Thermal Spray Processing and Testing of Advanced Environmental Barrier Coatings

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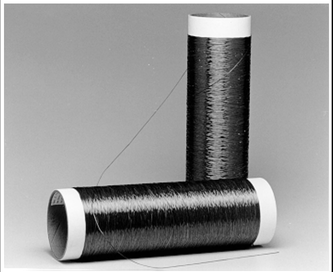
Introduction

Aircraft engine efficiency can be significantly improved with advanced materials

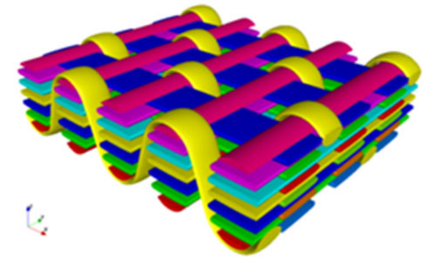
- Higher temperature materials require less cooling air
- Lower density materials can lead to lower weight of components
- Ceramic Matrix Composites (CMCs) offer a significant improvement
- Density ~ 2.8-3.2 g/cc
  - T_m > 2700°C
  - High stiffness
  - Acceptable fracture toughness, ductility

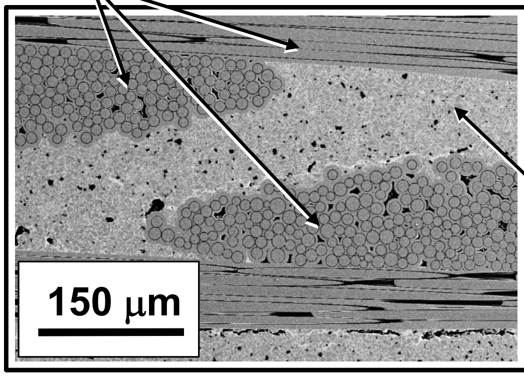
Began incorporation into turbine engines in 2016

SiC Fiber



2D or 3D Weave





Introduction

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- Lowe
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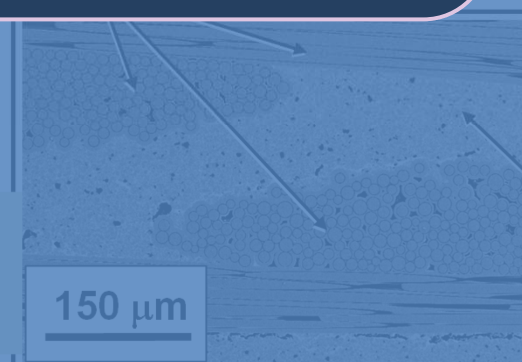
- Dens
  – T
  – High stiffness
  – Acceptable fracture toughness, ductility

SiC Fiber

2D or 3D Weave

NASA 2011 study:
A 56°C (100°F) increase in material capability in the 737 class equipment alone could provide **758 million gallons of fuel savings for the US market per year.**



Began incorporation into turbine engines since 2016

Degradation of Si-based Ceramics

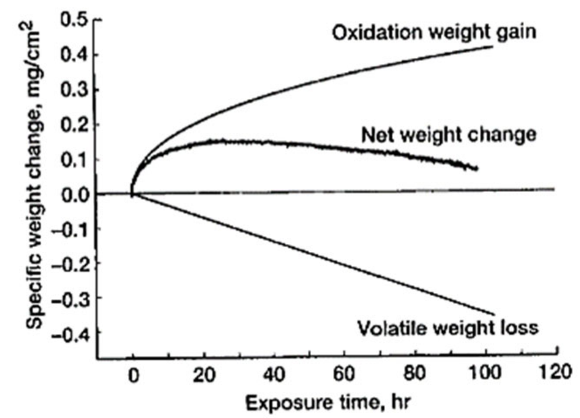
$$SiC + \frac{3}{2}O_{2(g)} = SiO_2 + CO_{(g)}$$ Oxidation

$$SiC + 3\,H_2O_{(g)} = SiO_2 + CO_{(g)} + 3H_{2(g)}$$ Oxidation

$$SiO_2 + 2H_2O_{(g)} = Si(OH)_{4(g)}$$ Recession

- H_2O increases oxidation rate and facilitates volatilization of the scale
- Temperature significantly increases mass loss

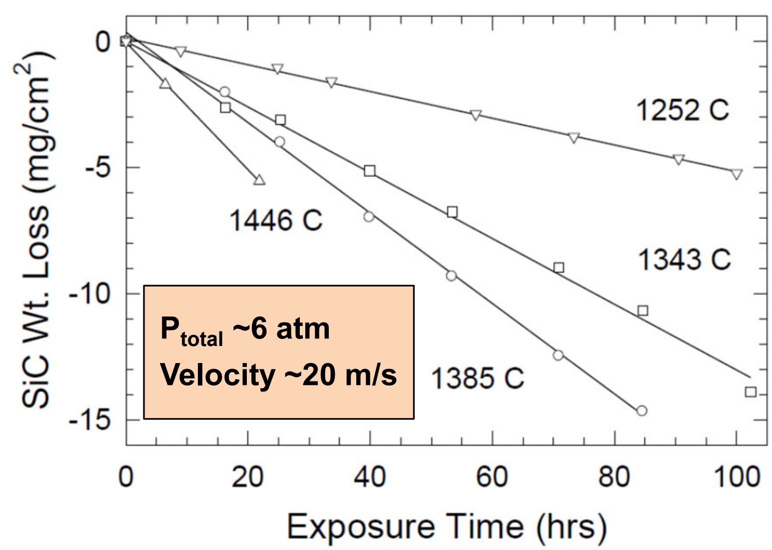
Paralinear weight change for CVD SiC
50% H_2O / 50% O_2 at 1200˚C



Smialek, et. al. (1999) NASA/TP – 1999-208696

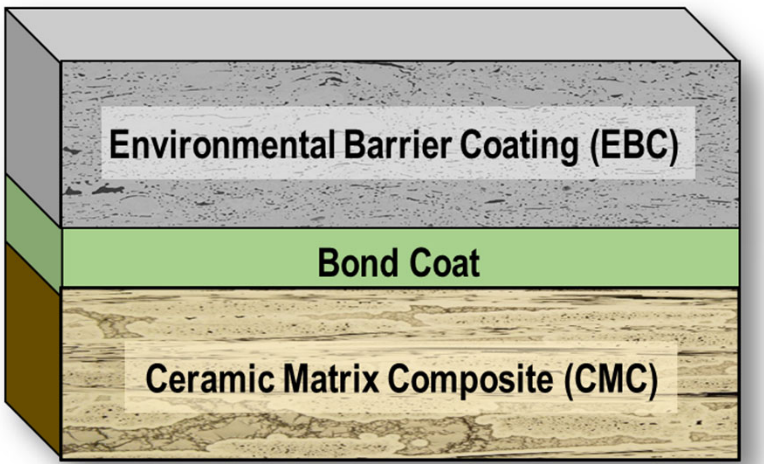
Turbine Engine Requirements

- Under relevant turbine conditions, 250 µm combustor liner consumed in 1000h at 1300°C!
- Subsonic turbine engines require ~300 hr at maximum temperature
- Current engines require materials to have durability >10,000 hrs
- Supersonic turbine engines require 6,000-9,000 hrs @ max temperature



Robinson and Smialek, J. Amer. Ceram. Soc., 82, 1817 (1999)

Environmental Barrier Coating (EBC) System



- Top coat "EBC layer"
  - Provides barrier from turbine environment
  - Can be multiple layers

- Bond coat
  - Provides bonding
  - Oxidation resistance

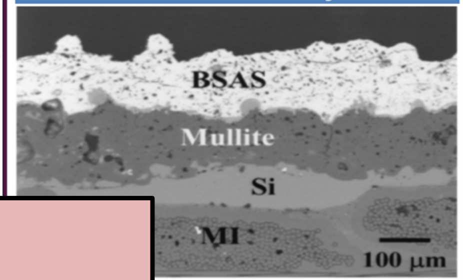
Intrinsic Material Selection Criteria

- **Coefficient of thermal expansion (CTE)**
- **Sintering resistance**
- **Low H_2O and O_2 diffusivity/solubility**
- **Phase Stability**
- **Low Modulus**
- **Limited coating interaction**

EBC Development History

- Gen I EBC[1] developed at NASA GRC in collaboration with GE and P&W
- BSAS/Mullite/Silicon multilayer
  - Low SiO_2 activity
  - Good CTE match
- Silicon (Si) bond coat added to improve adhesion and oxidation resistance
- Gen II EBC[2] developed at NASA GRC in early 2000s
  - Rare earth silicate-based system
  - RE_2SiO_5, $RE_2Si_2O_7$
  - RE = Y, Yb, Sc, Lu, etc.
  - Higher thermodynamic stability compared to Gen I EBC systems
  - Upper use temperature limited by Si bond coat (T_m ~ 1410°C)

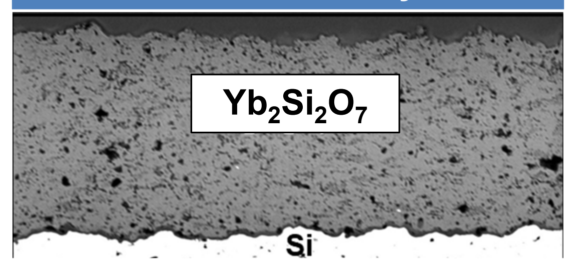
NASA Gen I EBC System

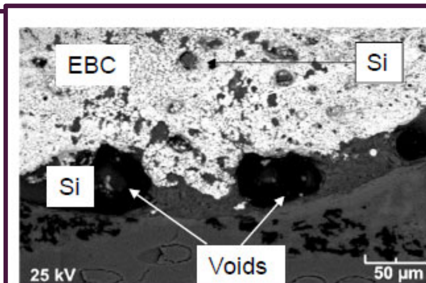


Mullite: ($3Al_2O_3{:}2SiO_2$)

BSAS: $1-xBaO \cdot xSrO \cdot Al_2O_3 \cdot 2SiO_2$, $0<x<1$

NASA Gen II EBC System





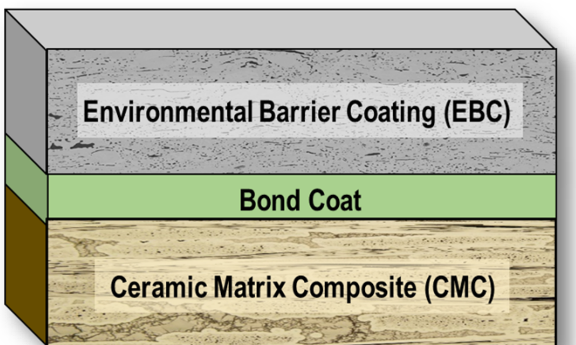
Si bond coat failure (1370°C)

[1]K. N. Lee et al., J. Am. Ceram. Soc. 86 [8] 1299-1306 (2003).
[2]K. N. Lee et al., J. Eur. Ceram. Soc. 25 1705-1715 (2005).

Potential Environmental Barrier Coating Materials

Top Coat (EBC)

- Prioritize water vapor resistance
- Can be multiple layers
- Coefficient of thermal expansion (CTE) can be limiting



Processing methods can influence mechanical and chemical properties

	Most Water Vapor Resistant
CTE too high ($>10\times10^{-6}$/°C)	HfO_2
	ZrO_2 (YSZ)
	$2(Y_2O_3) \bullet 3(ZrO_2)$
CTE too high or anisotropic	$3(Y_2O_3) \bullet 5(Al_2O_3)$ (YAG)
	$Yb_2O_3 \bullet SiO_2$
	$Y_2O_3 \bullet SiO_2$
	$Al_2O_3 \bullet TiO_2$
Currently used CTE ~4-6x10^{-6}/°C	$Yb_2O_3 \bullet 2(SiO_2)$
	$Y_2O_3 \bullet 2(SiO_2)$
	$Ba(Sr)O \bullet Al_2O_3 \bullet 2(SiO_2)$ (BSAS)
Not protective	Al_2O_3
	TiO_2
	SiO_2
	Cr_2O_3
	Least Water Vapor Resistant

SiC/SiC CMCs

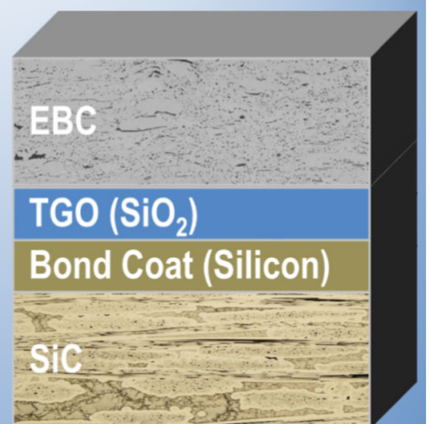
ASM Handbook, Volume 13B, Corrosion: Materials (#06508G), Environmental Performance of Nonmetallic Materials (574-575)

Low Temperature vs High Temperature Systems

EBC/CMC systems are usually designated as 'high temperature' or 'low temperature' depending on the presence of free silicon

"Low Temperature"

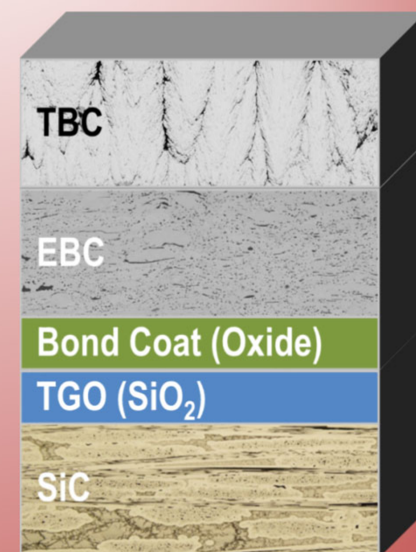
- Substrate contains free silicon
- Silicon bond coat
- **Currently flying today**
- Upper temperature limit ~1316ºC at bond coat
- Processed by thermal spray



