



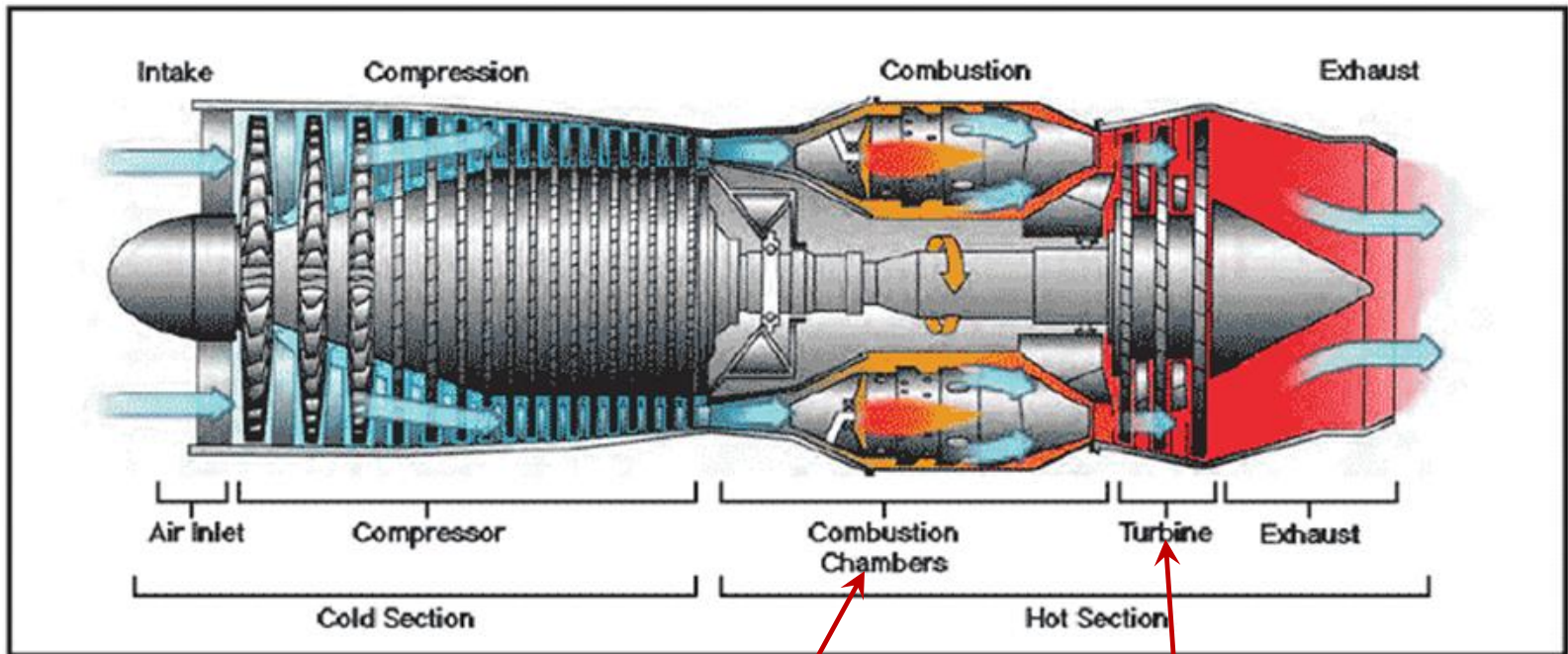
Slurry-Based Environmental Barrier Coatings

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Implementation of ceramic matrix composite (CMC) in Gas Turbines



Combustor liners

Bladetrack (Shroud)
Blades
Vaness

- **CFM International LEAPx**: HPT* Shroud in A320neo (2016) & B737max (2017)
- **GE 9X**: Combustor Liner, HPT* Shroud, HPT* Vanes in B777x (~2019)

* High Pressure Turbine

Evolution of Environmental Barrier Coatings (EBCs) at NASA



- An external coating to protect CMCs from recession by H₂O
- Enabling technology for CMCs

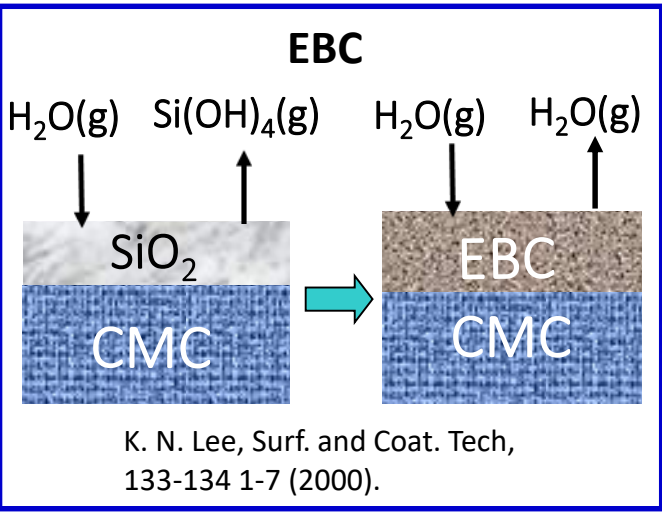
CMC Recession

$$\text{SiO}_2 (s) + 2\text{H}_2\text{O} (g) = \text{Si(OH)}_4 (g)$$

$$\text{Volatility} \propto \frac{v^{1/2} \times P(\text{H}_2\text{O})^2}{(P_{\text{TOTAL}})^{1/2}}$$

v : gas velocity
 P(H₂O) : water vapor pressure
 P_{TOTAL} : total pressure

E. J. Opila et al., Am. Ceram. Soc., 80[1], 197-205 (1997)



Mullite Coating (NASA-1993)

K. N. Lee et al., "J. Am. Ceram. Soc., 78(3) 705-710 (1995).

Gen 1 EBC (EPM: NASA-GE-PW-1997)

Silicon Bond Coat, mp = 1416°C(2580°F)

Gen 2 EBC (UEET: NASA-2003)

Next Gen EBC (NASA Developmental)

