

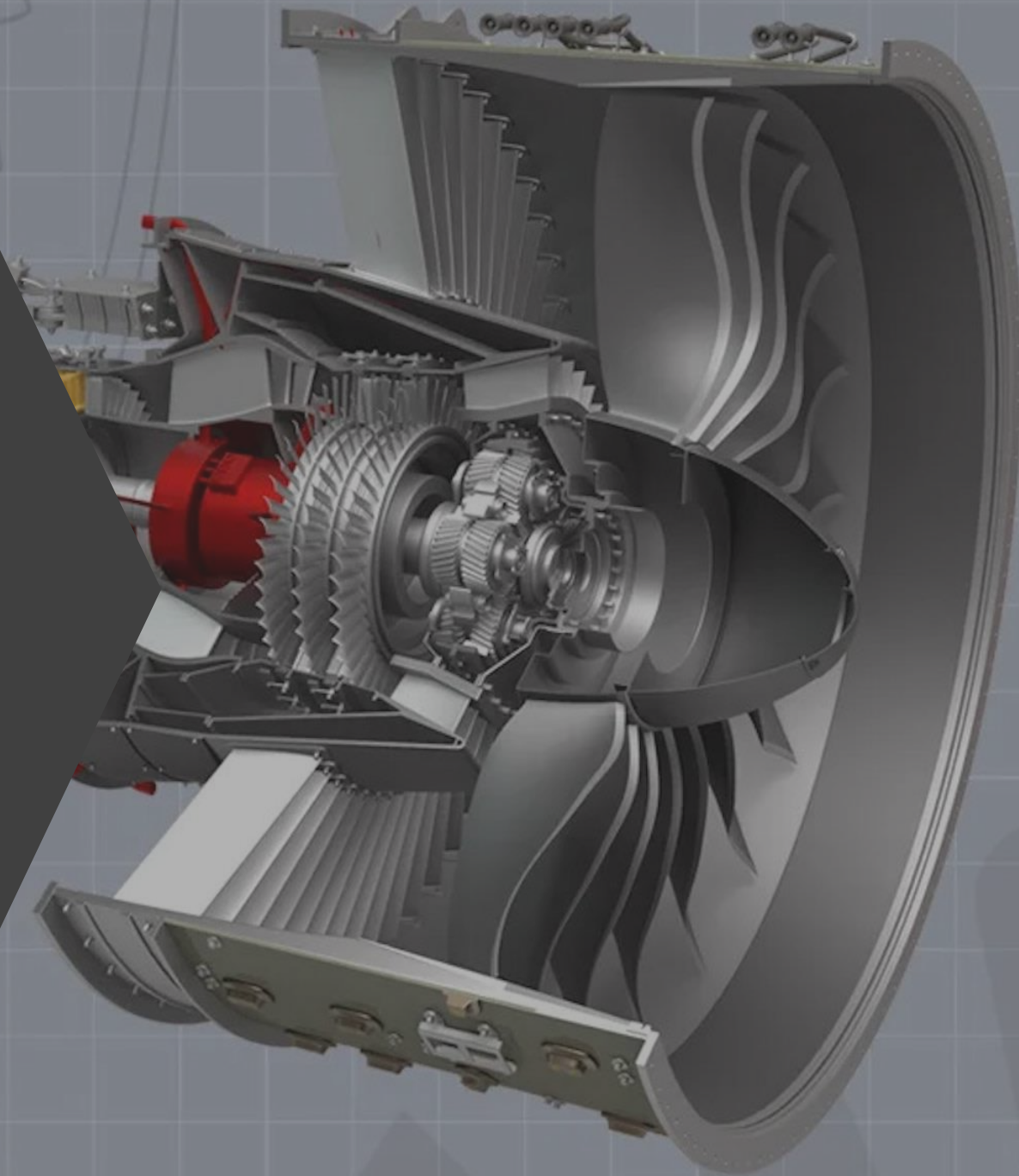


National Aeronautics and  
Space Administration

# Advanced Environmental Barrier Coating Testing and Development for Gas Turbine Engines

Penn State College of Engineering  
Center for Gas Turbine Research, Education, and  
Outreach