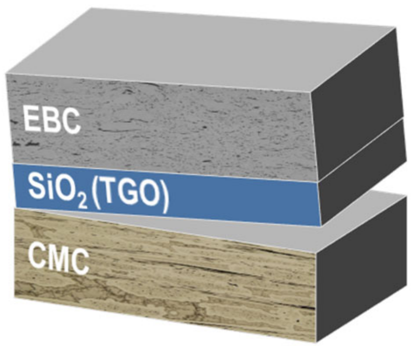
This system will be discussed today

"High Temperature"

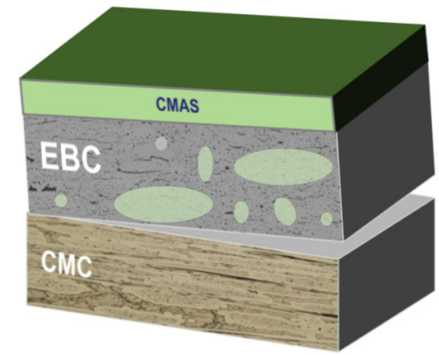
- Substrate has **<u>no</u>** free Si
- Silicon-free bond coat
- **Under development**
- Upper temperature limit >1482ºC
- Processed by slurry or thermal spray methods



Environmental Barrier Coating Failure Modes



Steam Oxidation

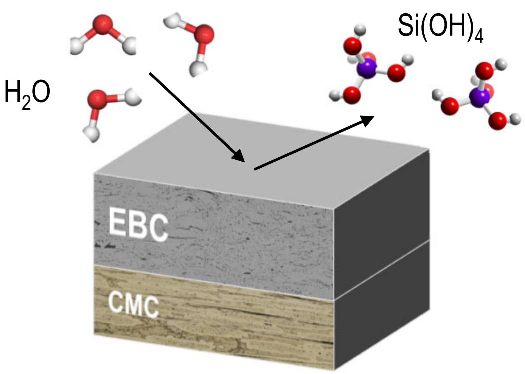


CMAS Attack & Infiltration

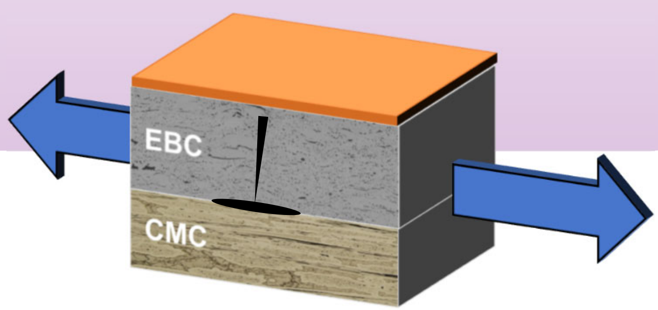
Testing of EBC systems is critical

Individual mechanisms must be well understood before evaluating combinatorial effects

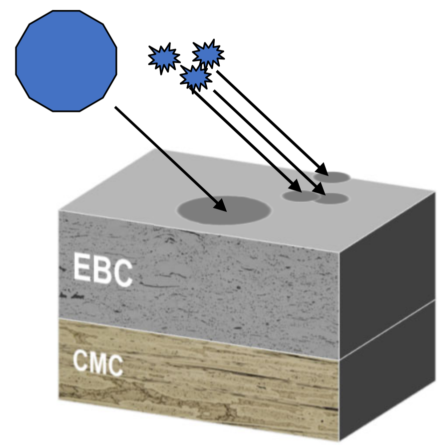
Synergies between extrinsic failure modes determine EBC lifetime and design requirements



Hydroxide Formation/Recession



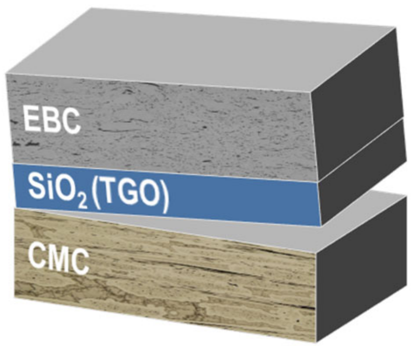
Thermomechanical Durability



Erosion and FOD

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

Environmental Barrier Coating Failure Modes



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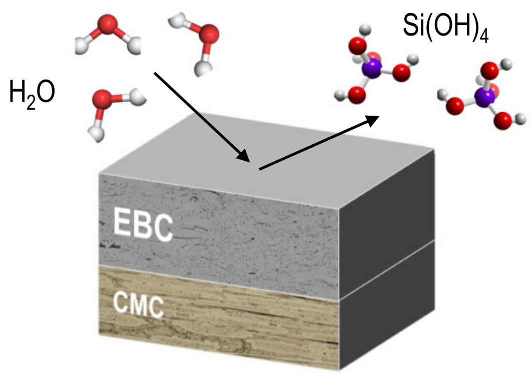

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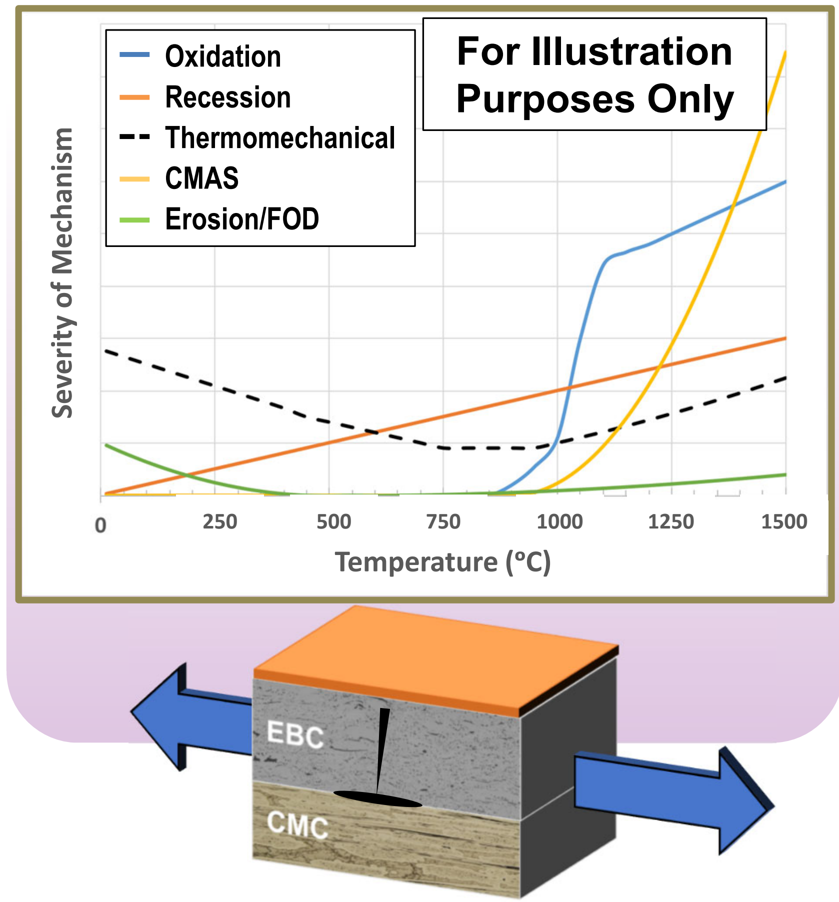
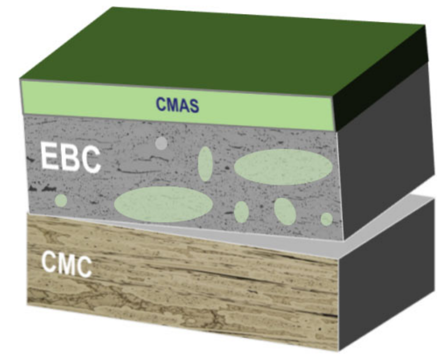


Thermomechanical Durability



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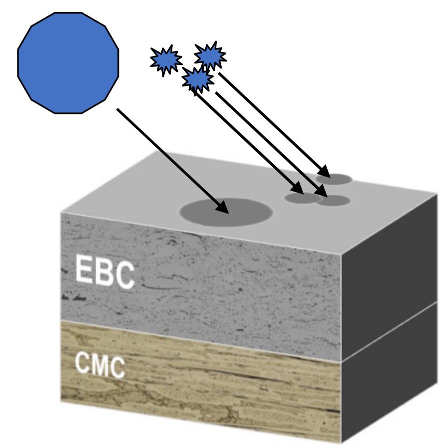


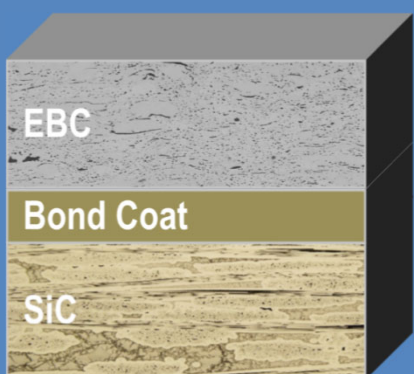
Erosion and FOD

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

Addressing EBC Design and Durability with Processing

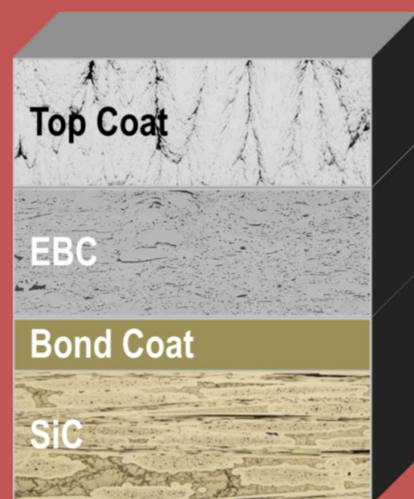
Microstructure

Can we change the porosity/form of the constituent material?



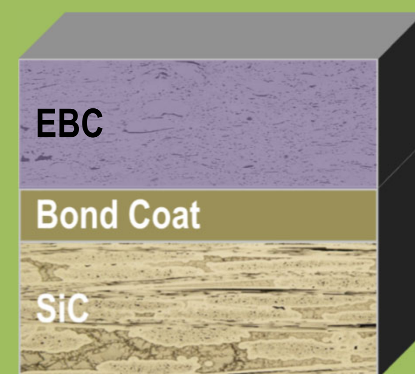
Architecture

Can we change the order of the layers or add additional layers?



Material Selection

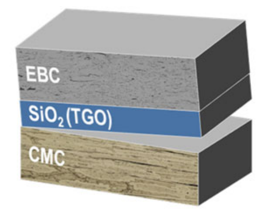
Can we utilize a different material to do the job?



Easiest → **Hardest**

Mechanism Outline

- Steam Oxidation



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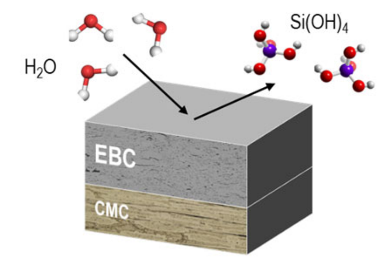
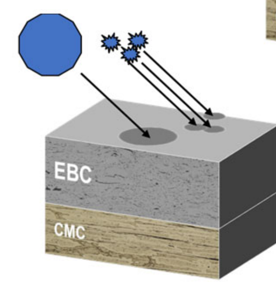
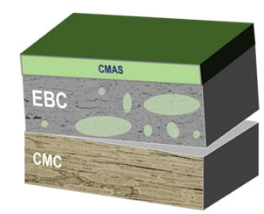


- Erosion/Foreign Object Damage (FOD)



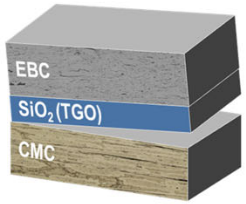
- Calcium-Magnesium-Aluminosilicate (CMAS)



- Combined Mechanisms

Mechanism Outline

- **Steam Oxidation**



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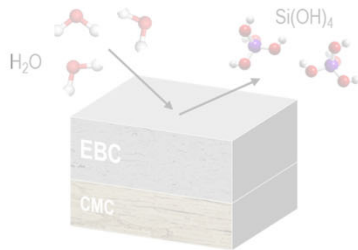

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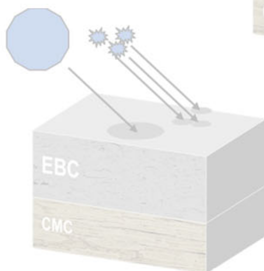

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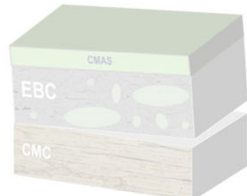
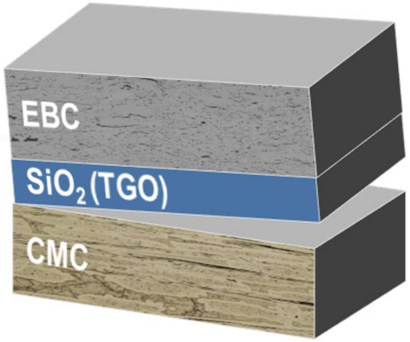
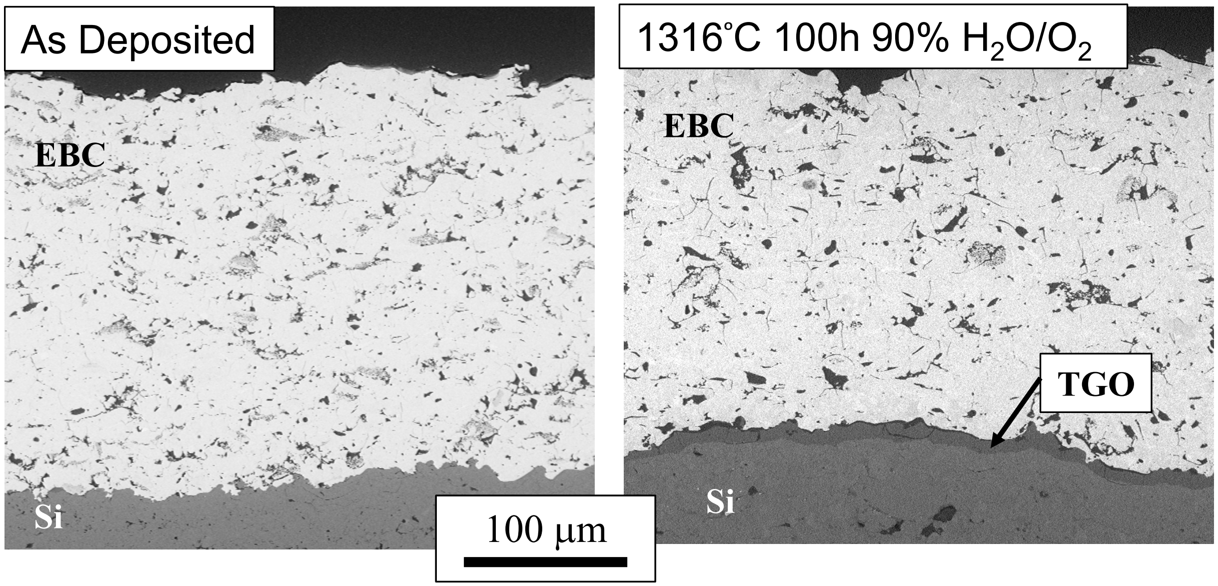