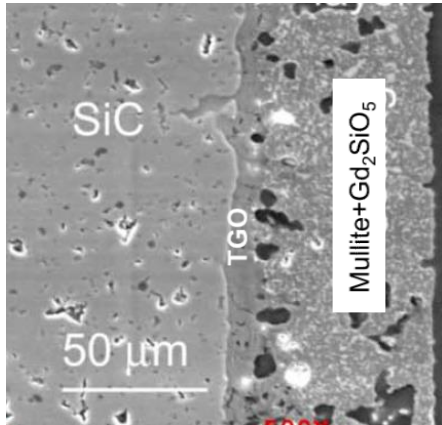
2700°F (1482°C) Bond Coat

Objective

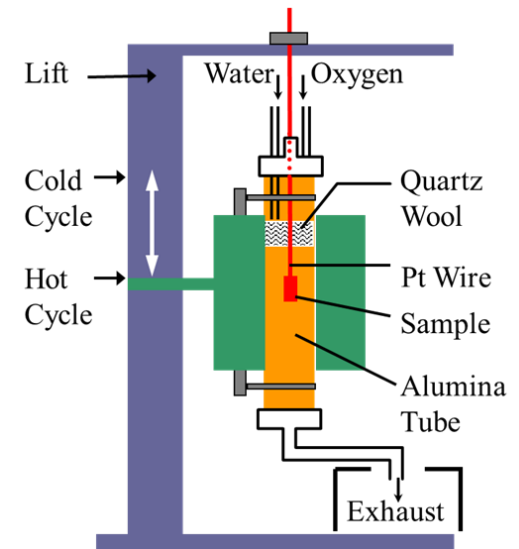
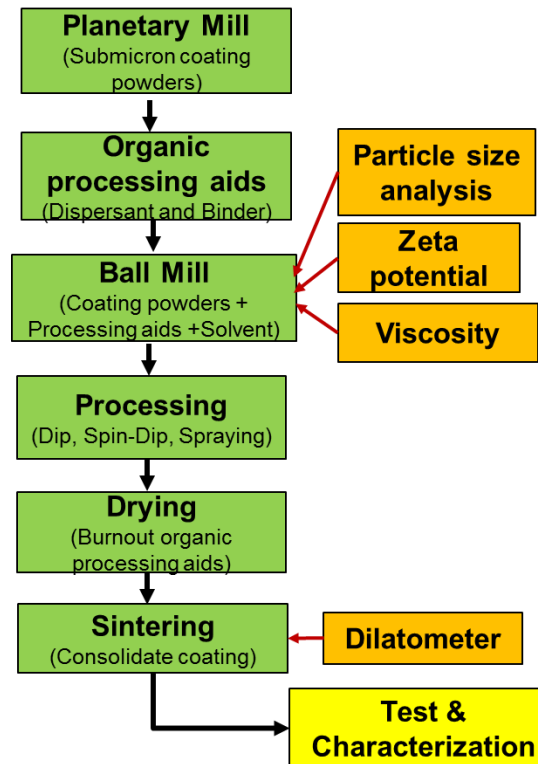
- Develop next Gen EBCs with 2700°F (1482°C) bond coat capability using slurry process
 - Bond coat: $\text{Yb}_2\text{Si}_2\text{O}_7$ -Based, Mullite-Based
 - Sintering aids: Oxide-based
 - Validation: Steam cycling rig, Combustion rigs

Feasibility Demonstrate of Slurry EBC (mid 2000's)

Steam Cycle, 1350°C, 90% H_2O , 100h
TGO ~10 μm



Cleveland State Univ./NASA
(J. Euro. Ceram. Soc., 1123-1130, 2011)

