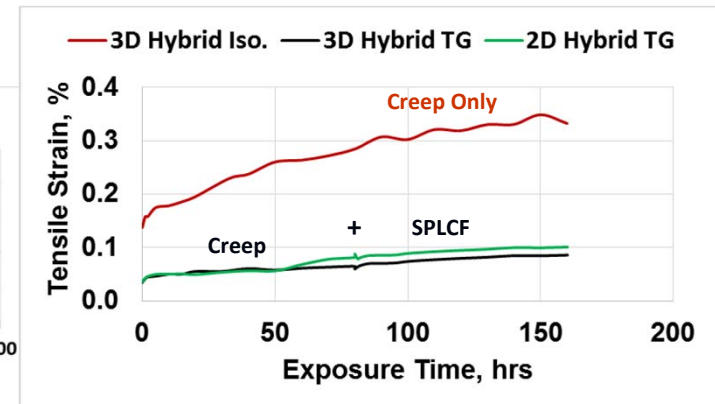
Creep Loading Segment



SPLCF Loading Segment



+



**Isothermal tensile creep test on 3D hybrid CMC generated highest tensile strain followed by TG test on 2D hybrid matrix CMC. Uncoated CMCs sustained steady thermal gradients for a total of 160 hr., with creep at 10 ksi for 80 hr. followed by SPLCF at 10 ksi max. stress for 80 hr., without an EBC.**

Contact: Sreeramesh.Kalluri-1@nasa.gov





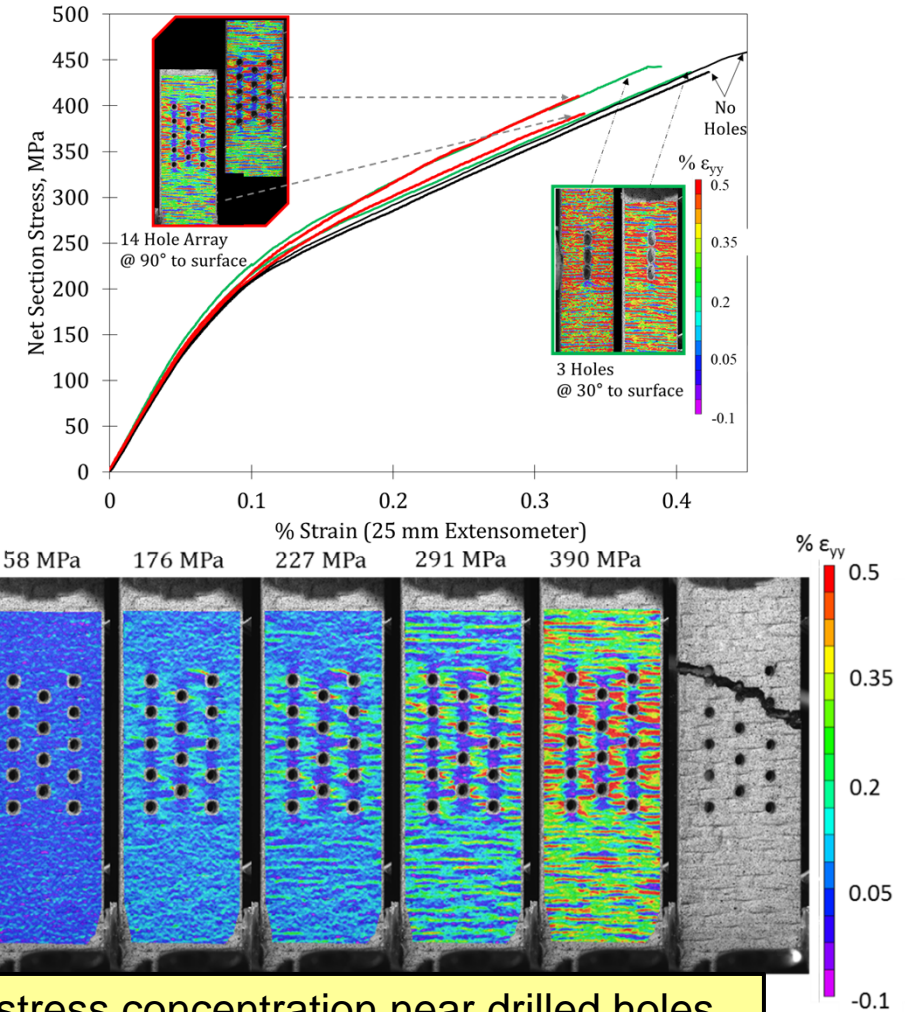
# Digital Image Correlation shows how cooling holes affect damage progression