- Combined Mechanisms

Steam Oxidation (TGO Growth)

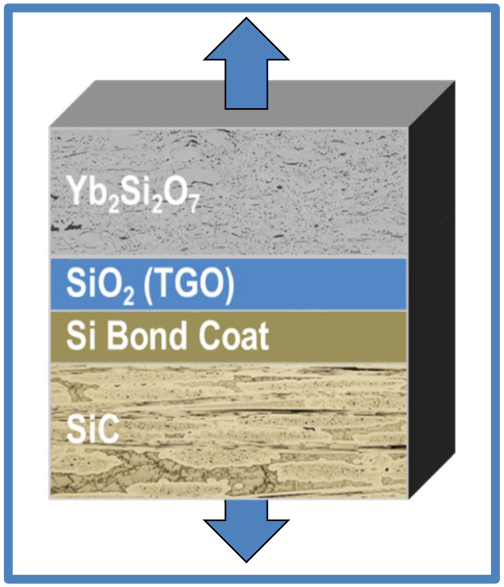


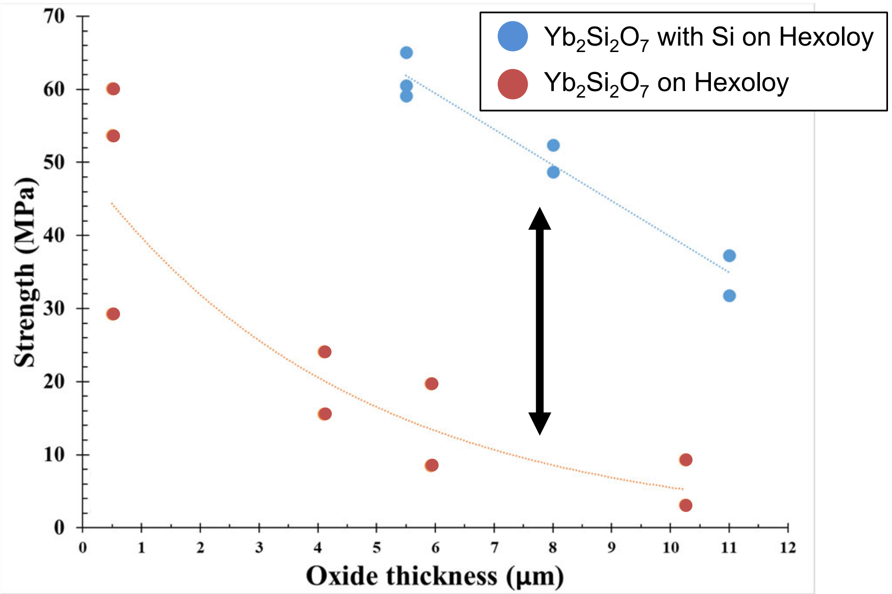
- Limiting the formation and growth of SiO_2 layer is critical to long-life and durability requirements
- Oxidation is an order of magnitude faster in water vapor than oxygen
- Thermally grown oxide (TGO) layer is the weak link in coating system due to CTE mismatch and phase transformation

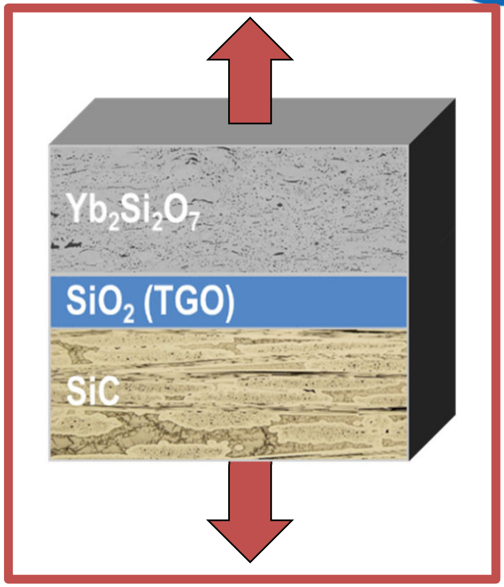


Observed limit for TGO thickness prior to spallation for APS $Yb_2Si_2O_7$ EBC is ~20µm

Steam Oxidation (TGO Growth)







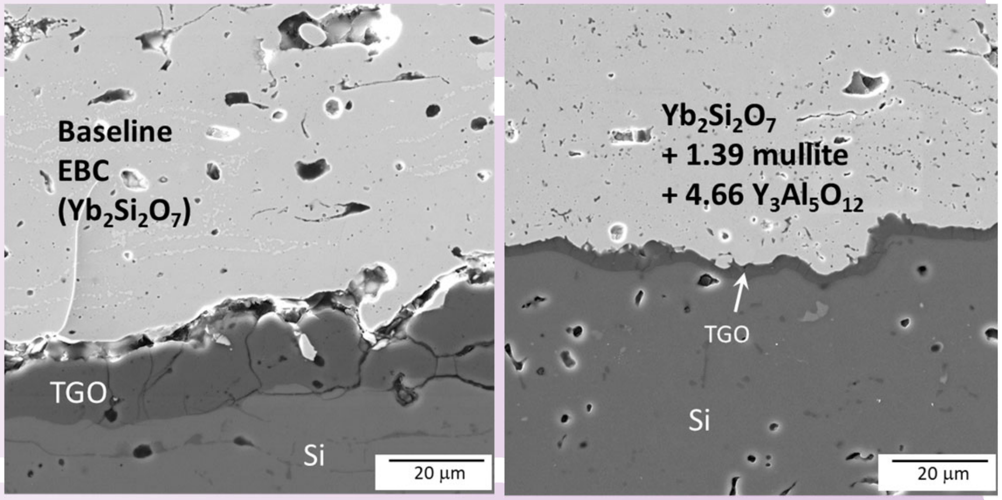
- Increasing thickness of thermally grown oxide (TGO) layer reduces the remaining strength in all EBC system architectures
- Presence of a Si bond coat increased residual strength by 30-40 MPa and systems without a Si bond coat are much more sensitive to TGO formation

Hoffman et. al. ASME 2023 SSDM. Harder et. al. (2020) J. Eng. Gas Turbine Power Mital et. al. (2021) NASA TM-20210014094

Low Temperature (Si-containing) EBC Systems

- Air plasma sprayed systems based on $Yb_2Si_2O_7$ EBCs on a Si bond coat
- Standard undoped $Yb_2Si_2O_7$ EBCs show life to ~1000hrs
  - Previously discussed 'Gen II' system
  - Limited by the formation of a thermally grown oxide (TGO)
- Modified $Yb_2Si_2O_7$ EBCs show TGO growth rates ~20x slower
  - Al_2O_3 presence showed the largest reduction in TGO growth rate

1000 hrs at 1316°C in 90% H_2O/O_2

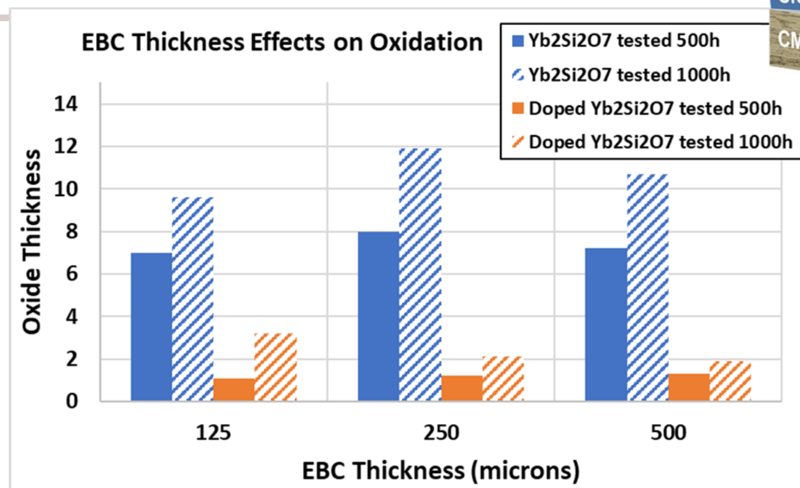


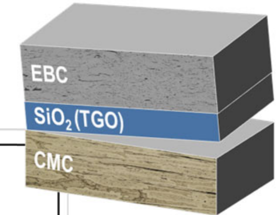
Material Selection Effects:
- Doping the base $Yb_2Si_2O_7$ EBC layer with Al_2O_3 additives provides significant improvements in oxidation performance

K. Lee et al., J. Amer. Ceram. Soc., 2019; 102:1507-1521.

Effects of Coating Thickness and Substrate on Oxidation

- EBC layer thickness was shown to have no effect on oxidation rate
  - Diffusion rates through EBC can increase by 1-2 orders of magnitude with 5-10% porosity
  - Oxidation rate controlled by TGO layer





Microstructure Effects:

- Coating thickness does **not** affect oxidant transport without reducing porosity below ~5%

K. Lee et al. J. Euro. Ceram. Soc 41 (2021) 5675-5685.
Navarre-Sitchler et al. J. Geo. Research 114 (2009) 1-14.

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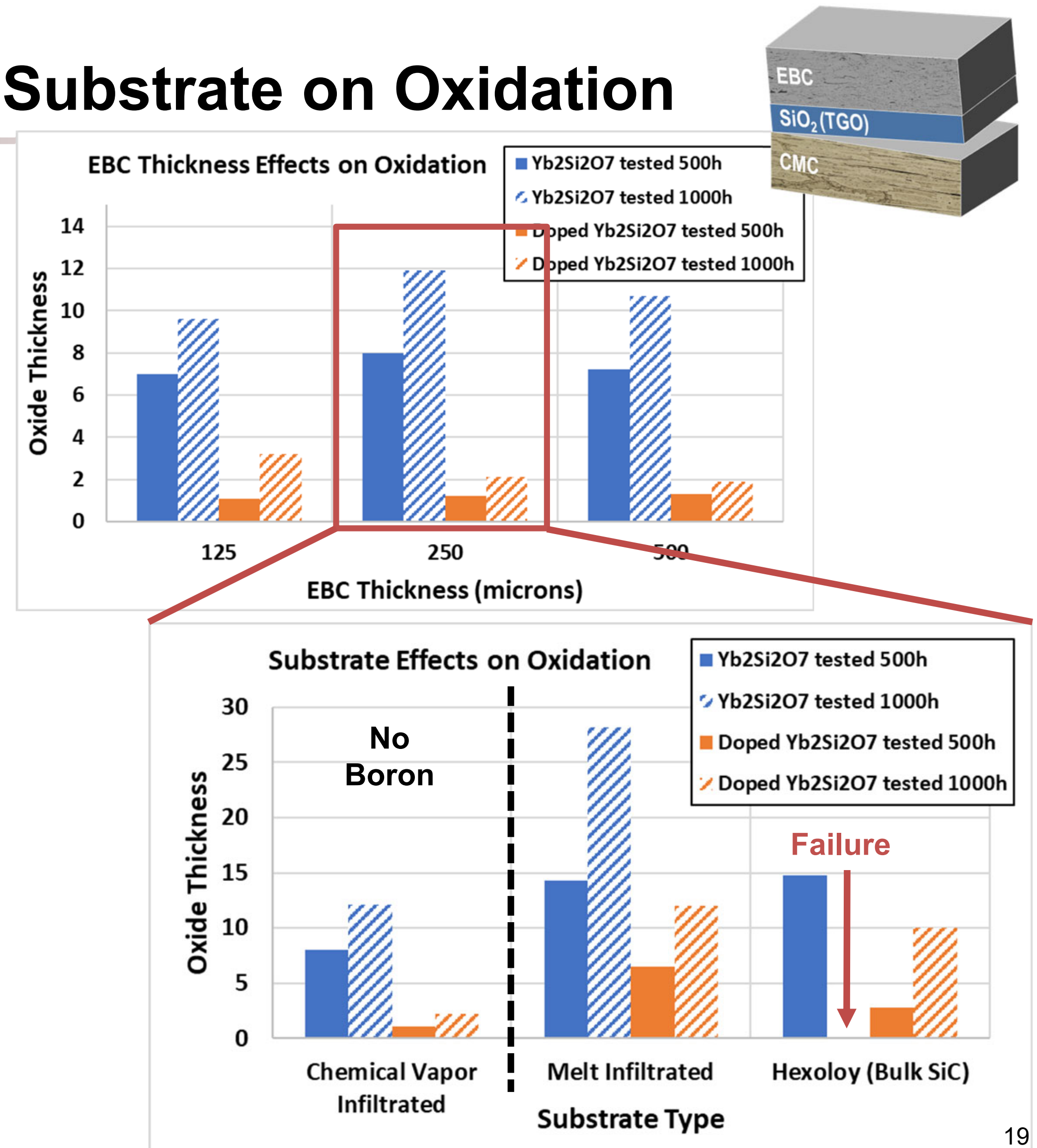
<u>Microstructure Effects:</u>
- Coating thickness does **<u>not</u>** affect oxidant transport without reducing porosity below ~5%

- Substrates containing boron had higher oxidation rates and failed earlier with identical EBC systems

<u>Architecture Effects:</u>
- Composition and processing of substrate can significantly influence oxidation rates and failure