November 7, 2024





# John H. Glenn Research Center – Cleveland, OH

- Groundbreaking in 1940 in the National Advisory Committee on Aeronautics (NACA) as the Aircraft Engine Research Laboratory (AERL)
- Became a part of NASA in 1958
- Expertise in several fields contributing to agency missions and goals
  - Power, Energy Storage and Conversion
  - Aircraft Propulsion
  - Space Propulsion and Cryogenic Fluids Management
  - Communications Technology Development
  - Physical Sciences and Biomedical Technologies in Space
  - **Materials and Structures for Extreme Environments**







# Aeronautics Research Mission Directorate

All U.S. commercial aircraft and air traffic control facilities incorporate NASA-developed technology. That heritage continues at NASA, where the first “A” stands for Aeronautics, and the efforts to safely and sustainably transform aviation for the 21st century are managed by the agency’s Aeronautics Research Mission Directorate (ARMD).

- **Advanced Air Vehicles Program**
- Airspace Operations and Safety Program
- Integrated Aviation Systems Program
- **Transformative Aeronautics Concepts Program**
- Aerosciences Evaluation and Test Capabilities

**NASA’s Sustainable Flight National Partnership: Net Zero Carbon Emissions by 2050**





# Aeronautics Research Mission Directorate



## HyTEC (Hybrid Thermally Efficient Core) Project

- Design and demonstrate a jet engine that has a smaller core but produces equivalent thrust as engines being flown today on single-aisle aircraft.
- Smaller core technology aims to reduce fuel burn and emissions by an estimated 5 to 10%.

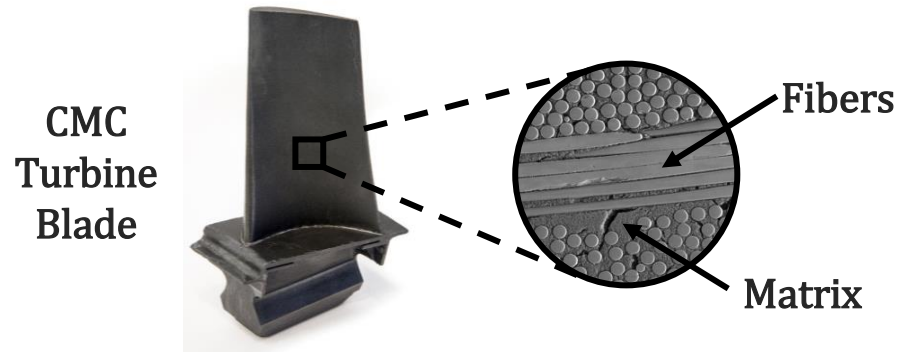
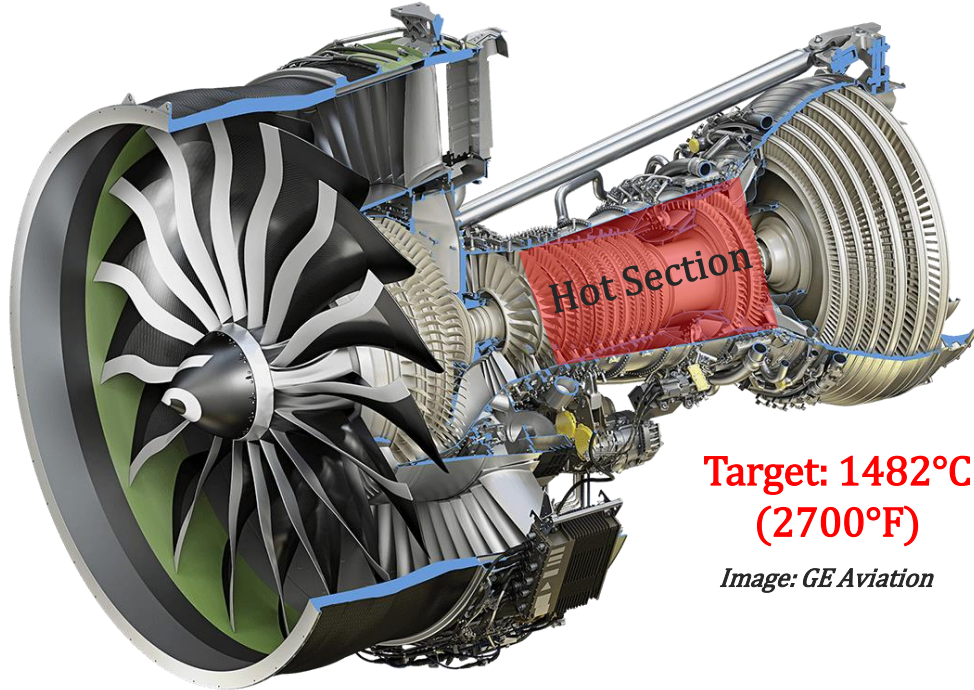
## Transformational Tools and Technologies (TTT) Project