## Objective

Quantify the effect of holes and hole orientation on the material properties of SiC / SiC composites with EBC. Monitor crack evolution and compare to baseline

## Results

- Tensile samples were tested with cooling holes ultrasonically drilled at 30° and 90° to the loading direction.
- The net-section Proportional Limit (PL) was the same as it was for samples without holes.
- The ultimate strength of samples with 90° holes was reduced by 10%, while samples with 30° holes showed no reduction.
- Local DIC strain accumulated near the 90° holes at stress well below the PL.
- Local DIC strain did not accumulate near 30° holes until the PL.
- For EBC coated samples, local DIC strain indicated that near 90 holes the EBC cracked before the CMC. Near 30° holes, the EBC and CMC cracked at the same time.



Proportional limit stress was not affected by stress concentration near drilled holes.

Contact: [Craig.E.Smith@nasa.gov](mailto:Craig.E.Smith@nasa.gov)

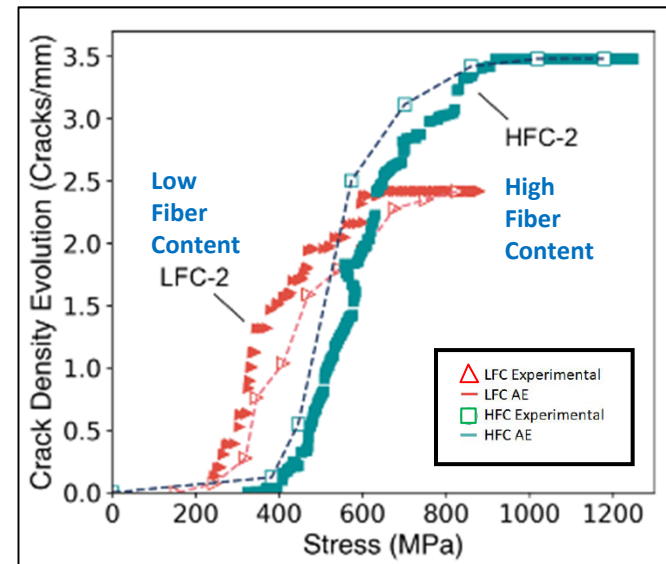
# Multi-Modal Characterization of CMC Damage Accumulation

## Objective:

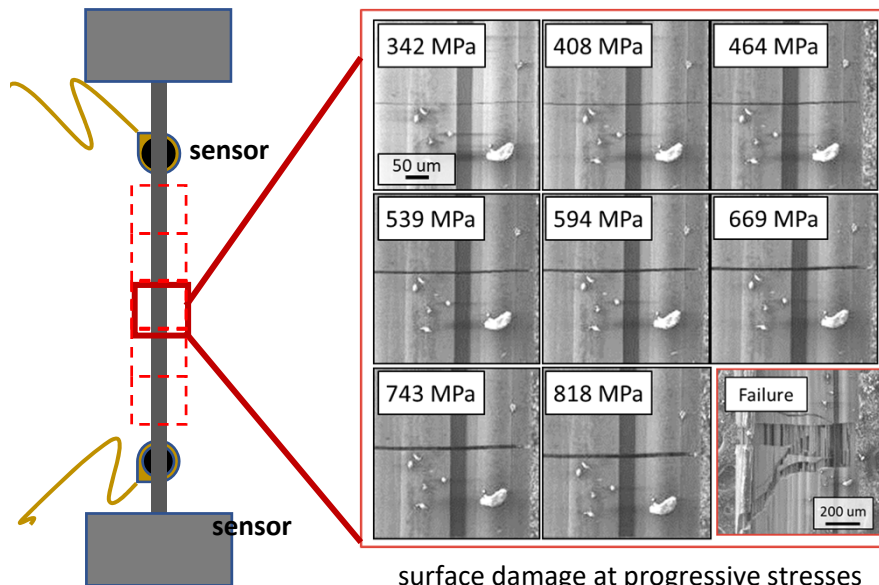
- Quantify damage initiation and evolution at room temperature in SiC/SiC CMCs towards understanding microstructure effect on damage mechanisms