K. Lee et al. J. Euro. Ceram. Soc 41 (2021) 5675-5685.
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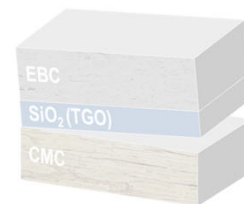
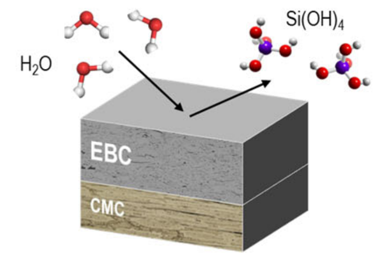
Mechanism Outline

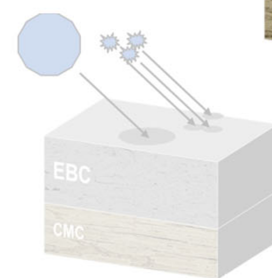
- Steam Oxidation



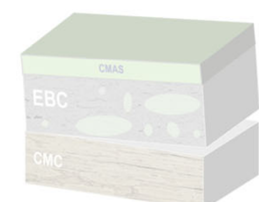
- **Hydroxide Formation/Recession**



- Erosion/Foreign Object Damage (FOD)

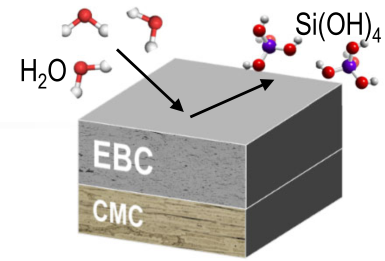


- Calcium-Magnesium-Aluminosilicate (CMAS)



- Combined Mechanisms

Hydroxide Formation / Volatility



Laminar Flow

$$J_{Si(OH)_4} = 0.664(Re)^{0.5}(Sc)^{0.33}\frac{D_{Si(OH)_4}}{RTL}Ka_{SiO_2}P_{H_2O}^2$$

- Re – Reynolds number
- Sc – Schmidt number
- $D_{Si(OH)_4}$ – volatile specie diffusion coefficient
- R – universal gas constant
- T – temperature
- $P(H_2O)$ – water vapor partial pressure

- a_{SiO_2} – activity of volatile specie

Costa, G. C., J. Euro. Ceram. Soc., 35 (2015) 4259-4267.

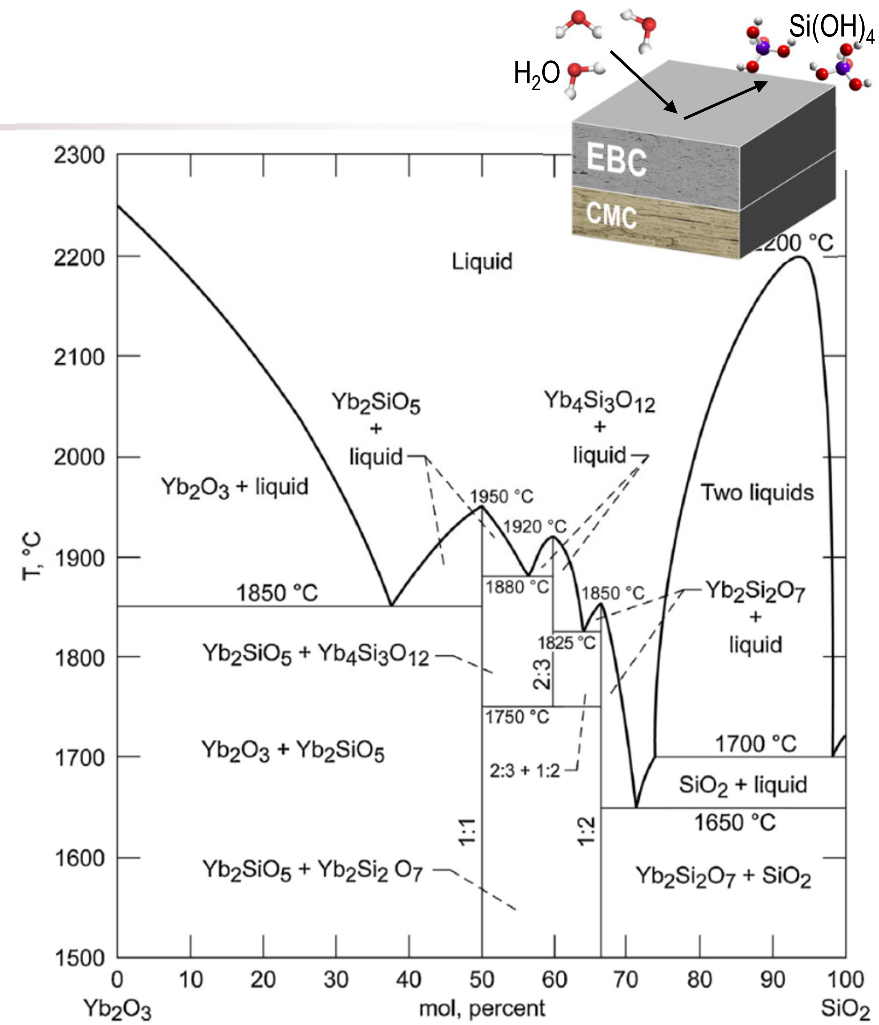
Hydroxide Formation / Volatility

Laminar Flow

$$J_{Si(OH)_4} = 0.664(Re)^{0.5}(Sc)^{0.33}\frac{D_{Si(OH)_4}}{RTL}K a_{SiO_2} P_{H_2O}^2$$

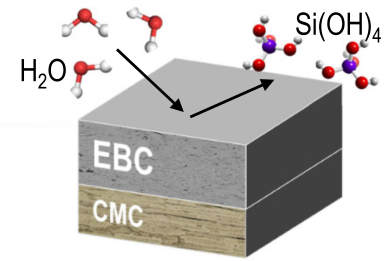
- Re – Reynolds number
- Sc – Schmidt number
- $D_{Si(OH)_4}$ – volatile specie diffusion coefficient
- R – universal gas constant
- T – temperature
- $P(H_2O)$ – water vapor partial pressure

- a_{SiO_2} – activity of volatile specie



Costa, G. C., J. Euro. Ceram. Soc., 35 (2015) 4259-4267.

Hydroxide Formation / Volatility



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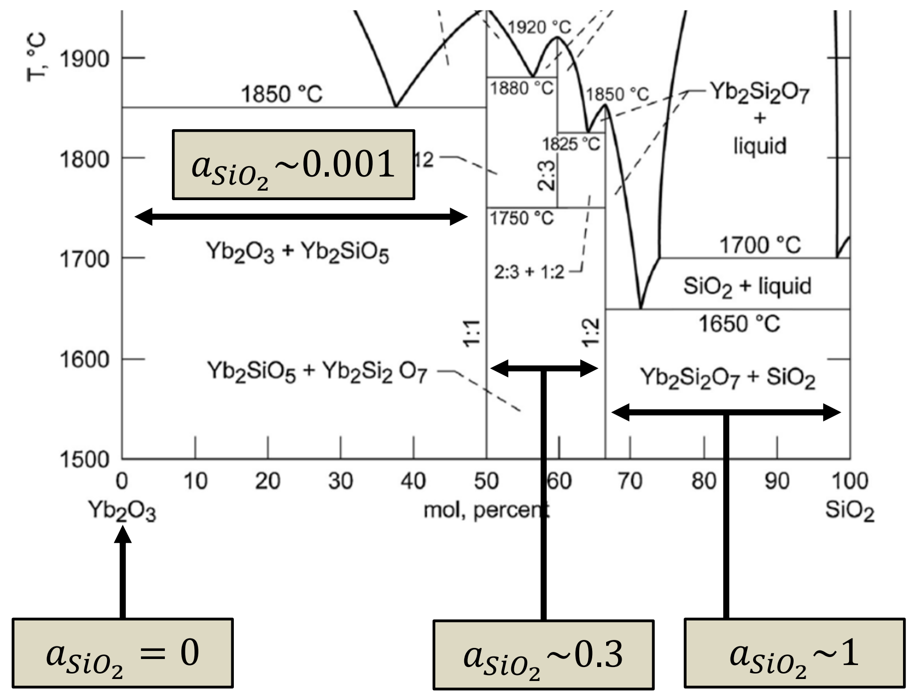


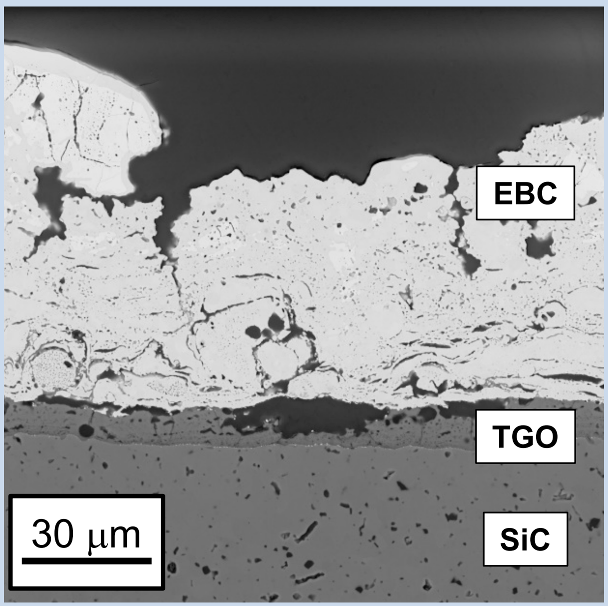
<u>Material Effects</u>
- EBC recession is driven primarily by the activity of the volatile species which makes material selection critical

Costa, G. C., J. Euro. Ceram. Soc., 35 (2015) 4259-4267.

$Yb_2Si_2O_7$ Coating Compositional Effects on Performance

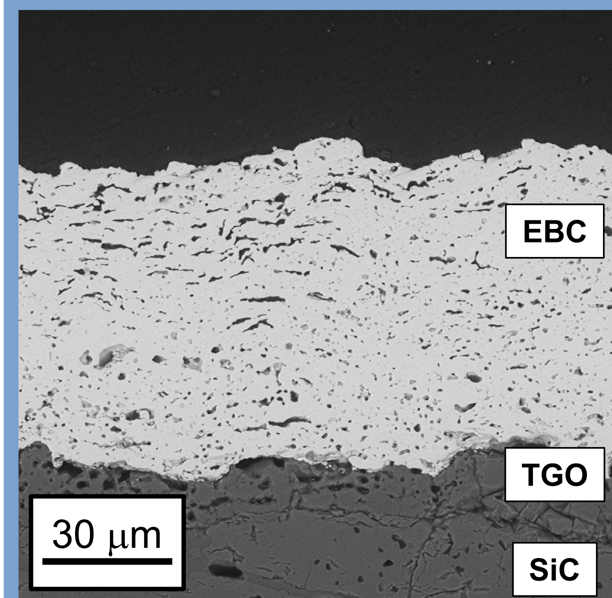
SiO_2-lean
$Yb_2Si_2O_7$ + Yb_2SiO_5

Anisotropic CTE causes tensile stresses which crack coating and allow for recession



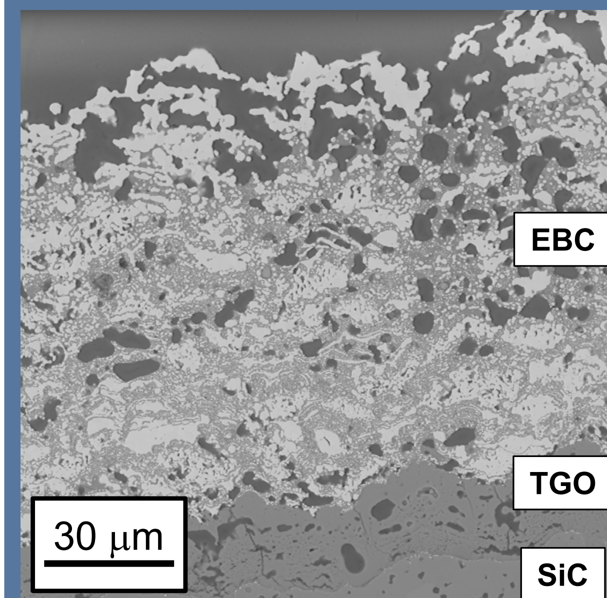
Stoichiometric
$Yb_2Si_2O_7$

Ideal CTE and phase stability in steam environments to handle thermal cycling



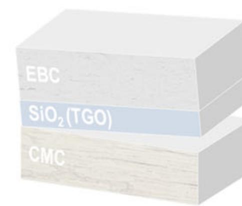
SiO_2-rich
$Yb_2Si_2O_7$ + SiO_2

Excess SiO_2 in coating volatizes to generate interconnected porosity

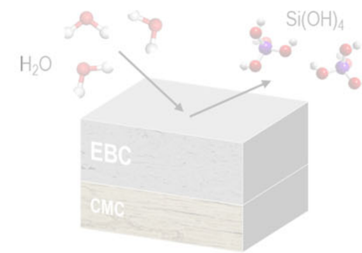


Mechanism Outline

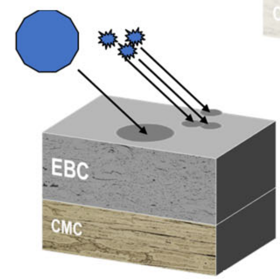
- Steam Oxidation



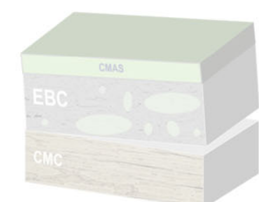
- Hydroxide Formation/Recession



- **Erosion/Foreign Object Damage (FOD)**



- Calcium-Magnesium-Aluminosilicate (CMAS)