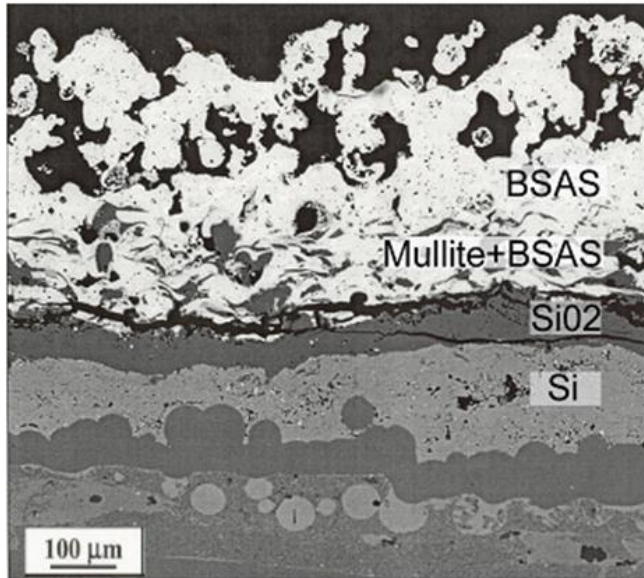


Temp	Up to ~2700F
Velocity	~10 cm/s
Water vapor	Up to ~0.9 atm
Pressure	1 atm

Key EBC Failure Mode: Oxidation-Induced Spallation

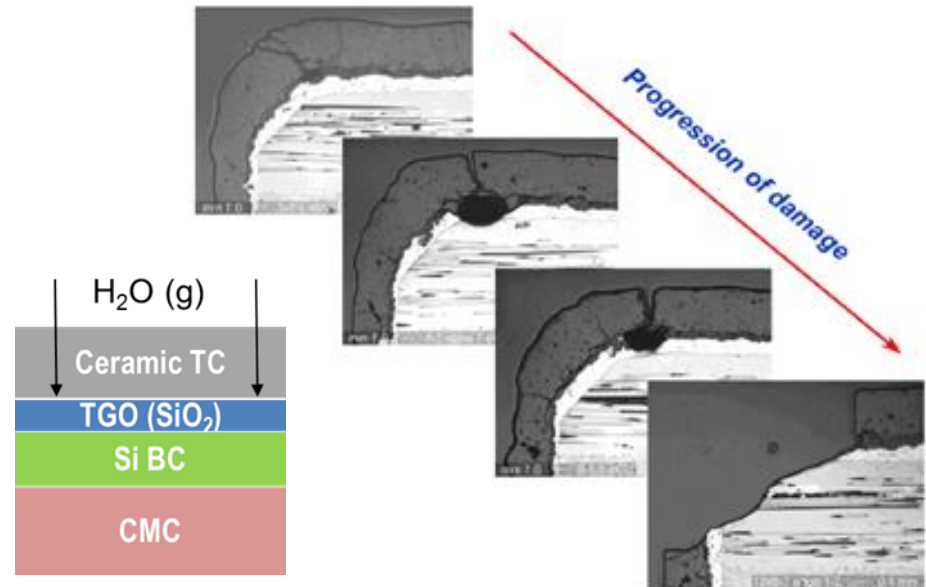


15,144-h Solar Combustor Liner Engine Test



J. Kimmel et al., ASME paper GT2003-38920, ASME TURBO EXPO, Atlanta, GA, USA, June 16-19, 2003.

5,366-h GE Shroud Rig Test



GE Final Report – DOE AMAIGT Program, Dec. 2010

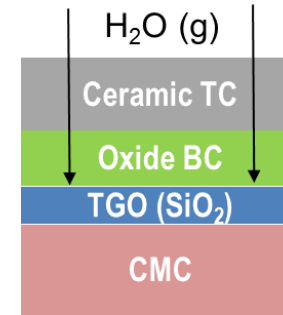
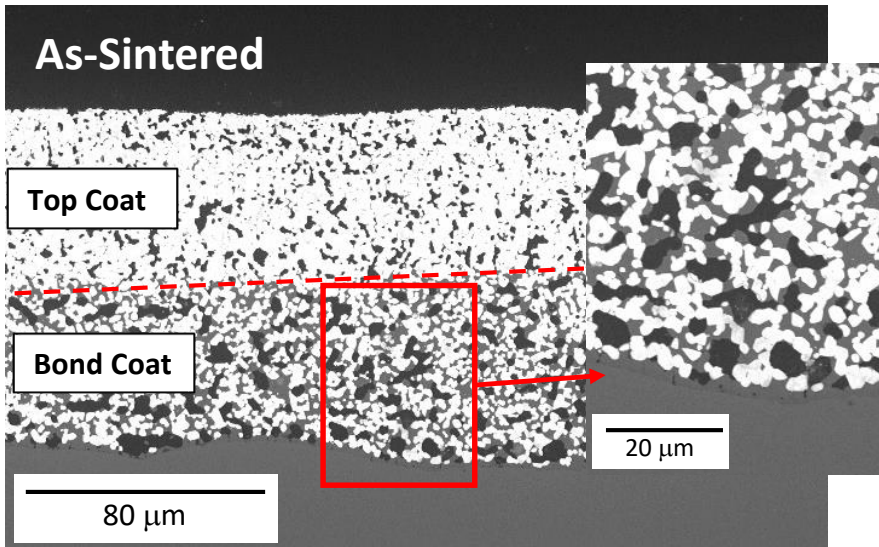
- **Water vapor (H_2O) is the primary oxidant**
- **SiO_2 TGO (thermally grown oxide) forms due to Si bond coat oxidation**
 - Growth stress ($\sim 2.2x$ volume expansion)
 - Phase transformation stress ($\sim 5\%$ volume expansion due to β to α cristobalite at $\sim 200^\circ C$)
 - CTE* mismatch stress (α cristobalite = $10.3 \times 10^{-6}/C$ vs. $Yb_2Si_2O_7 = 4.7$, $Si = 4.4$, $SiC = \sim 5$)
- **Causes large residual stresses that provide the strain energy release rates required to drive EBC delamination cracks**

*Coefficient of thermal expansion

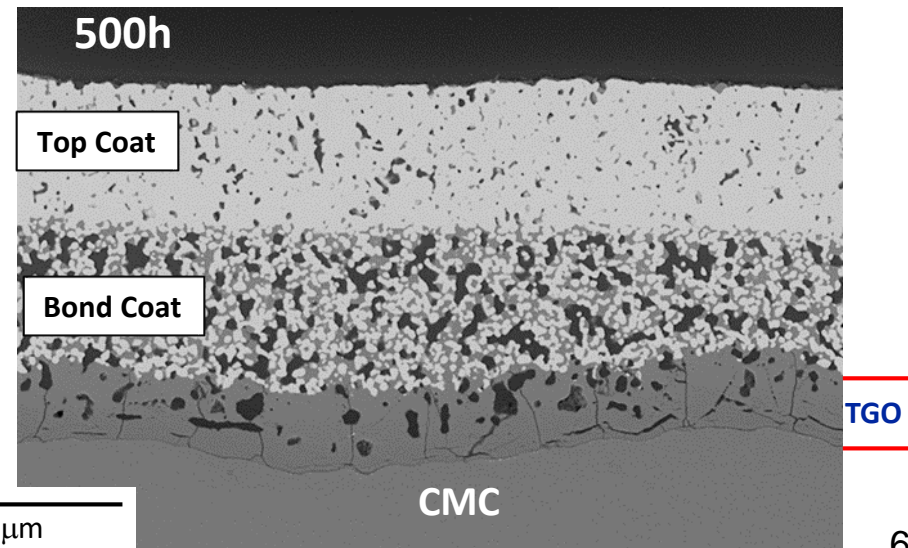
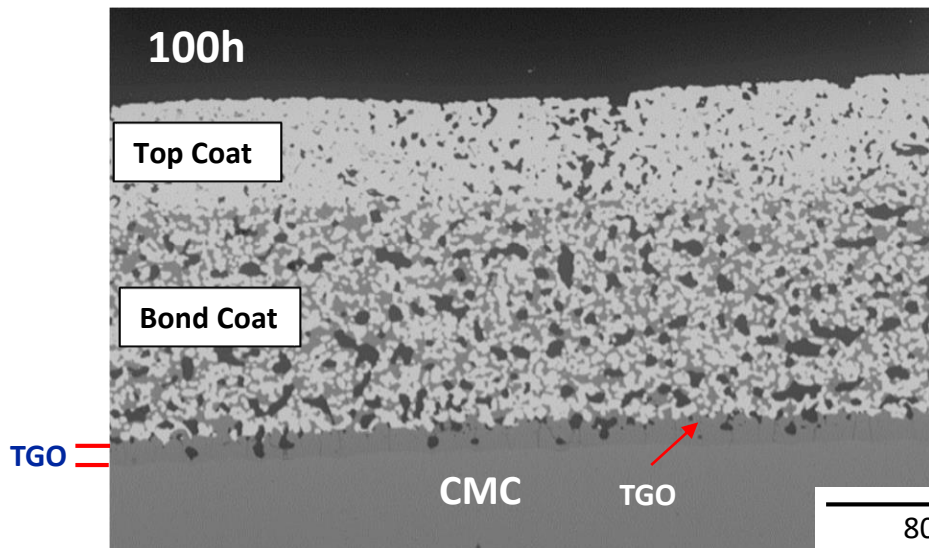
Slurry EBC w/ $\text{Yb}_2\text{Si}_2\text{O}_7$ -Based Bond Coat