## Approach:

- Conducted tensile tests of CVI SiC/SiC mini-composites in SEM
- Documented damage evolution while making Acoustic Emission measurements to determine damage location and magnitude



Observed crack density vs AE signal



surface damage at progressive stresses

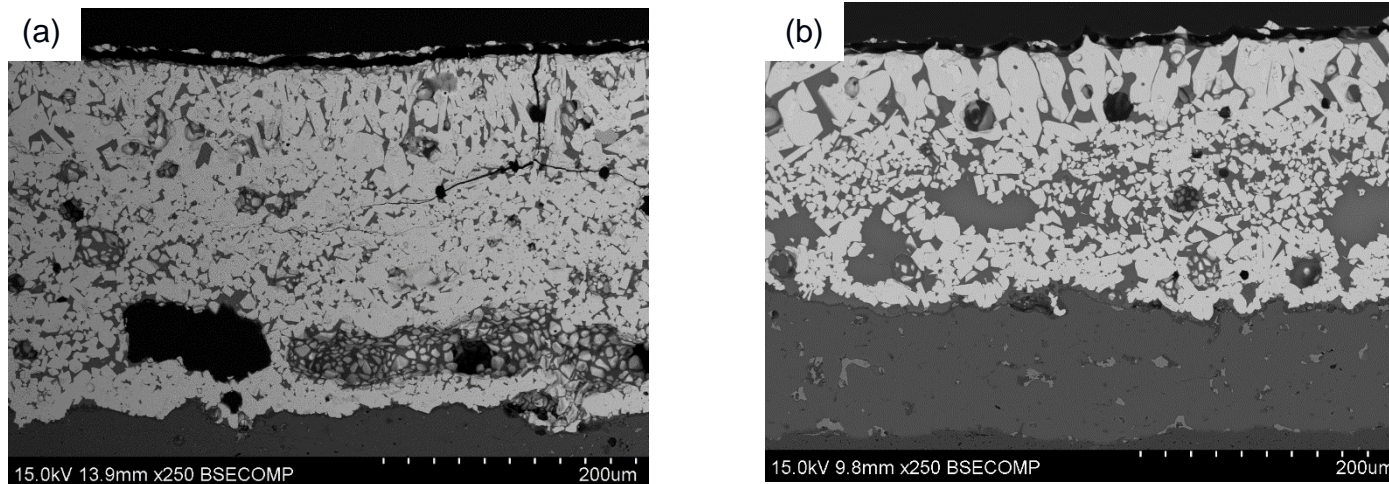
## Results

- Characterized CMC damage in two systems (LFC, HFC)
- Detected damage initiation & progression below the proportional limit
- Correlated AE measurements to microscale damage development
- Obtained Crack Opening Displacements vs. stress

**Contact:** James.D.Kiser@nasa.gov,  
Amjad.S.Almansour@nasa.gov, or  
Bhavana Swaminathan (UCSB)

# CMAS Studies for Advanced EBCs

- Investigation of EBC degradation by molten CMAS at low concentrations
  - Microstructural characterization of first set of specimens exposed to 10 mg/cm<sup>2</sup> of CMAS at 1310°C for 10h completed
  - Heat treatment of second set of specimens with ~1-2 mg/cm<sup>2</sup> of CMAS underway
  - Preliminary results were summarized at MS&T'19 in Portland, OR (Oct 2019)