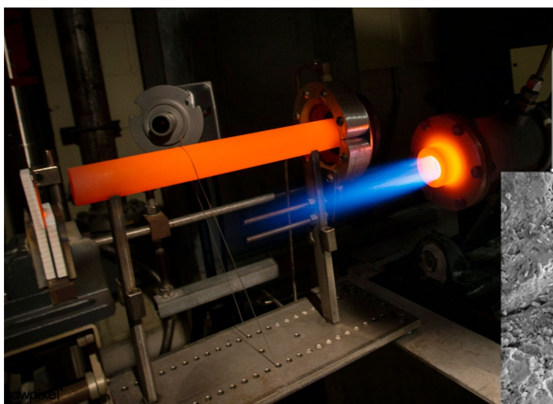
- Combined Mechanisms

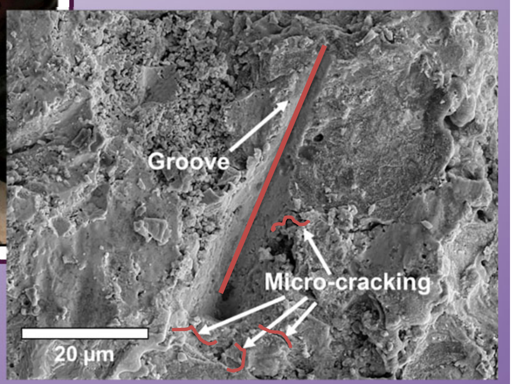
Erosion and Foreign Object Damage (FOD)

Solid Particle Erosion (SPE)

Material removal due to the repeated impact of solid particles

- Ground: Runway dust on taxiing and takeoff
- Altitude: Volcanoes, dust storms



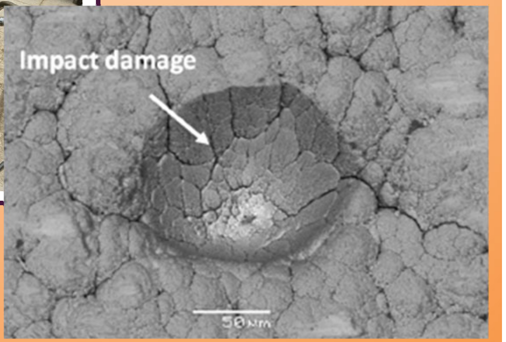


Presby, et. al. J. Amer. Ceram. Soc. - 2023
Presby, et. al. J. Eng. Gas Turbine Power – 2020

Foreign Object Damage (FOD)

Any object traveling into or downstream an engine that causes damage

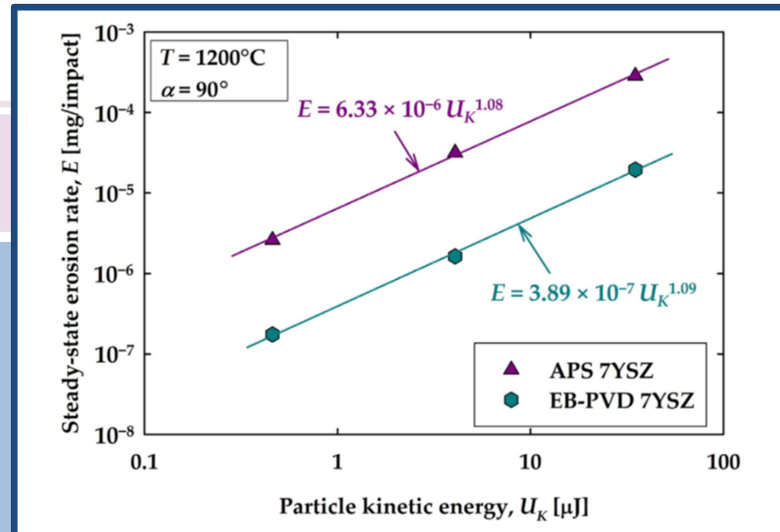
- External: Ice, pebbles, runway debris, etc
- Internal: Spalled coating, metal, nuts/bolts



S. Choi, (2014) ASME J. Eng. Gas Turbine Power

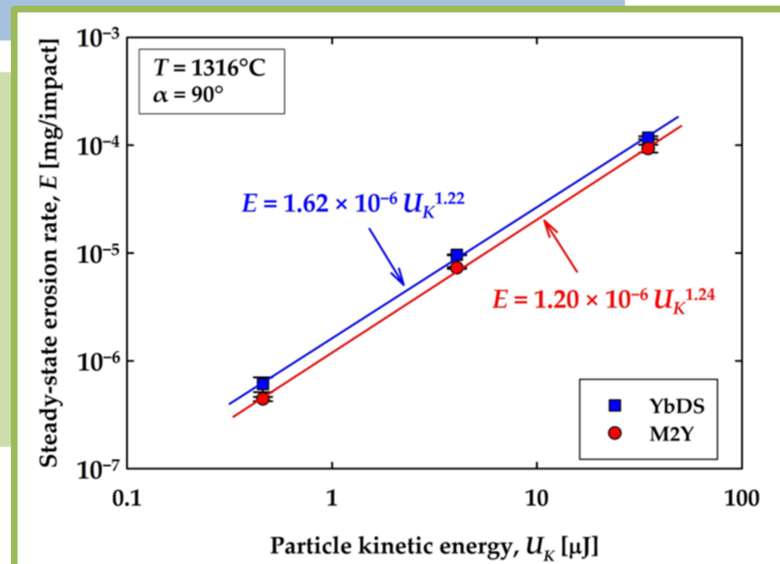
Solid Particle Erosion (SPE)

Limited amount of research on solid particle erosion of EBCs, although TBC erosion has been well-characterized



Microstructure Effects

- Coating morphology has been shown to significantly affect erosion rates
  - Columnar microstructure (EB-PVD) ~7-25 times better SPE performance over lamellar microstructure (APS)



Material Selection Effects

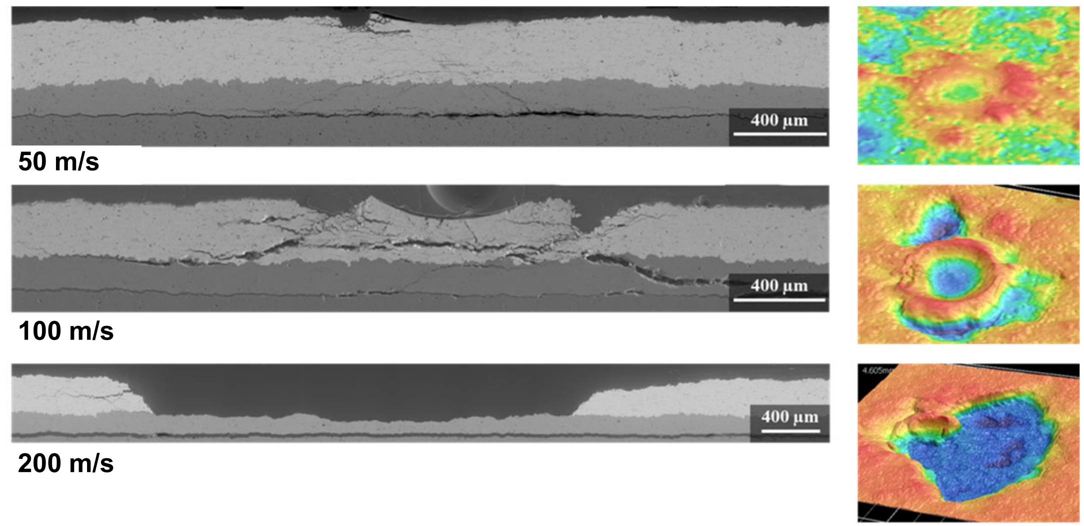
- Identically processed (lamellar) EBCs:
  - YbDS: $Yb_2Si_2O_7$
  - M2Y: $Yb_2Si_2O_7$ + 1.39 wt% mullite + 4.66 wt% $Y_3Al_5O_{12}$
- Additives improved SPE performance by 25% (1.25 times)

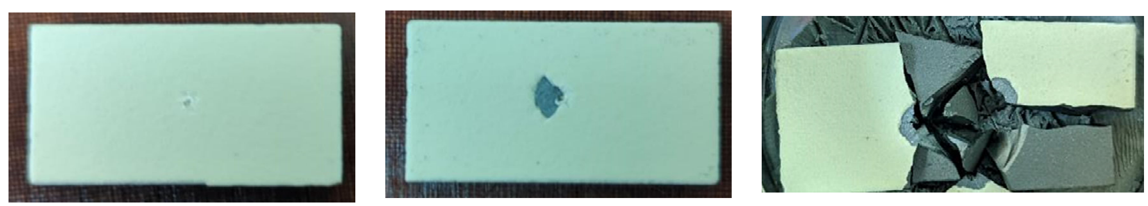
Shin, D., Hamed, A. (2018) Wear.

Presby, et. al. (2023) – Coatings

Foreign Object Damage of EBCs

- Limited amount of research in the literature on FOD of EBC systems
- Low velocity generates cone cracking and delamination on Hexoloy (α-SiC)
- High velocity impacts resulted in coating spallation or bulk SiC substrate fracture
- Crater depth and diameter increased with velocity with spallation at ~175-200 m/s





Hoffman, et. al. ASME 2023 SSDM.   S. Choi (2014) J. Eng. Gas Turb & Pow.

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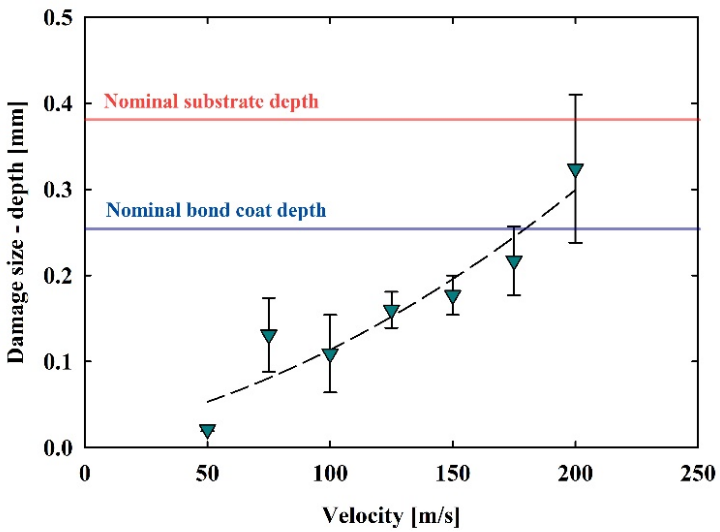
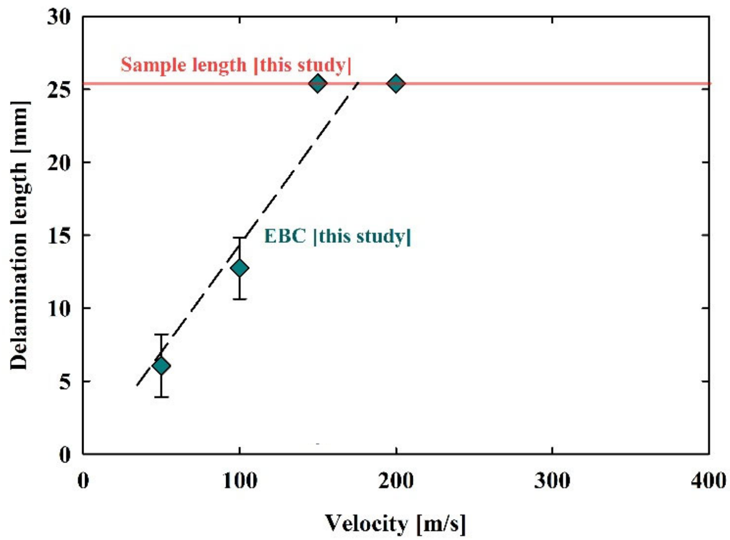


Architecture Effects

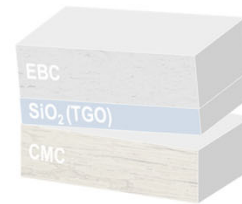
- Columnar YSZ could be utilized as an additional topcoat as it shows significantly better resistance to high velocity impact



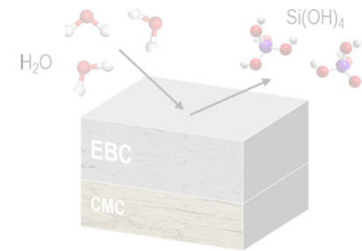
Hoffman, et. al. ASME 2023 SSDM.   S. Choi, (2014) J. Gas Turbo

Mechanism Outline

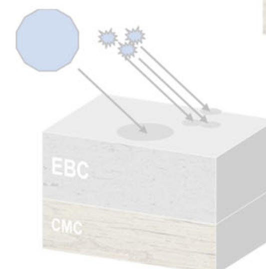
- Steam Oxidation



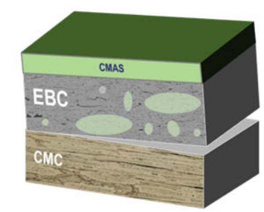
- Hydroxide Formation/Recession



- Erosion/Foreign Object Damage (FOD)

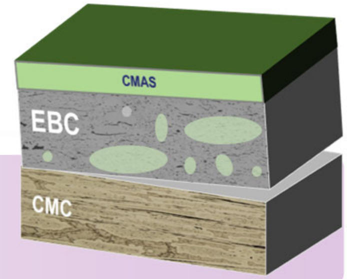


- **Calcium-Magnesium-Aluminosilicate (CMAS)**



- Combined Mechanisms

CMAS Attack and Infiltration



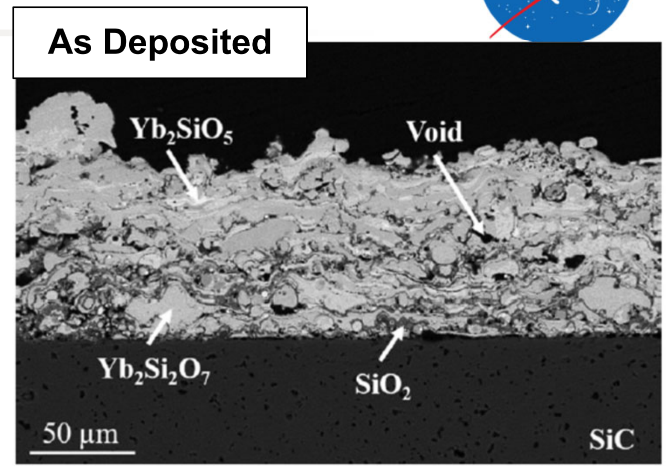
- Ingested particles melt into **C**alcium-**M**agnesium-**A**lumino **S**ilicate (**CMAS**) glass >1200ºC