[Steam Oxidation: 1 hr at 2600°F (1427°C) / 20 min at $T < 100^\circ\text{C}$, 90% H_2O]



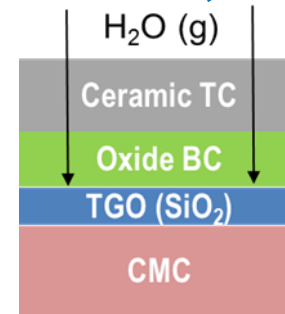
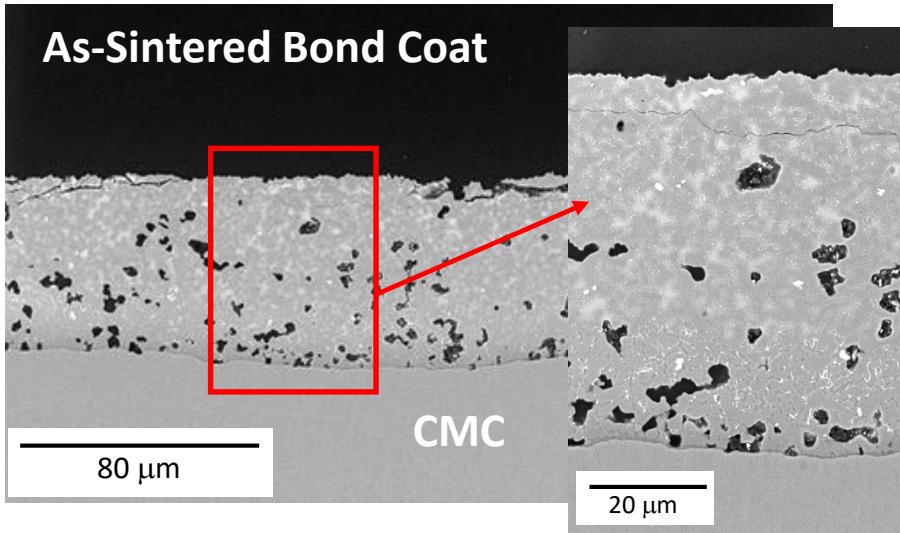
- $\text{Yb}_2\text{Si}_2\text{O}_7$ -based top coat for recession resistance
- Sintered at $T > 2700^\circ\text{F}$ (1482°C)
- Excellent microstructural & chemical stability
- EBC remained adherent after 500h