- encourages revolutionary concepts, environment for researchers to experiment with new ideas
- Promote converging advancements in aeronautics and non-aeronautics sectors





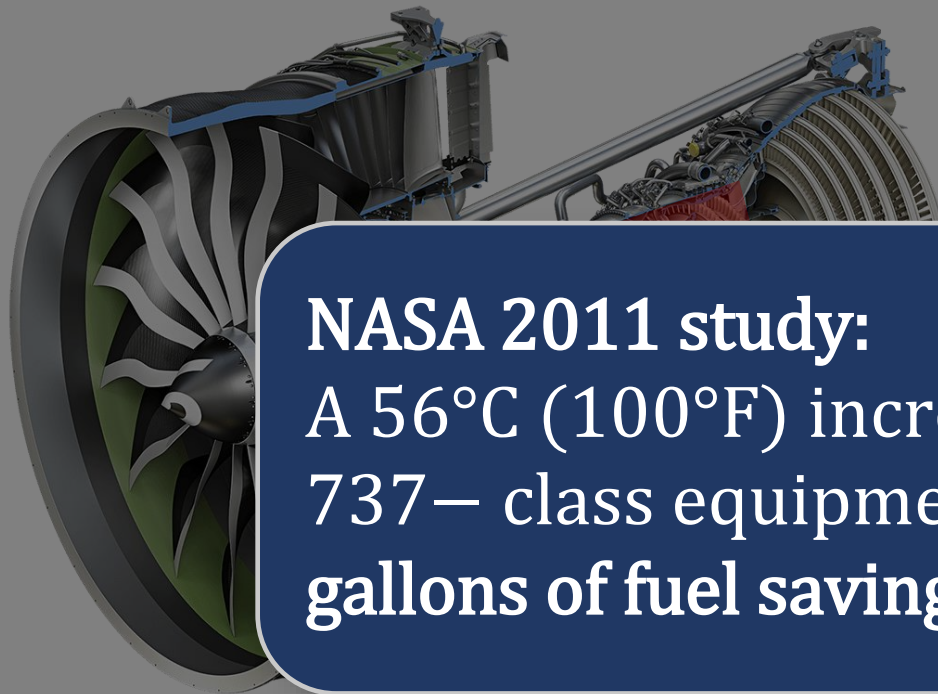
# Protective Coatings for Gas Turbine Engines



- Multi-decade development push to replace metallic airplane components with **lightweight composite materials**
    - Boeing 787 Dreamliner – Carbon-fiber composite fuselage
  - Replacement of Ni-based superalloy engine components with SiC-based ceramic matrix composites (CMCs) to **increase turbine engine efficiency (incorporated into aero engines in 2016)**
    - Lower (1/3) density than conventional metal-based components
    - Allow for higher operating temperatures (>1200°C)
- $$\varepsilon = 1 - \frac{T_c}{T_H}$$
- **6% increase in fuel efficiency savings of ~\$400,000 per plane per year**



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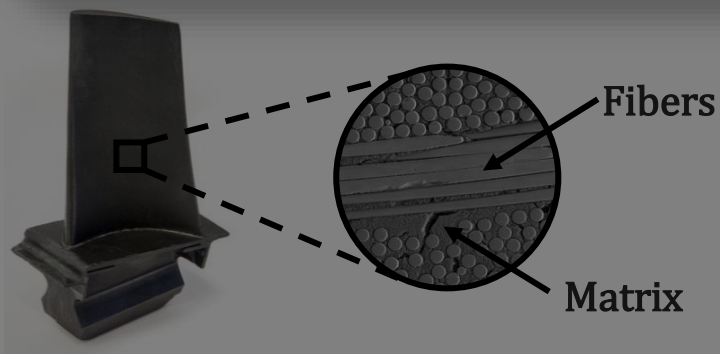