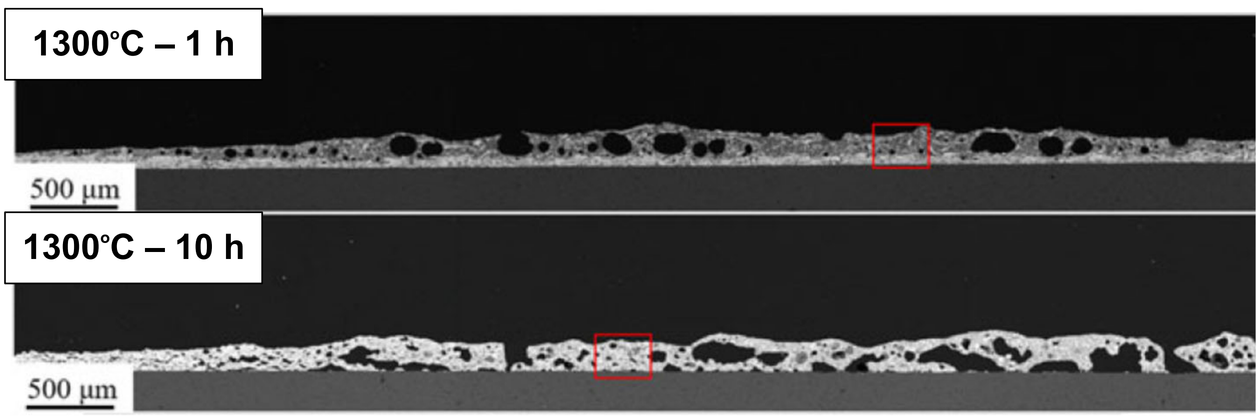
- Thermomechanical
  - Infiltration due to low viscosity and grain boundary transport
  - Dissimilar CTEs and densification produce (unwanted) stresses

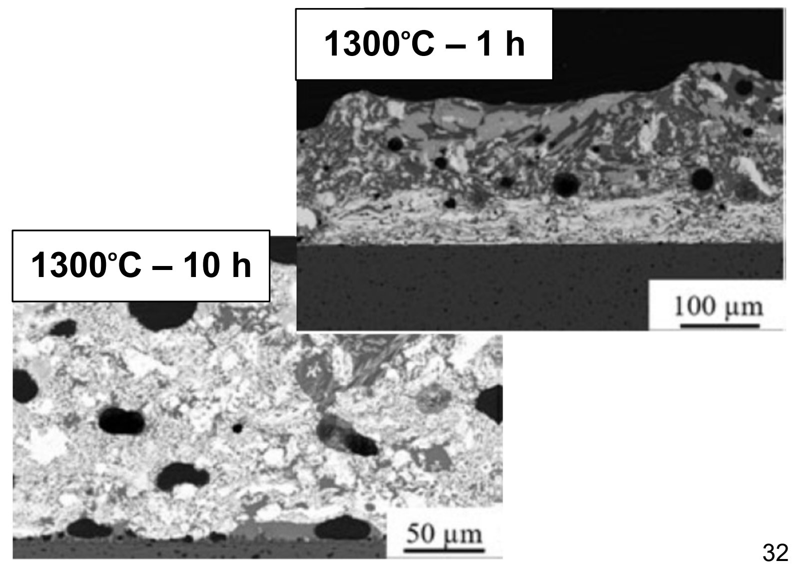
- Thermochemical
  - Interactions of CMAS glass with coating material to form new (unwanted) phases

CMAS Exposure of Plasma Sprayed $Yb_2Si_2O_7$

- CMAS attack is a serious challenge for any current or advanced EBC system
- Large quantities of CMAS exposure cause dissolution of rare earth silicates such as $Yb_2Si_2O_7$ and generate rapid pore formation and coating delamination
- Disilicate-CMAS reaction products contain SiO_2, resulting in no net consumption of SiO_2 in glass

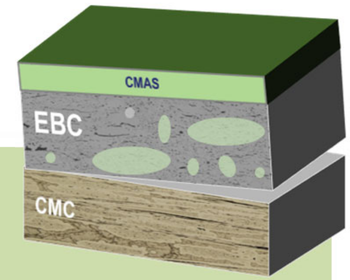






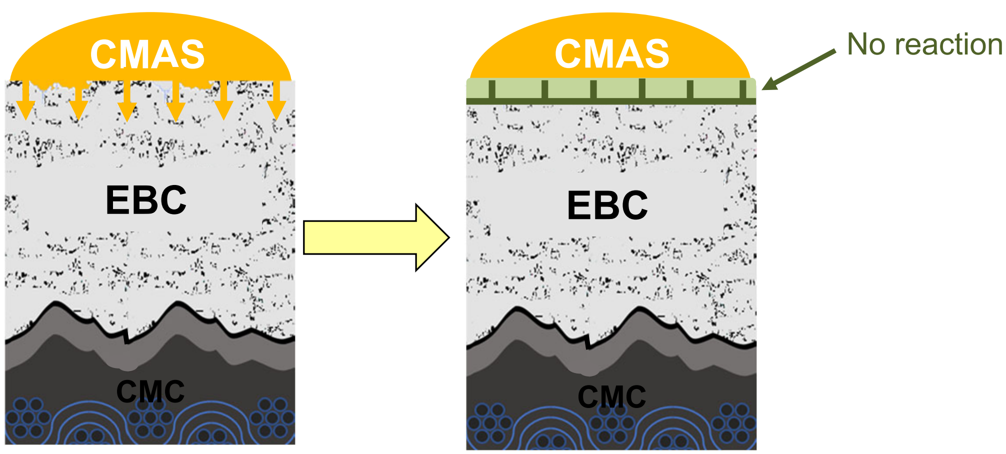
Wiesner, V. L., Jour. Mat. Res. - 2020

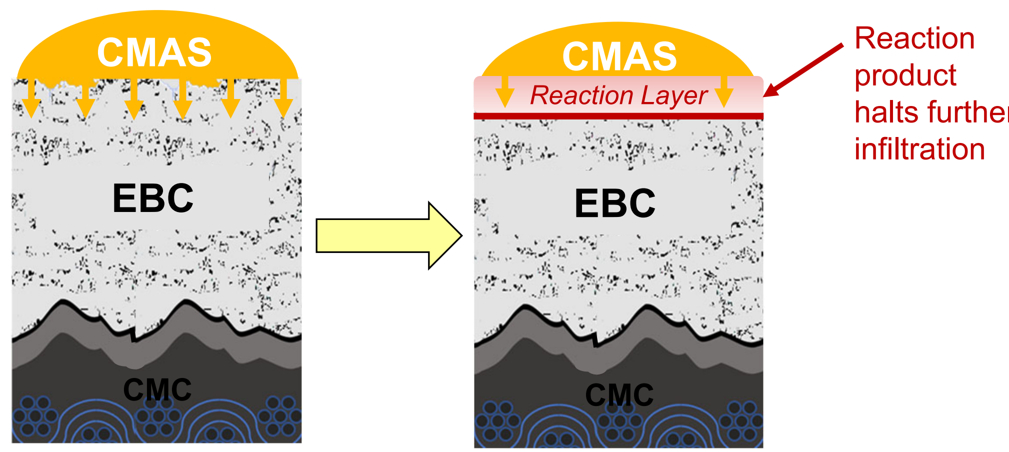
CMAS Mitigation Strategies for EBCs



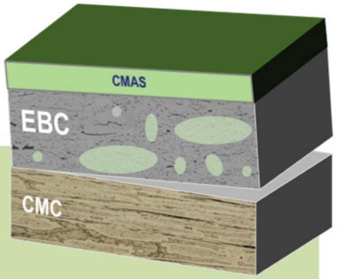
<u>Material Selection Effects:</u>

- *<u>Minimize</u>* reactivity of coating material with CMAS deposits
  - Thermodynamic stability over reaction products
- *<u>Maximize</u>* reactivity of coating material with CMAS deposits to induce crystallization
  - Crystallized reaction product barrier





CMAS Mitigation Strategies for EBCs

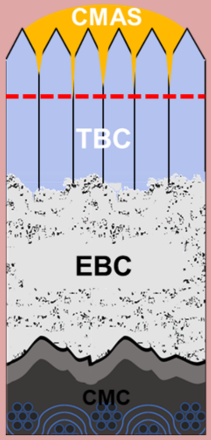


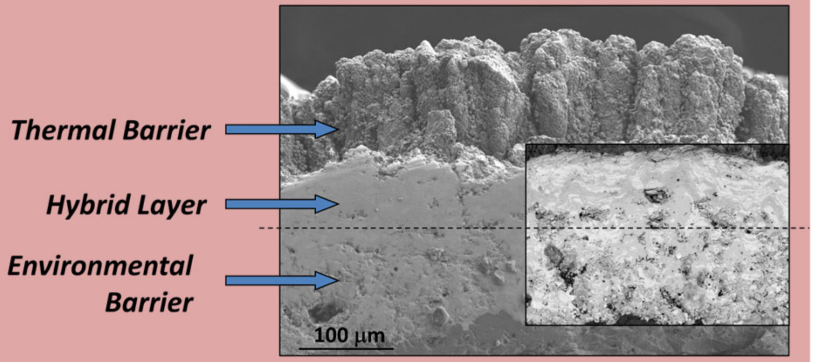
Material Selection Effects:

- *Minimize* reactivity of coating material with CMAS deposits
  - Thermodynamic stability over reaction products
- *Maximize* reactivity of coating material with CMAS deposits to induce crystallization
  - Crystallized reaction product barrier

Architecture Effects:

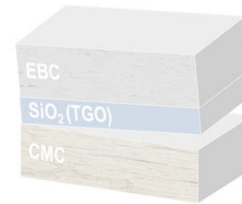
- Multi-layered /EBC architecture
  - Sacrificial topcoat
  - Larger thermal gradient to arrest glass penetration



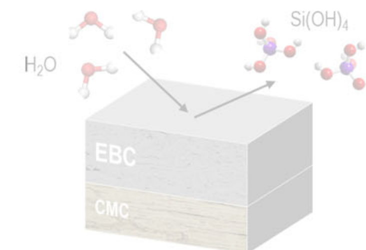


Mechanism Outline

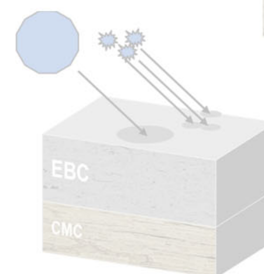
- Steam Oxidation



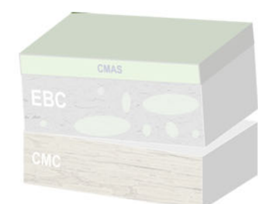
- Hydroxide Formation/Recession



- Erosion/Foreign Object Damage (FOD)

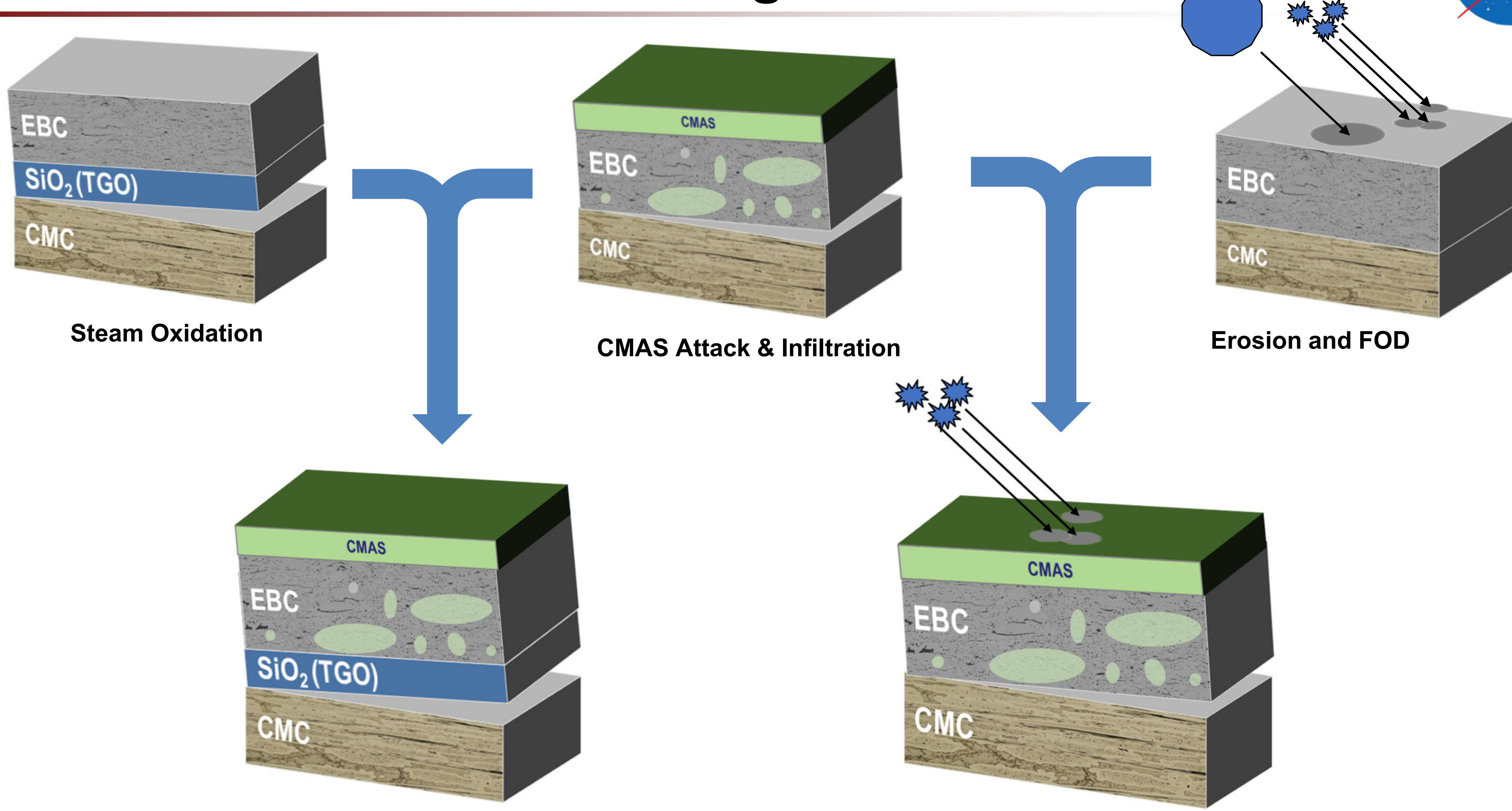


- Calcium-Magnesium-Aluminosilicate (CMAS)