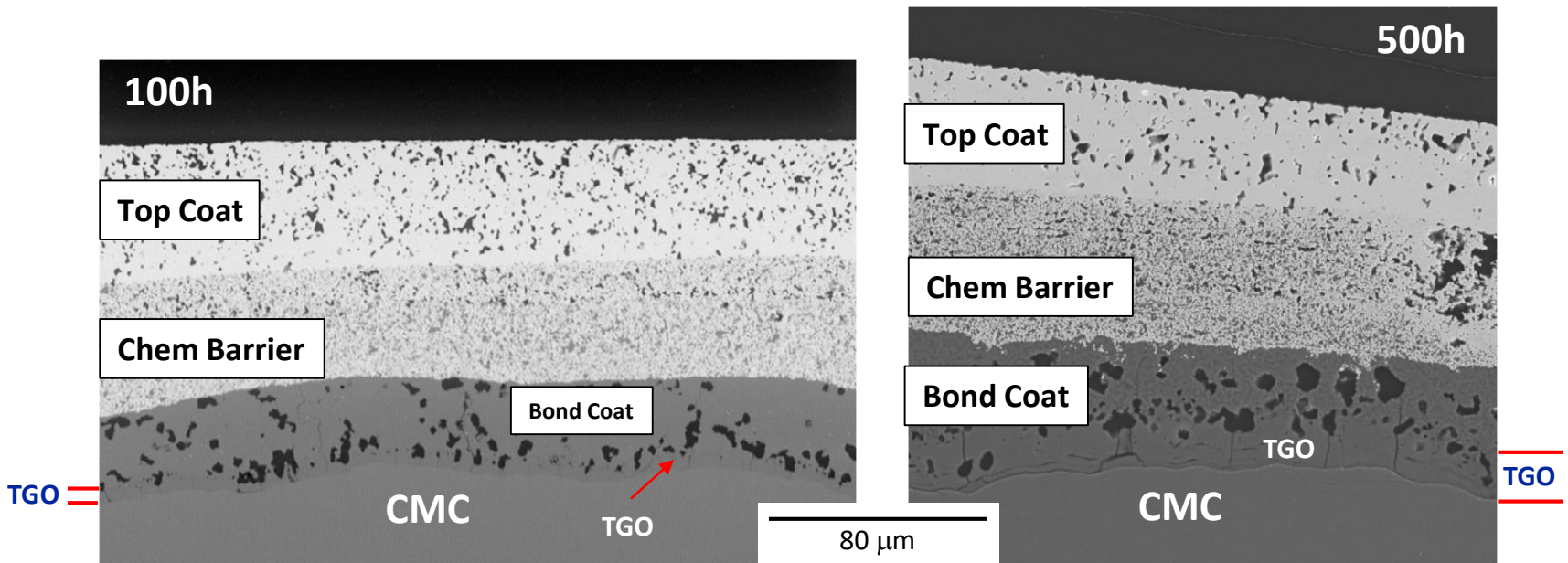
Slurry EBC w/ Mullite-Based Bond Coat



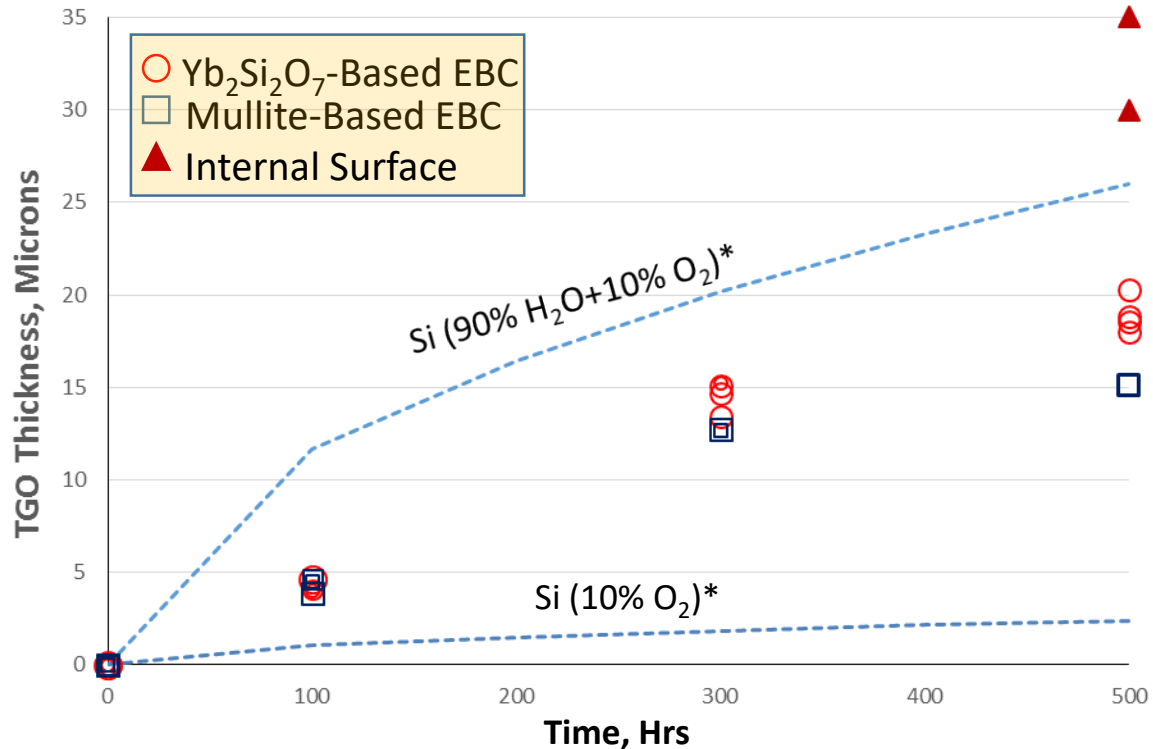
[Steam Oxidation: 1 hr at 2600°F (1427°C) / 20 min at T<100°C, 90% H₂O]



- Yb₂Si₂O₇-based top coat for recession resistance
- HfSiO₄-based chemical barrier
 - Yb₂Si₂O₇-Mullite Eutectic = ~1500°C
- Sintered at T > 2700°F (1482°C)
- Excellent microstructural & chemical stability
- EBC remained adherent after 500h



Steam Oxidation Rates of Slurry EBCs in Steam Cycling [2700°F (1482°C) in 90% H₂O+10% O₂, 1h cycles]



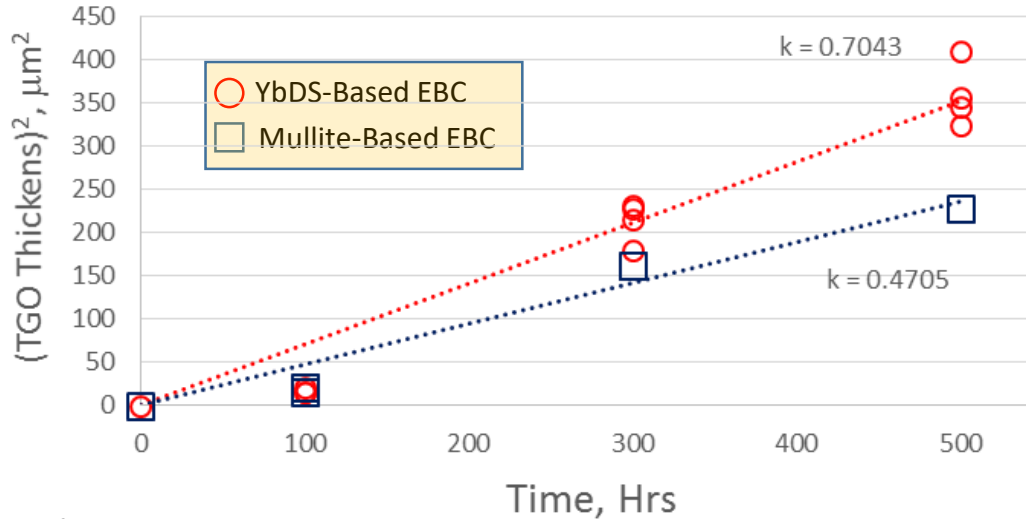
* Extrapolated from Deal and Grove, J. Appl. Phys., 36[12] 3770-78 (1965)

- Both slurry coatings provide substantial mitigation of steam oxidation
 - Mullite-based bond coat shows slightly lower TGO growth rates
- TGO is thicker on the internal surface (~25%) than under the EBC
 - Oxidation mechanism studies in progress