BSE-SEM micrographs of (a) ytterbium disilicate (YbDS) and (b) YbDS-alumina EBC topcoats exposed to CMAS at 1310°C for 10h revealing degradation via dissolution-precipitation mechanism

- Investigation of thermochemical interactions between CMAS and Apatite EBCs
  - Thermochemical interactions between  $\text{Ca}_2\text{Y}_8(\text{SiO}_4)_6\text{O}_2$  apatite and CMAS have been investigated at 1200 – 1500 C using XRD, SEM, TEM and EDS
  - A paper “Thermochemical interactions between CMAS and  $\text{Ca}_2\text{Y}_8(\text{SiO}_4)_6\text{O}_2$  apatite co-authored by J. Sleeper, A. Garg, V. Wiesner and N.P. Bansal has been published in J. European Ceramic Society, **39** (2019)5380-5390

**Contact: [Narottam.P.Bansal@nasa.gov](mailto:Narottam.P.Bansal@nasa.gov) or [Valerie.L.Wiesner@nasa.gov](mailto:Valerie.L.Wiesner@nasa.gov)**



# CMC / EBC Durability Modeling & Validation

- Developed & validated an enhanced oxidation (TGO) model for silicon bond coat
- Studying TGO formation conditions in steam and effect on the mechanical behavior of coated SiC/SiC minicomposites.
- Validated a computational approach to simulate CMC damage development under flexural fatigue in steam environment

# Reformulation of Oxide Growth Equations for Silicon Bond Coat Oxidation in Environmental Barrier Coatings



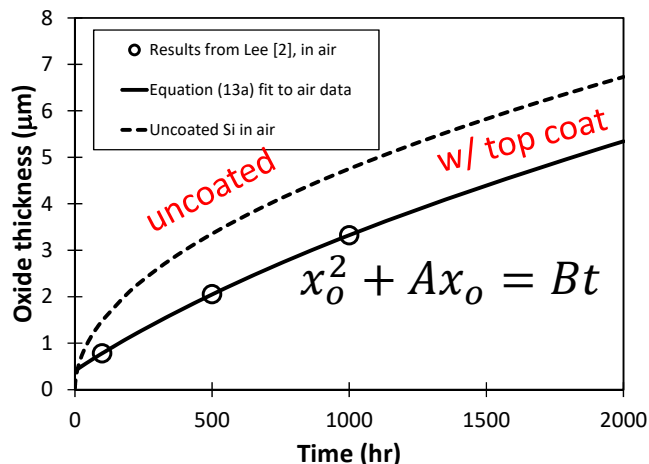
**Objective:** Revisit Deal and Grove's original formulation for silicon oxidation and include the effect of a  $\text{Yb}_2\text{Si}_2\text{O}_7$  top coat.

## Approach:

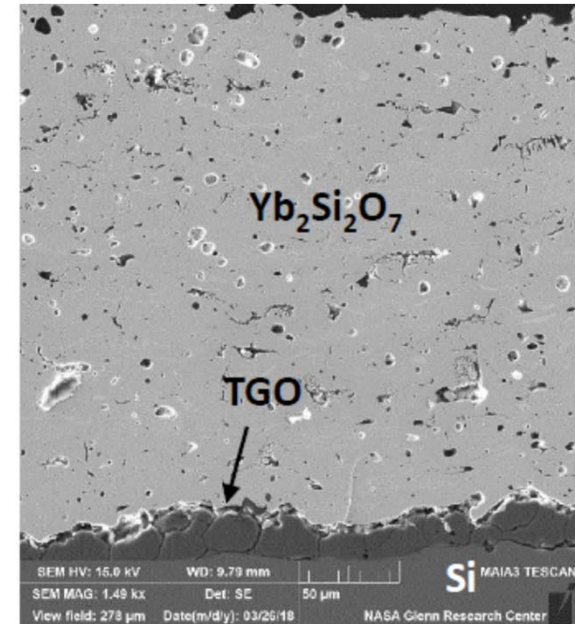
- Assume oxidant diffusion mechanisms through the oxide and coating layers. Derive oxidant mass flux equations.
- Derive equation for oxide thickness as a function of time.