- Multi-decade development push to replace metallic airplane components with **lightweight composite materials**
- Boeing 787 Dreamliner – Carbon-fiber composite

## 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.**

CMC  
Turbine  
Blade



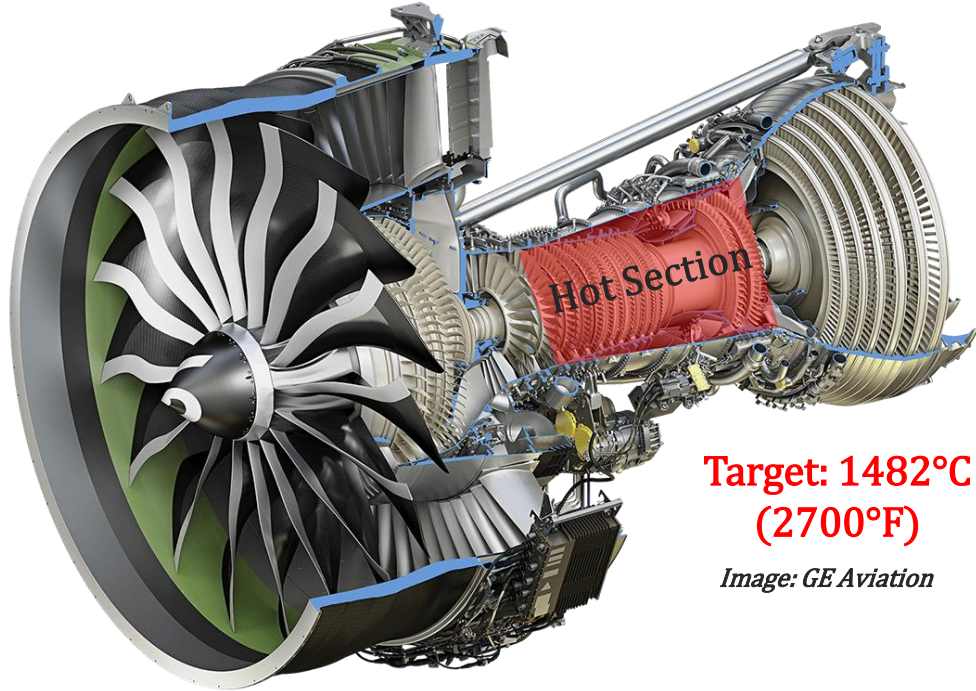
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$$\varepsilon = 1 - \frac{T_c}{T_H}$$

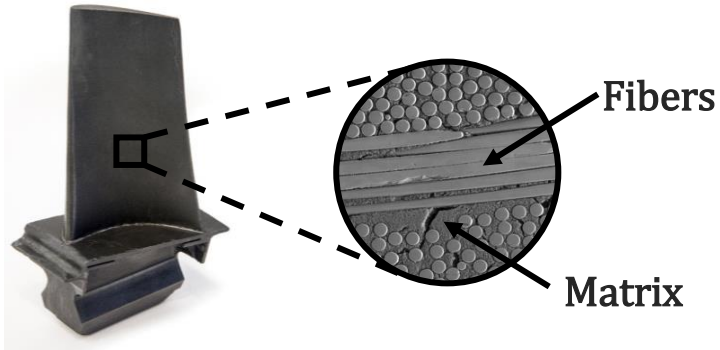
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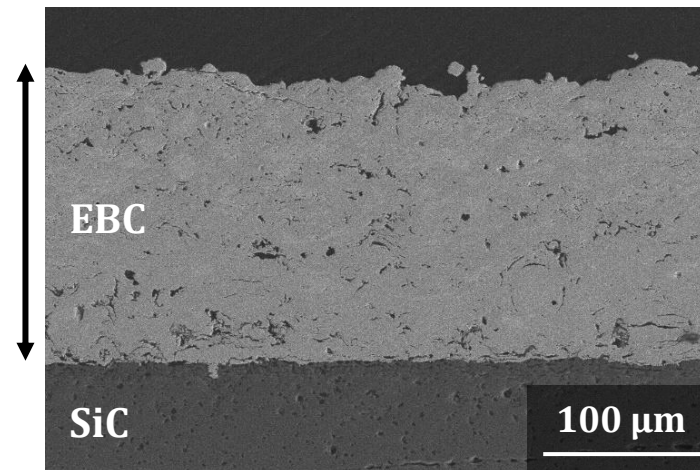