Parabolic Oxidation Plot of Slurry EBCs

[1 hr at 2600°F (1427°C) / 20 min at T<100°C, 90% H₂O]

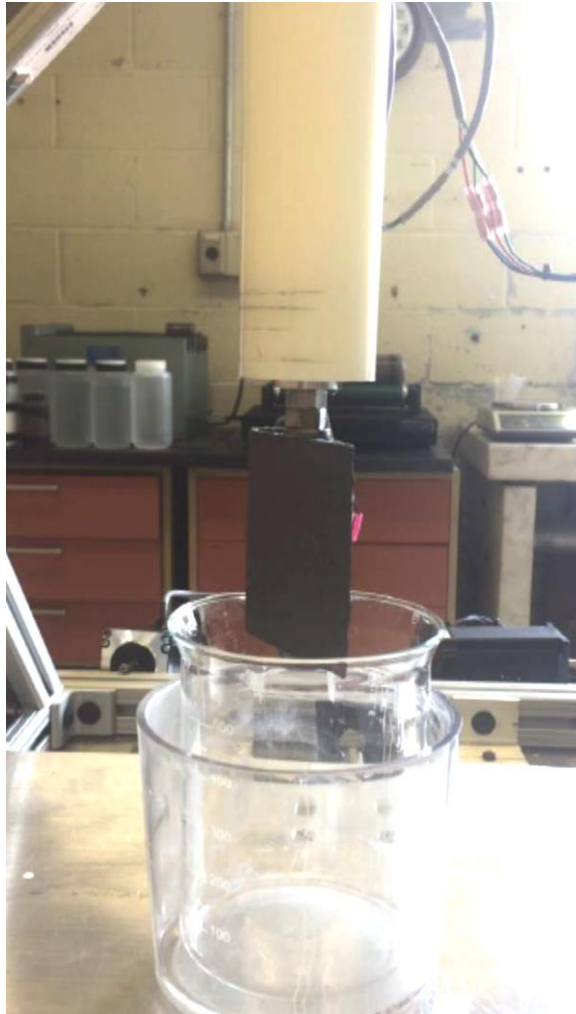


*Chemical vapor infiltration

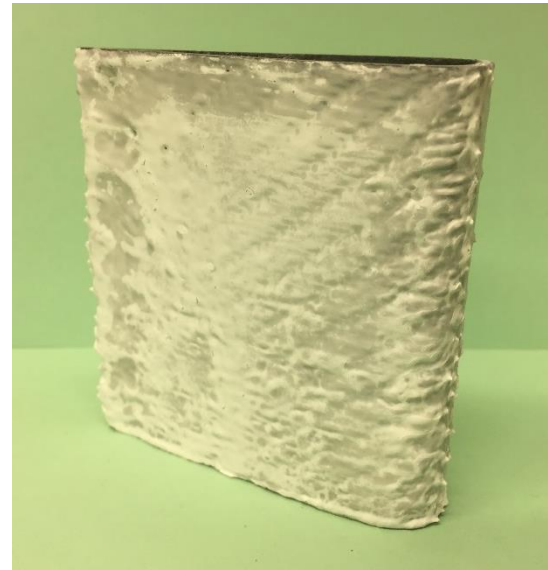
Substrate	CVI* CMC	CVI* CMC	CVI* CMC
Coating	Yb ₂ Si ₂ O ₇ -Based	Mullite-Based	Uncoated (Internal Surface)
k (μm ² /h)	0.70	0.47	2.11

- Parabolic rate constant is ~50% higher with Yb₂Si₂O₇-Based bond coat
- Uncoated CMC (Internal surface) shows ~2x – 3.5x higher parabolic rate constant than coated CMC

Spin-Dip Coater

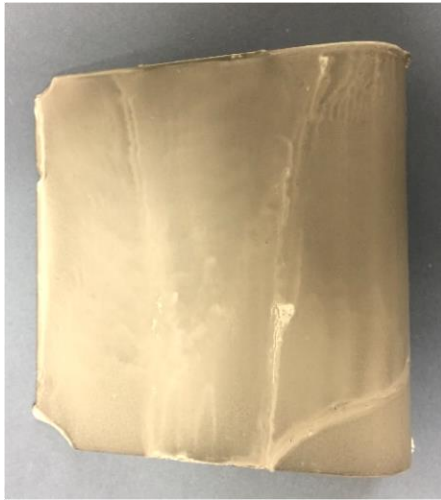


Slurry EBC-coated
3"x3" CMC Airfoil

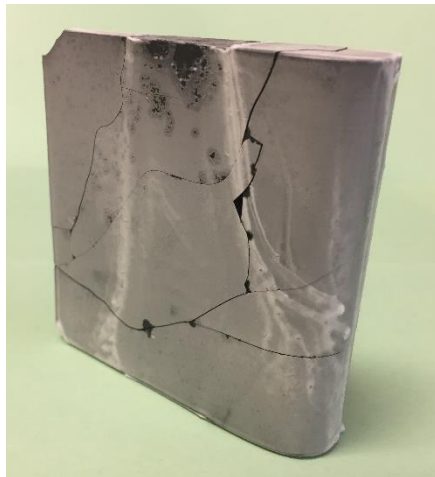


- Durability testing in a combustion rig at 2500°F-2700°F (1371-1482C) is in progress
- Steam cycling test of witness coupons at 2600°F (1427C) in progress

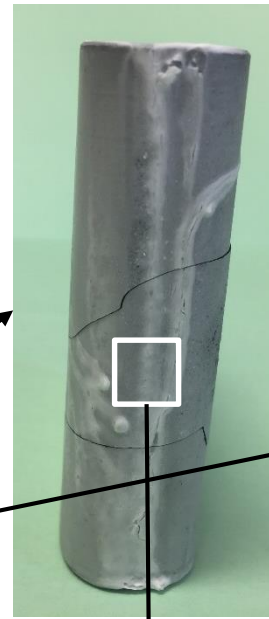
EBC on Monolithic SiC (3" x 3") – 1st Airfoil Spin-Dip Trial