## Results:

- The original linear-parabolic growth equation ( $x_o^2 + Ax_o = Bt$ ) developed for uncoated silicon surfaces is still applicable, except A is modified to include the effect of the top coat:  $A' = A + 2(\gamma_{ox}/\gamma_c)\delta$ , where  $\delta$  is the top coating thickness and  $\gamma_{ox}$  and  $\gamma_c$  are the oxidant permeability in the oxide and coating layers, respectively.



Oxide thickness  $x_o$  vs. time for coated and uncoated silicon surfaces in air at 1316 °C



**Contact:** [Roy.M.Sullivan@nasa.gov](mailto:Roy.M.Sullivan@nasa.gov), "Reformulation of oxide growth equations for oxidation of silicon bond coat in environmental barrier coating systems," *Journal of the European Ceramic Society* Vol. 39 pp. 5403-5409 (2019).

## Accomplishments:

- Understand how top coat affects oxide growth on bond coat
- Simple approach for sizing top coat thickness of EBC

# Effects of High Temperature Steam Exposure on 2700°F EB-Coated SiC/SiC Minicomposites

## Objective:

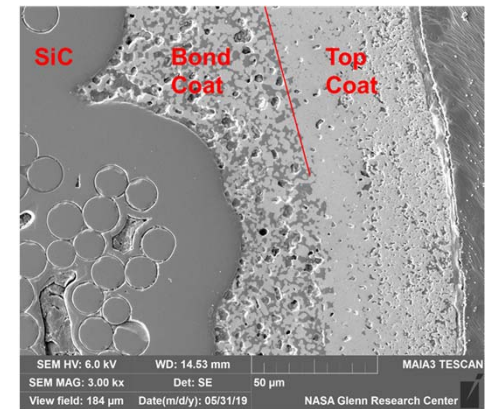
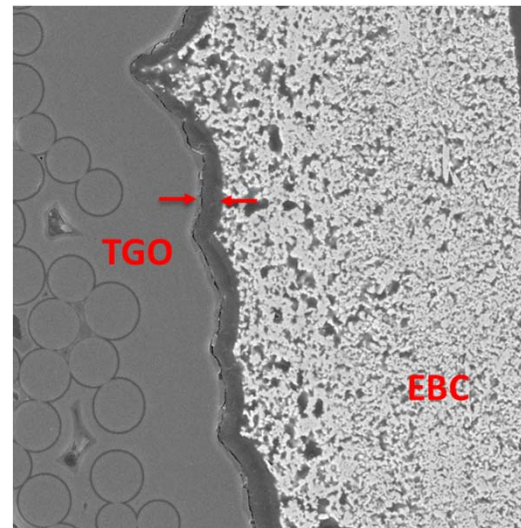
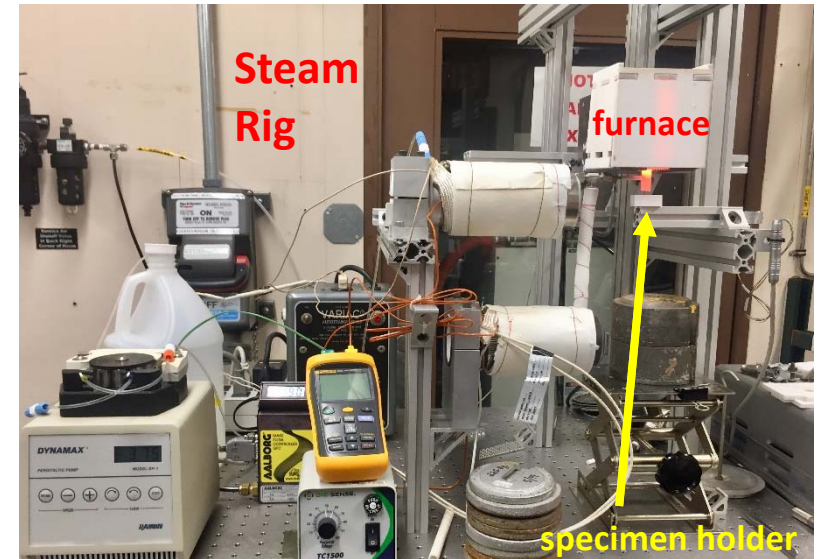
- Establish temperature and time dependence of TGO (thermally grown oxide) growth in steam. Identify effects of TGO growth on EBC and CVI-SiC matrix cracking.