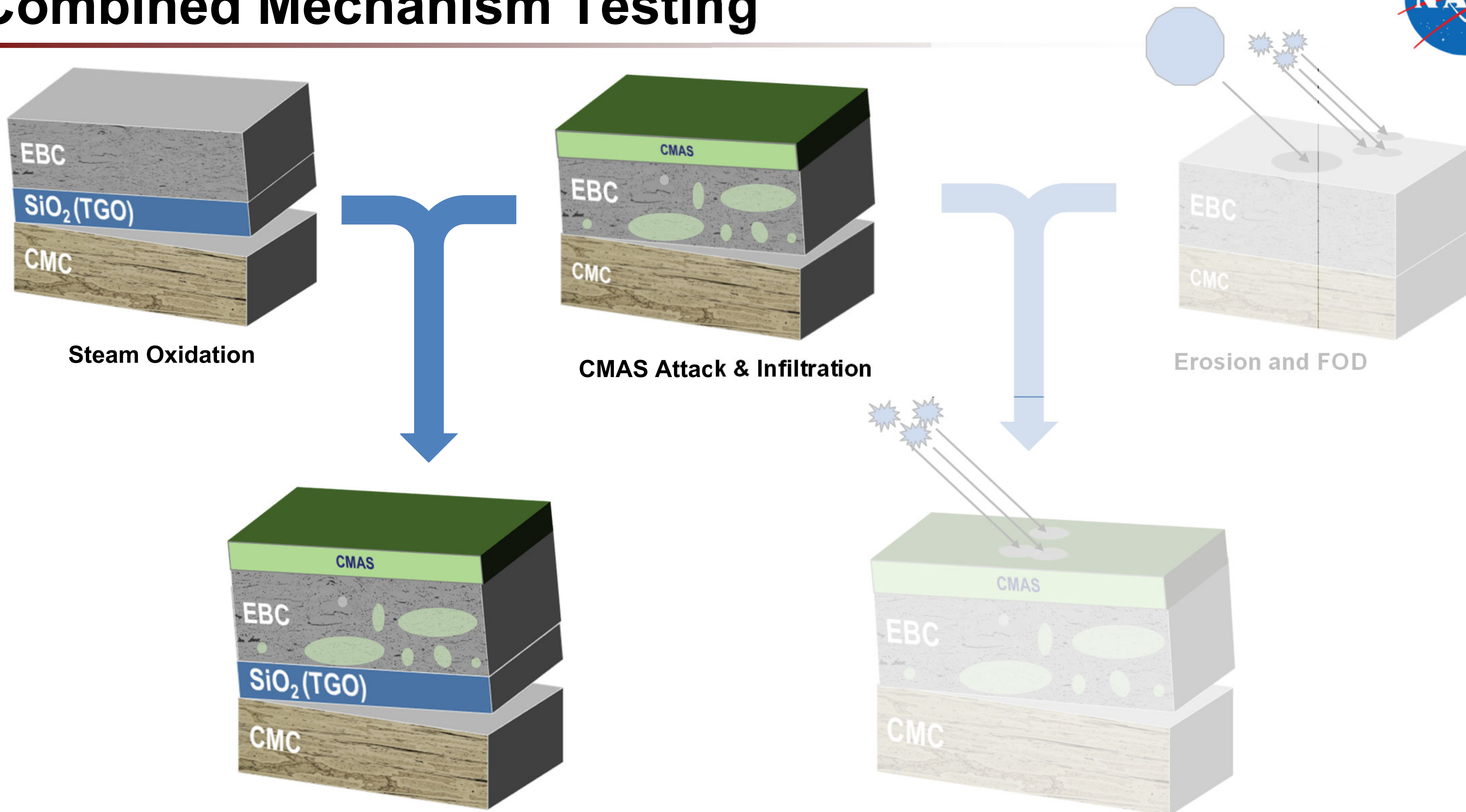


- **Combined Mechanisms**

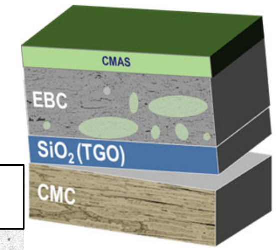
Combined Mechanism Testing



Combined Mechanism Testing



Steam Oxidation Durability of CMAS-exposed EBCs



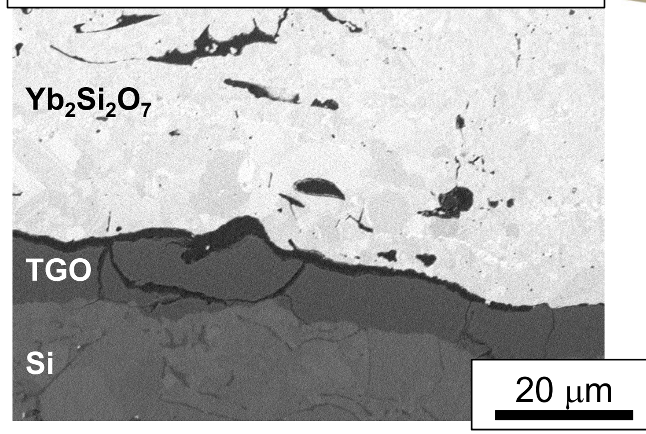
When low CMAS loading (2 mg/cm^2) is applied onto EBC systems <u>before</u> steam exposure:

- Glass penetration observed down to bond coat in 4h
- Significant grain growth occurred in $Yb_2Si_2O_7$
- <u>Apparent oxidation rate was reduced by factor of ~2</u>
  - Possibly due to SiO_2 consumption or transport effects

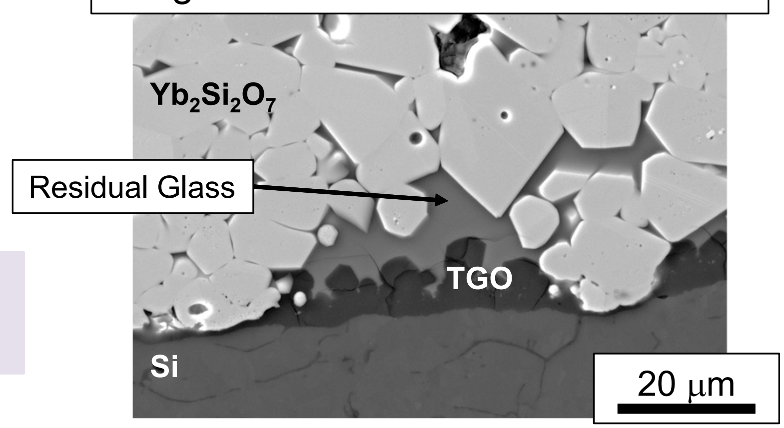
1316C 90% H_2O/O_2	No CMAS	2 mg/cm^2 CMAS
100h	6.8 μm	4.9 μm
200h	9.9 μm	7.9 μm
300h	12.5 μm	8.5 μm
Apparent kp (μm^2/hr)	**0.52**	**0.25**

<u>CMAS composition (mol%):</u>
48% SiO_2, 31% CaO, 13% Al_2O_3, 8% MgO

No CMAS, 100h steam 1316C

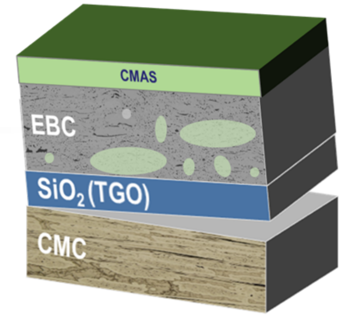


2mg/cm^2 CMAS 100h steam 1316C



Harder, B. J., In Review, J. Euro. Ceram. Soc. (2023)

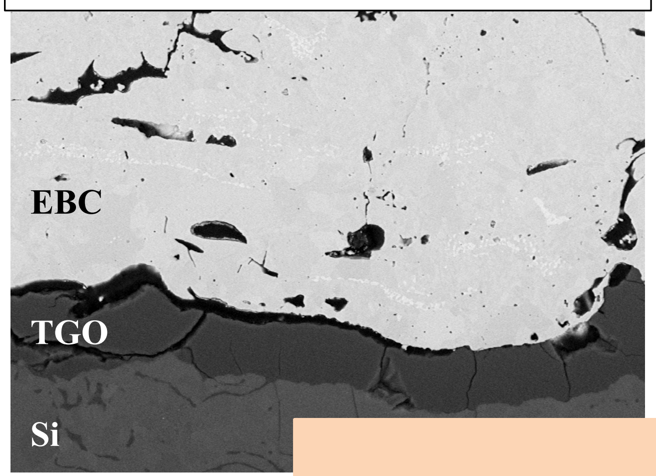
Steam Oxidation Durability of CMAS-exposed EBCs



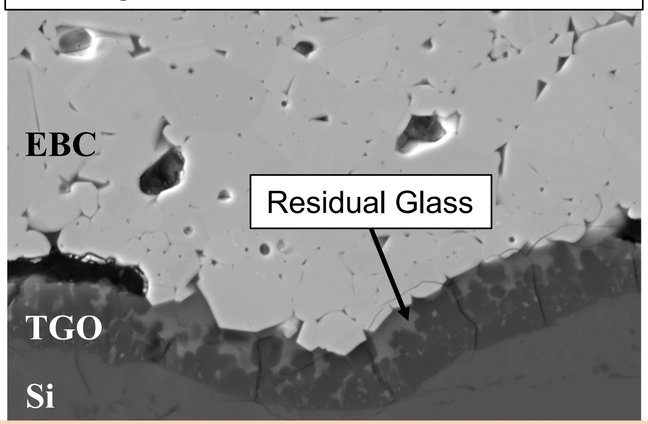
When low CMAS loading (2 mg/cm^2) is applied onto EBC systems <u>after</u> 100h of preliminary steam exposure:

- When TGO was present before CMAS, the TGO was consumed, leaving behind significant porosity and a <u>much weaker EBC/TGO interface</u>

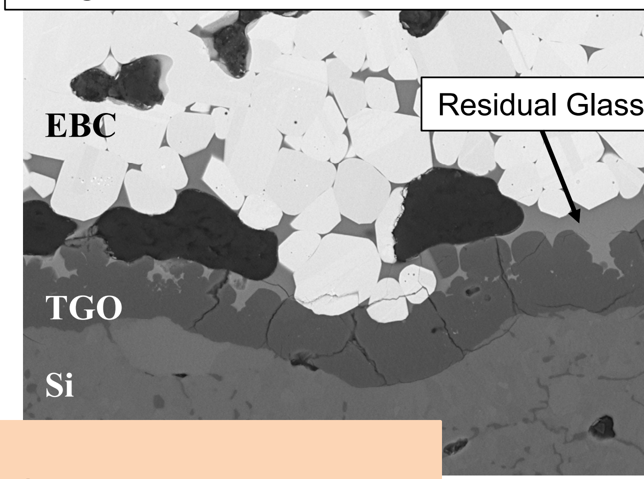
No CMAS, 100h steam



2mg/cm^2 CMAS, 4h 1316C Air



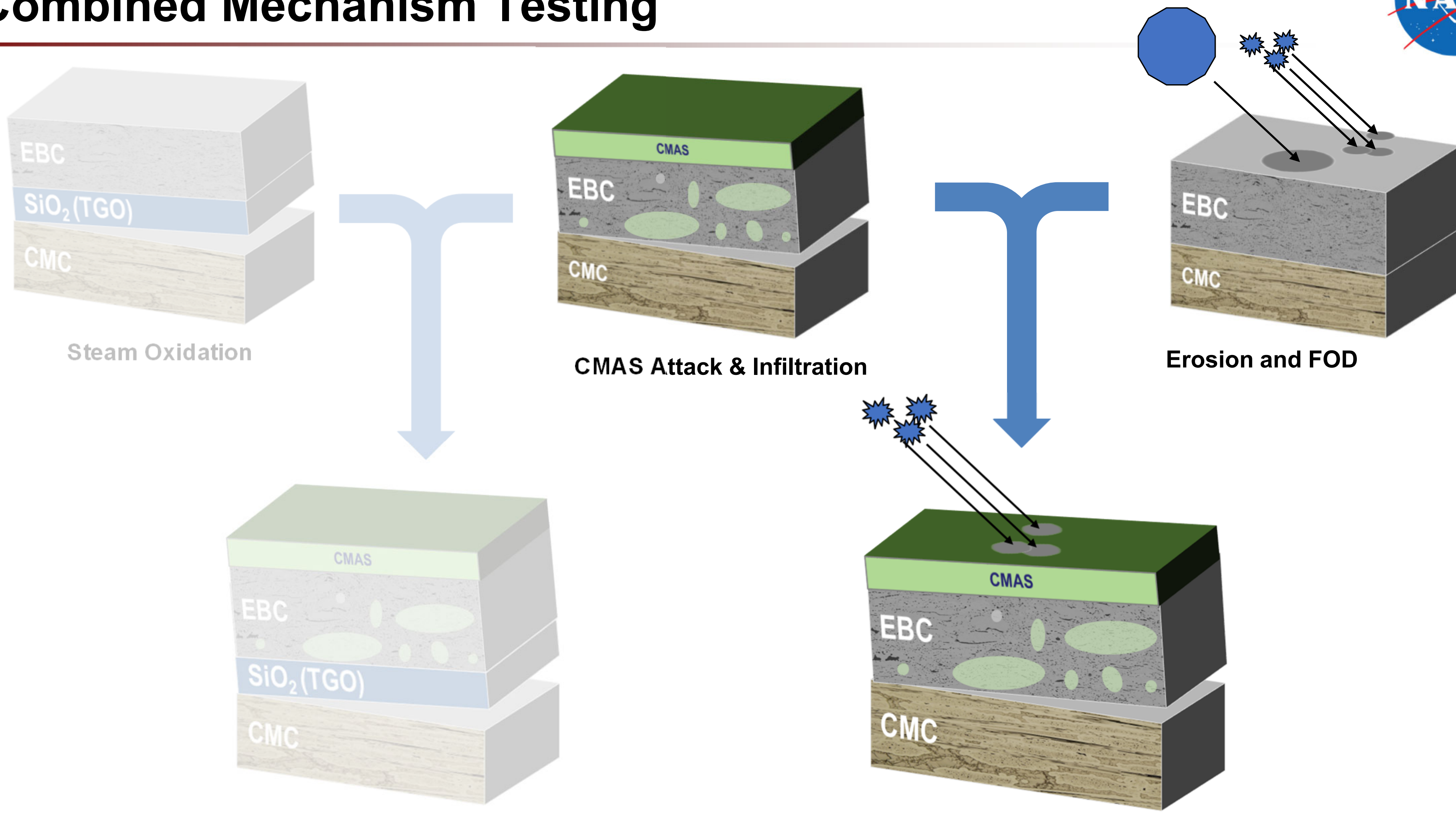
2mg/cm^2 CMAS, 100h 1316C steam



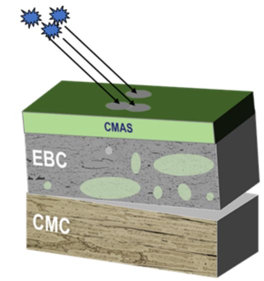
Much more common scenario for CMAS interaction!