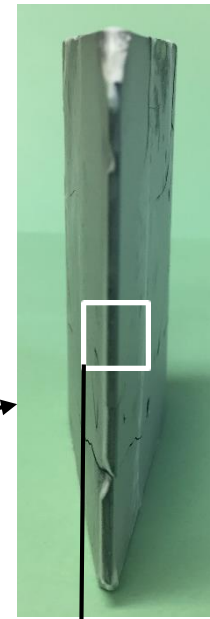
As-Coated



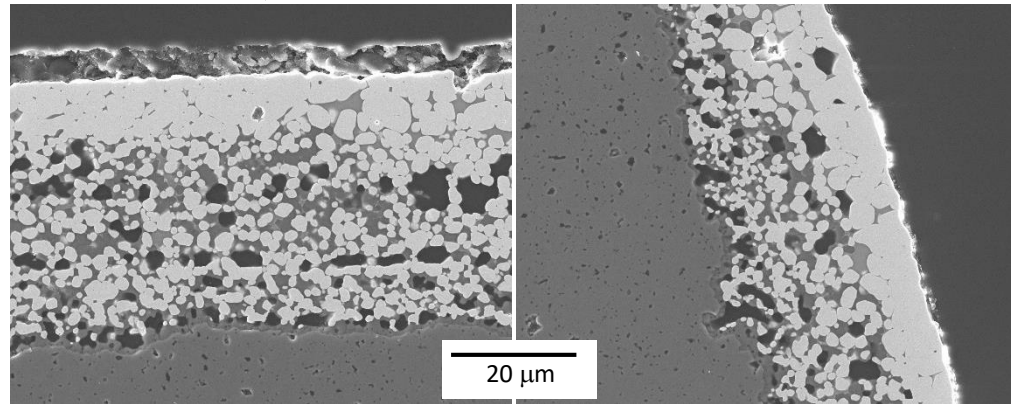
100h-100 cycles at 2700°F in Air



Leading Edge



Trailing Edge



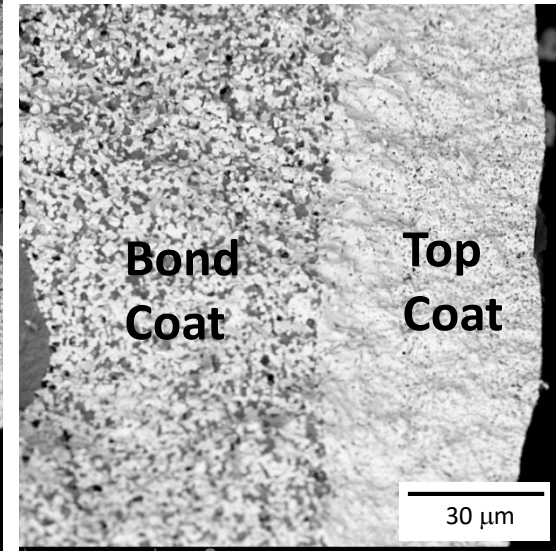
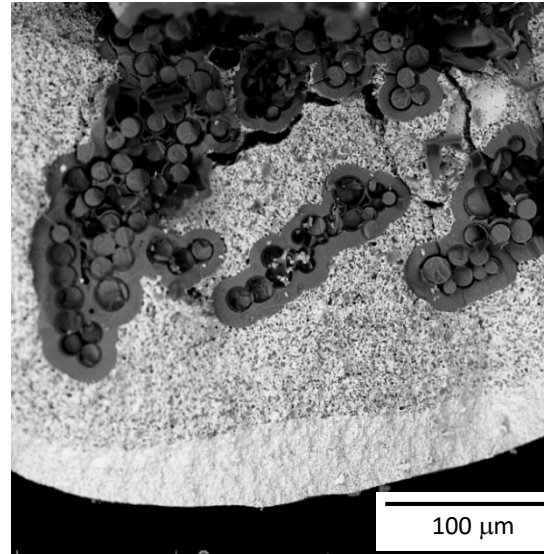
20 μ m

- Demonstrated 2700°F (1482C) temperature capability in air cycling
 - SiC substrate broke due to thermal shock

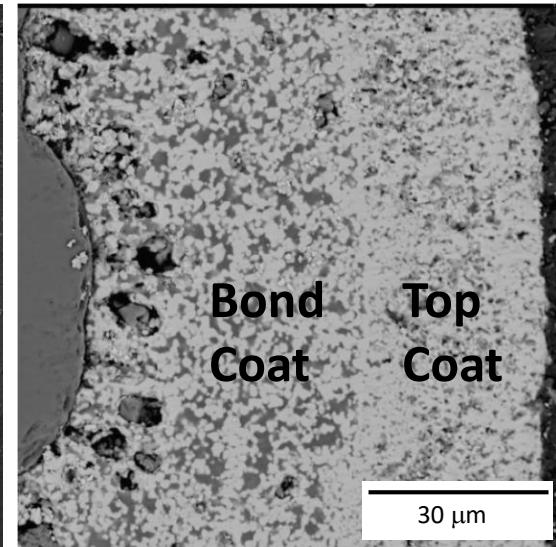
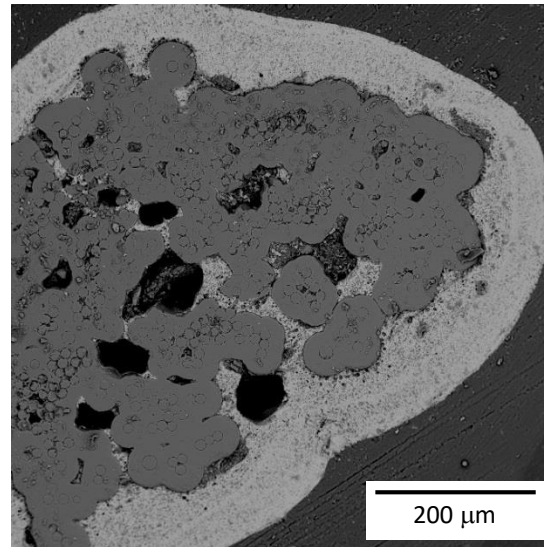
Slurry EBC Fabrication on Mini-composite (via Air Brush)