CMC  
Turbine  
Blade



- Silicon carbide (SiC) CMCs susceptible to environmental attack at temperatures  $>800^{\circ}\text{C}$  in oxygen and water vapor
  - Silica ( $\text{SiO}_2$ ) scale formation that volatilizes in  $\text{H}_2\text{O}$  environment
  - Surface recession
- Require **environmental barrier coatings (EBCs)** to protect CMC component from harsh environment

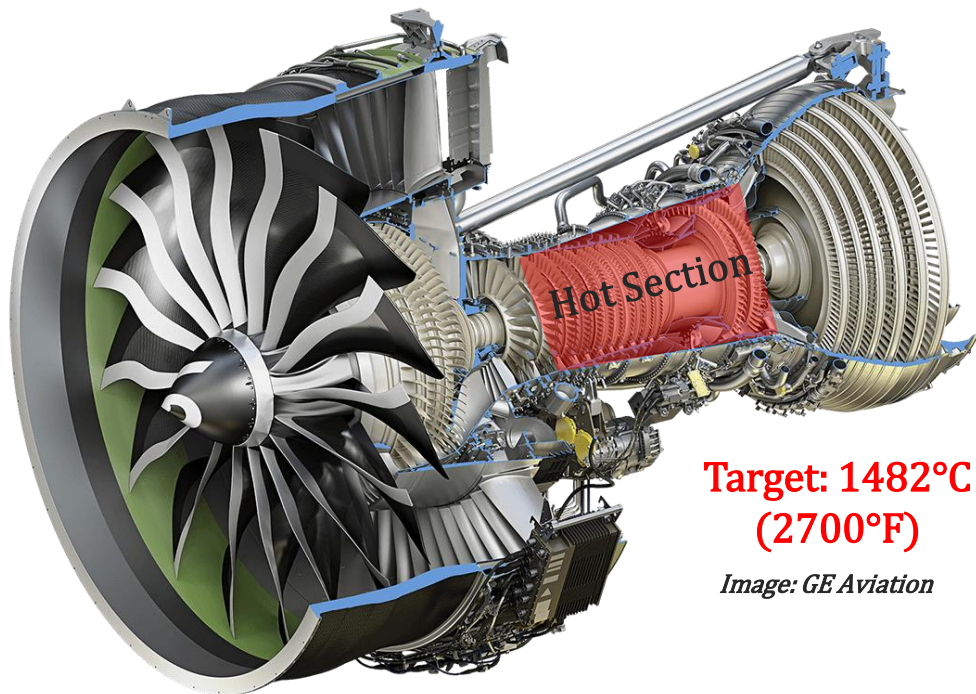
~150 to  
400  $\mu\text{m}$







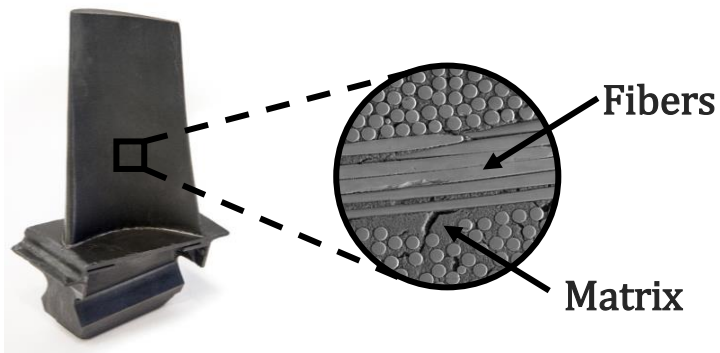
# Protective Coatings for Gas Turbine Engines



Target: 1482°C  
(2700°F)