Harder, B. J., In Review, J. Euro. Ceram. Soc. (2023)

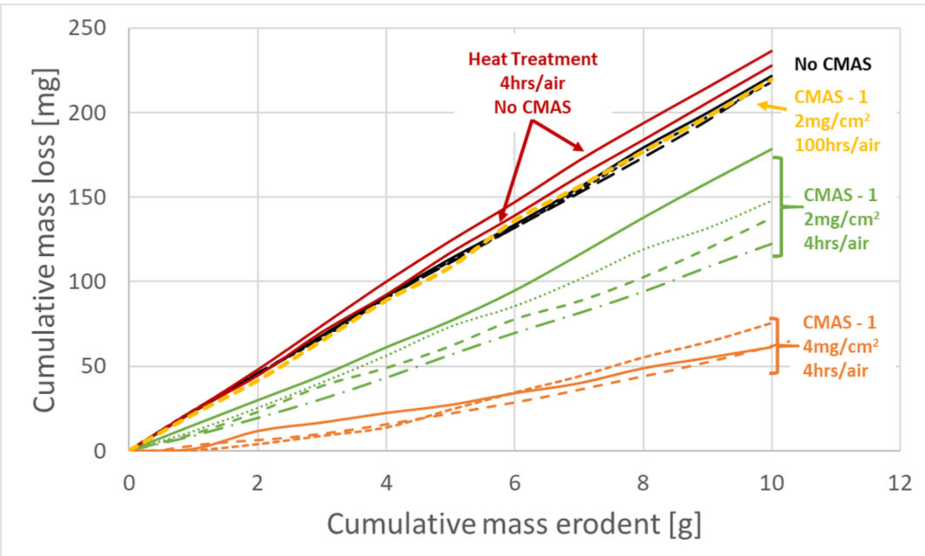
Combined Mechanism Testing



Solid Particle Erosion of CMAS-exposed EBCs



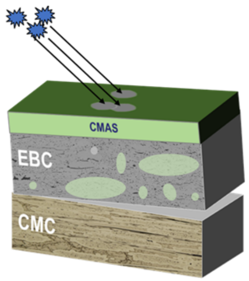
When low CMAS loading (2 mg/cm²) is applied onto EBC systems <u>before</u> solid particle erosion:



<u>CMAS composition (mol%):</u>
48% SiO_2, 31% CaO, 13% Al_2O_3, 8% MgO

Erosion Rate	Erosion Rate (mg loss/mg erodent)
No CMAS	22.5
2 mg/cm² CMAS	14.9
4 mg/cm² CMAS	6.3
2 mg/cm² CMAS After 100h at 1316C	21.0

Solid Particle Erosion of CMAS-exposed EBCs



When low CMAS loading (2 mg/cm²) is applied onto EBC systems <u>before</u> solid particle erosion:

- Exposure of $Yb_2Si_2O_7$ EBCs to 2 mg/cm^2 CMAS loading reduced the erosion rate by ~50%.
- Increasing the CMAS loading to 4 mg/cm^2 reduced the erosion further to ~25% of baseline.
- Heat treating for 100hr in air at 1316ºC eliminated the CMAS effect for low exposures, suggesting the reduction is related to reaction products (Yb-Ca-apatite).

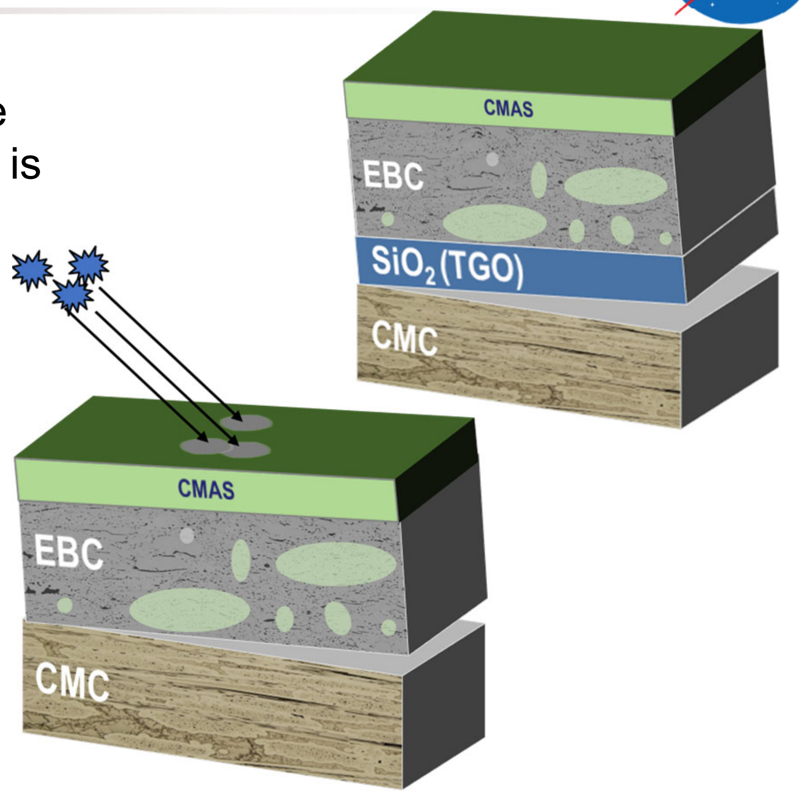
<u>CMAS composition (mol%):</u>
48% SiO_2, 31% CaO, 13% Al_2O_3, 8% MgO

Erosion Rate	Erosion Rate (mg loss/mg erodent)
No CMAS	22.5
2 mg/cm^2 CMAS	14.9
4 mg/cm^2 CMAS	6.3
2 mg/cm^2 CMAS After 100h at 1316C	21.0

Combined Mechanism Testing

- Presence of CMAS can reduce the apparent oxidation rate but significantly reduces the adhesion strength when TGO is already present.

- CMAS exposure reduces the erosion rate due to formation of secondary phases but over time the effect dissipates.

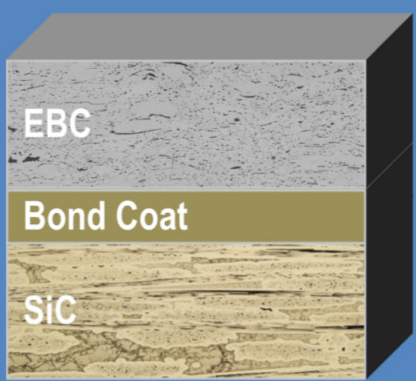


Combined mechanism testing shows
interactions can produce unexpected and complex results

Addressing EBC Design and Durability with Processing

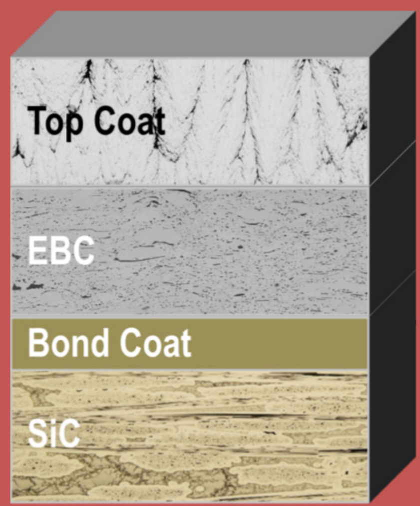
Microstructure

Can we change the porosity/form of the constituent material?



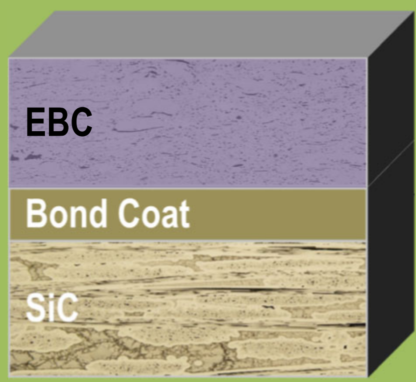
Architecture

Can we change the order of the layers or add additional layers?



Material Selection

Can we utilize a different material to do the job?



Easiest → Hardest

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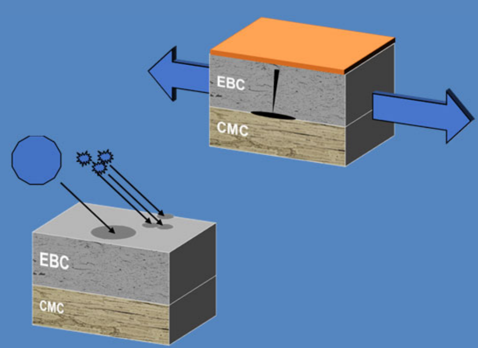

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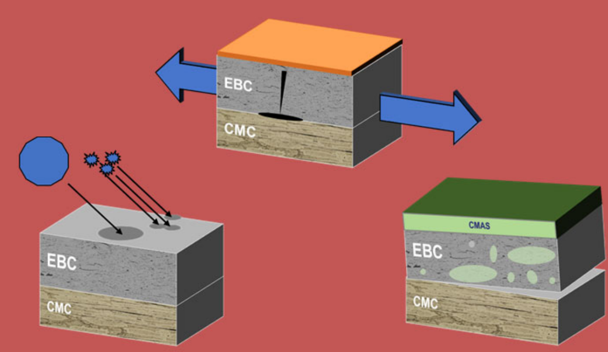

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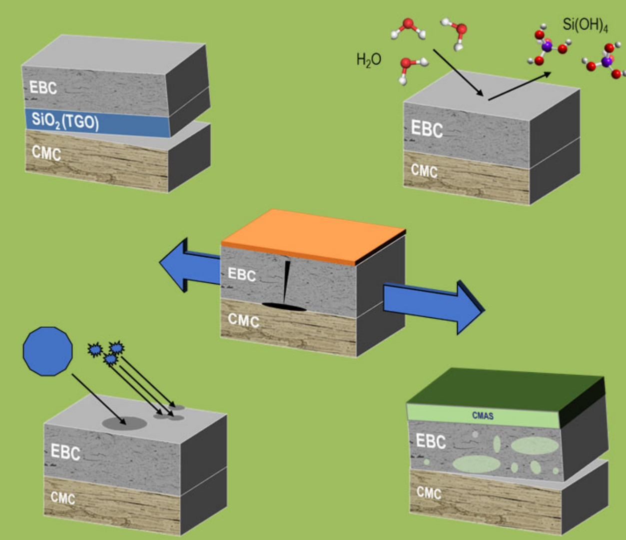
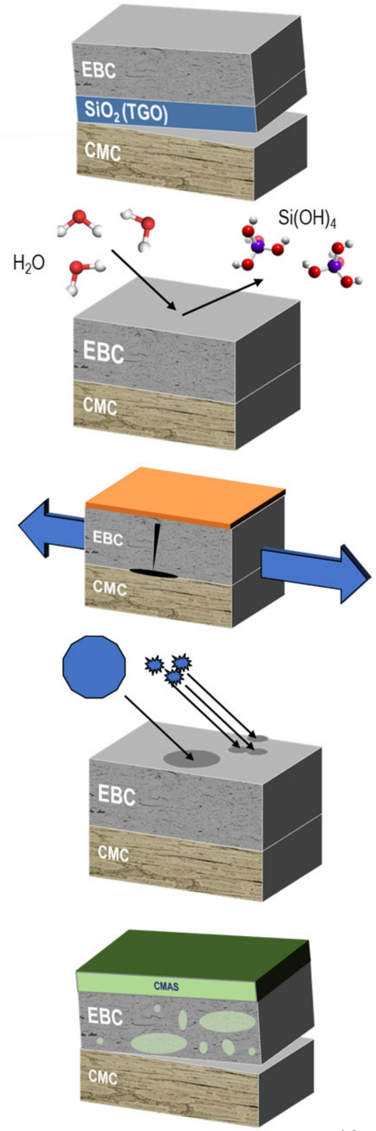


Easiest → **Hardest**

Future Direction and Challenges

- Design and process optimization of EBC systems will require a future emphasis on chemistry **<u>and</u>** mechanics due to the added complexity of these materials.
- Processing knowledge and experience as well as their connection to testing outcomes and failure will become increasingly important as more CMC components are integrated into service.
- Increased temperatures, longer life requirements and more aggressive environments will make a “one-size-fits-all” approach more difficult for all components for both material selection and coating processing.
- The combined effects of multiple failure modes will be critical to understand via testing to meet the aggressive life requirements of engine applications.
- CMAS is likely the most challenging problem on the horizon, as it will require new materials and test methods to design coatings capable of withstanding the thermochemical and thermomechanical (physical) effects of glass corrosion.



Acknowledgments

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