## Approach:

- Coat minicomposites with ytterbium disilicate-based EBC bond coat and top coat
- Expose EB-coated SiC/SiC minicomposites to 2200, 2400, and 2600°F steam environment to establish temperature and time dependence of TGO growth
- Conduct RT tensile tests of coated minicomposites with insitu AE and digital imaging to estimate EBC cracking stress
- Use polished sections to establish TGO growth temperature and time dependence, and quantify EBC and CVI-SiC cracking

## Results:

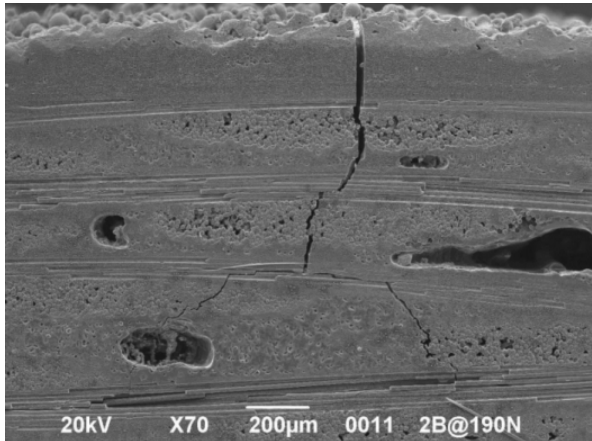
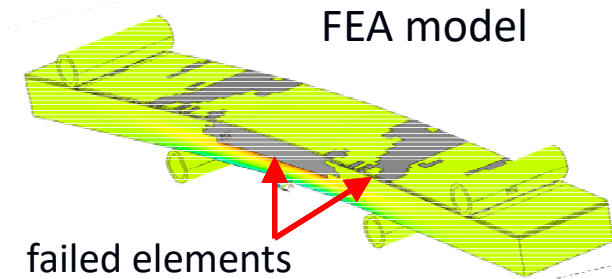
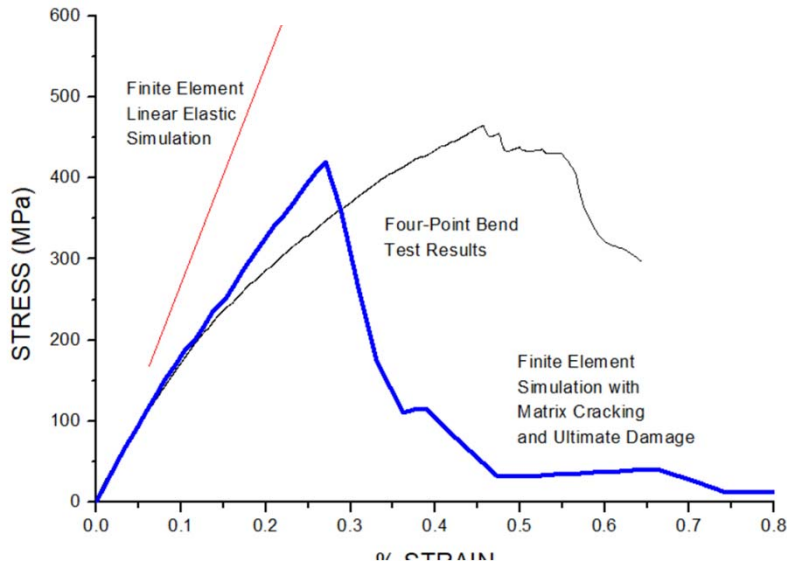
- Measuring TGO thickness for a given exposure condition and comparing that with EBC thickness