**As-Deposited and Dried
(Fractured Surface)**

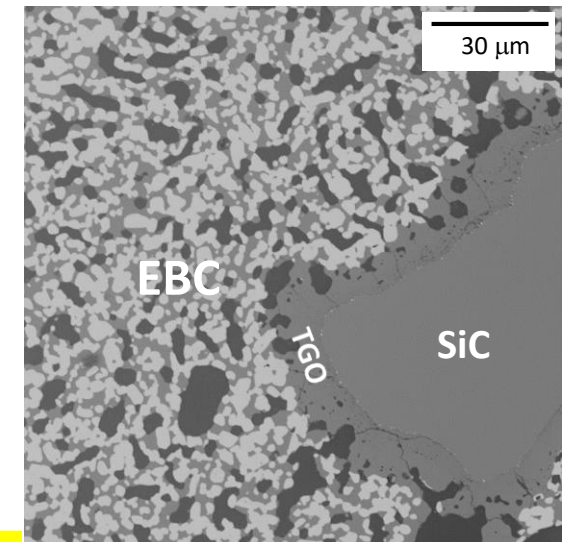
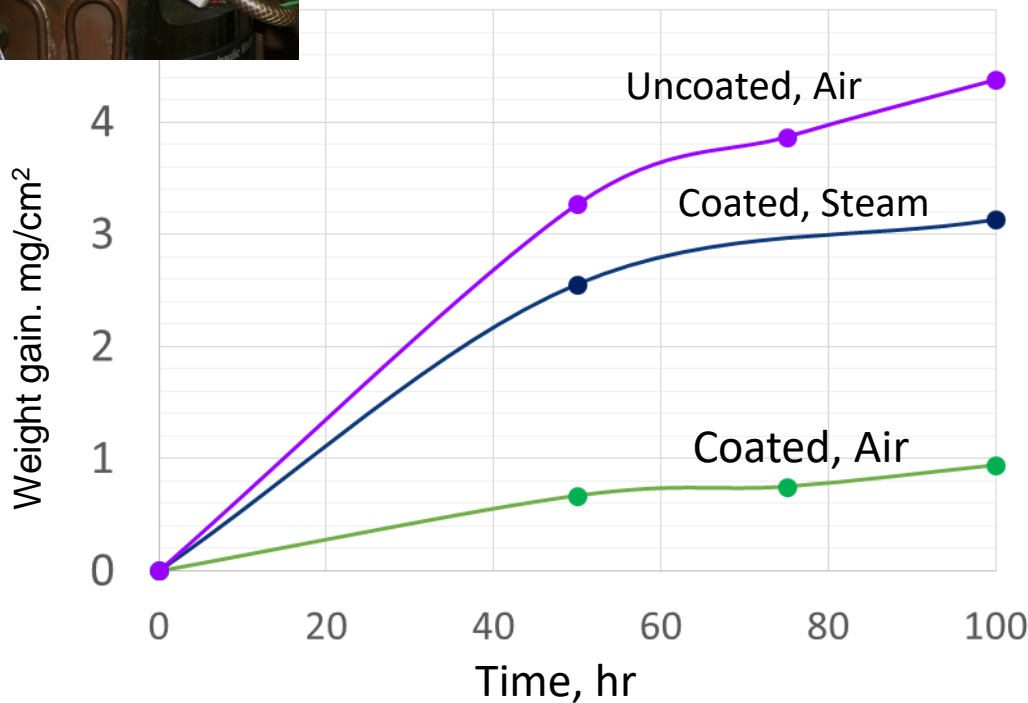
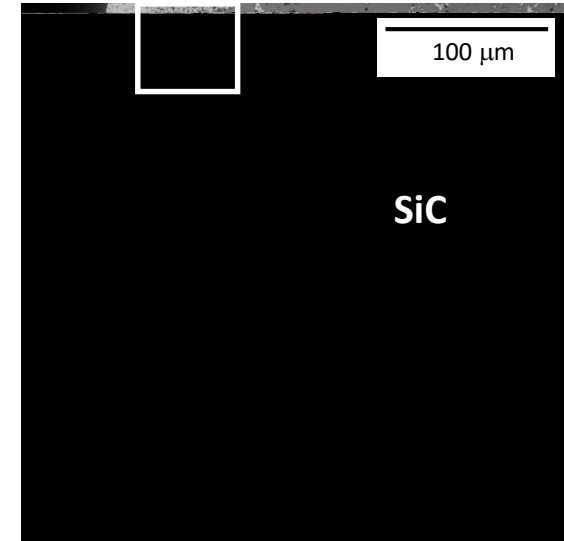


**As-Sintered
(Polished Surface)**



- Steam oxidation and mechanical testing in progress

Cyclic Oxidation of Slurry EBC on SiC Heating Element (1 hr at 2600°F (1427°C) / 20 min at T<100°C, 90% H₂O)



- High oxidation rate of uncoated SiC due to additives and high porosity
- EBC is effective in reducing oxidation rate in air and steam



Conclusion

- Two slurry-based EBCs capable of 2700°F have been developed
 - Low-cost, non-line-of-sight EBC technology
 - Coating can be readily applied on components of various complexities and shapes
- Coated coupons demonstrated 500h-500 cycle steam oxidation durability at 2600°F (1427C)
 - Parabolic oxidation rate is ~1/3 of the rate on uncoated internal CMC surface
- Coated SiC airfoil demonstrated 100h-100 cycle cyclic durability in air at 2700°F (1482C)
 - A spin-dip coating process developed for airfoils
- Coated CMC airfoil combustion rig test at 2500°F-2700°F (1371-1482C) in progress



Acknowledgement

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- **John Setlock for air brush spraying of slurry EBC on mini-composites (University of Toledo)**
- **Dagny Sacksteder for testing of EBC-coated SiC heating elements (Summer Intern, The Ohio State University)**