**EBC/CMC System**

# Modeling effects of steam environment on CMC durability & failure modes

## Finite Element analysis of CMC 4-point bend specimen



Matrix crack propagation in CMC bend specimen

Results will provide the baseline to assess effects of steam on CMC/EBC fatigue life

Contact: [Jerry.Lang-1@nasa.gov](mailto:Jerry.Lang-1@nasa.gov)



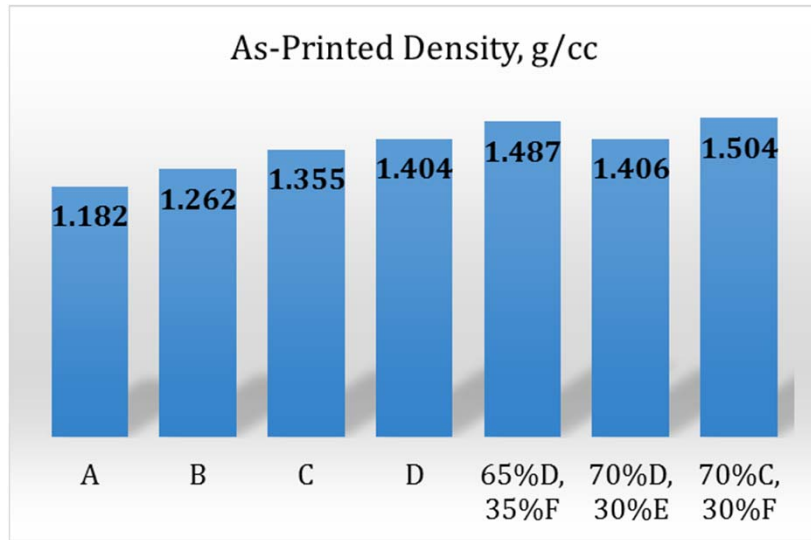
# Additive Manufacturing

- Used Additive Manufacturing Binder Jet process to evaluate SiC processing parameters
- Fabricated lightweight and compact electric motor components using Direct Printing & Selective Laser Sintering processes



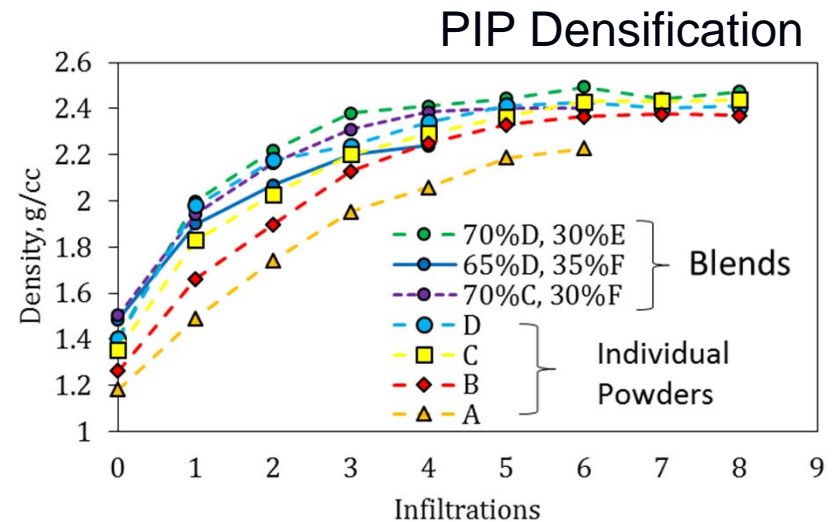
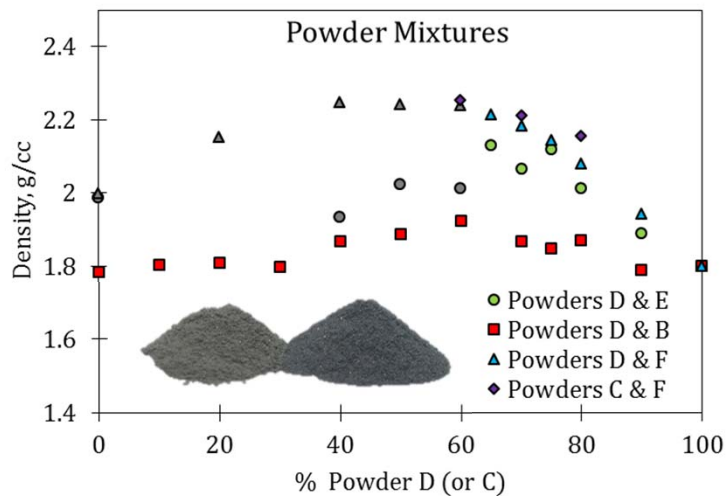


# Densification of Binder Jet Fabricated SiC



Binder Jet Machine

## Density of Green Printed SiC



Contact: [Craig.E.Smith@nasa.gov](mailto:Craig.E.Smith@nasa.gov)

# Additive Manufacturing for Electric Motor Fabrication

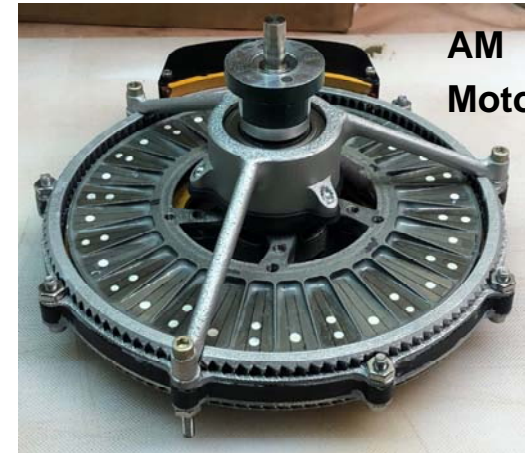


**Objective:** Use additive manufacturing methods to fabricate new motor designs that have significantly higher power density and efficiency



**Baseline Motor**

- Stator designs with direct printing and iron cores, increase max temp, torque and motor constant
- Selective Laser Sintering of structural components reduce mass & improve cooling



**AM Motor**

*Improvements include:*



Light Weight Motor Housing



Integrated Cooling



Higher Temp Stator



A 2x improvement in power density (10 kW/kg) is estimated using AM methods

Contact: [Michael.C.Halbig@nasa.gov](mailto:Michael.C.Halbig@nasa.gov)



# NASA GRC Focus in 2020

## CMC Development & Model Validation

- Determine durability limits and model failure process of 3D Hybrid and Melt-infiltrated CMC under fatigue load in steam environment
- Extend capability for fatigue testing in steam environment to 2700°F
- Extend temperature capability of Digital Image Correlation measurements
- Validate model of cooling hole effect on failure initiation & progression
- Evaluate durability of alternate turbine blade / disk attachment designs

## Additive Manufacturing

- Fabricate stator conductive coils and insulation for a large-scale electric generator using additive manufacturing technologies
- Optimize Binder Jet fabrication & densification processes for SiC with chopped-fiber reinforcement



**This work is sponsored by the Aeronautics Research Mission Directorate**  
and the following projects:

- Advanced Air Transport Technology
- Convergent Aeronautics Solutions
- Transformational Tools and Technologies
- Revolutionary Vertical Lift Technology