Image: GE Aviation

CMC  
Turbine  
Blade



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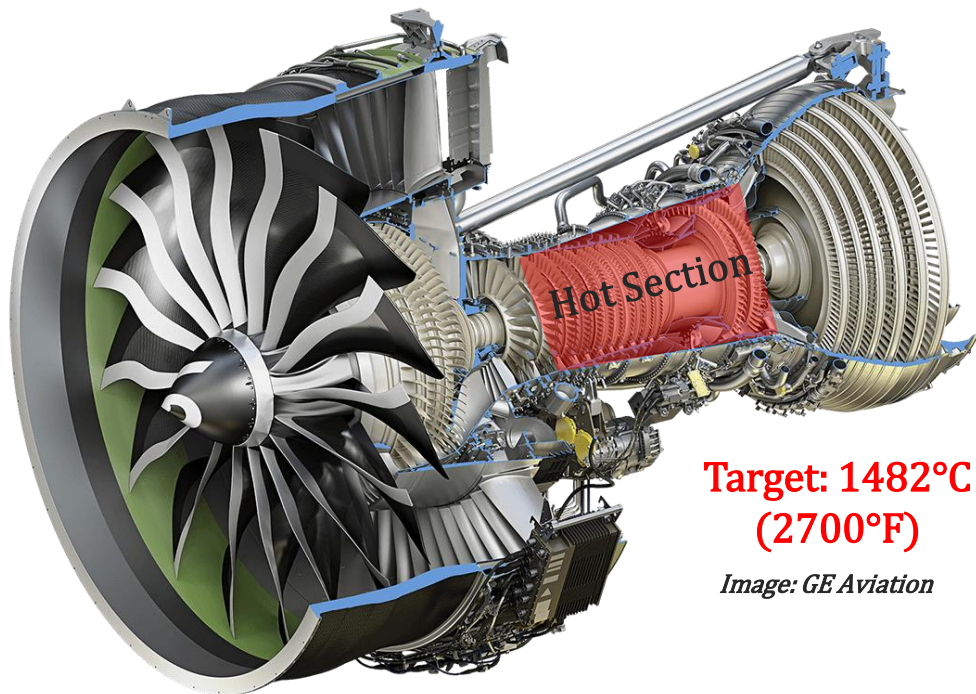
## Intrinsic Material Selection Criteria

- |  |                               |
|--|-------------------------------|
| • Coefficient of thermal expansion (CTE)                           | • Phase Stability             |
| • Sintering resistance   | • Low Modulus                 |
| • Low $\text{H}_2\text{O}$ and $\text{O}_2$ diffusivity/solubility | • Limited coating interaction |





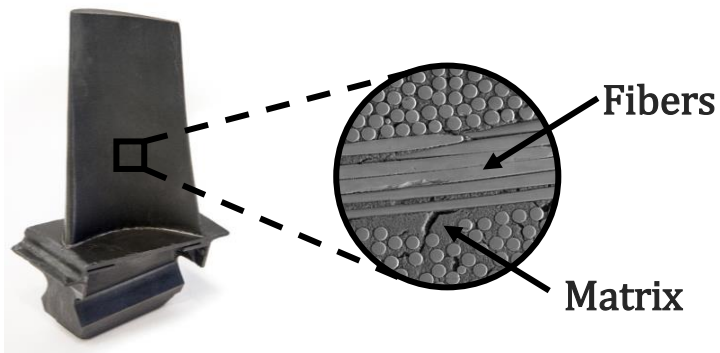
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**Target: 1482°C  
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CMC  
Turbine  
Blade



## Turbine Engine Requirements

SiC Recession: Fuel-lean mixture -  $P_{total} \sim 6 \text{ atm}$ ,  $v \sim 20 \text{ m/s}$

Time [hours]	1,200°C [ $\mu\text{m}/\text{side}$ ]	1,300°C [ $\mu\text{m}/\text{side}$ ]	1,400°C [ $\mu\text{m}/\text{side}$ ]
100	6.187	11.01	18.29
1,000	61.87	110.1	182.9
10,000	618.7 ( $\sim 0.62 \text{ mm}$ )	1101 ( $\sim 1.1 \text{ mm}$ )	1829 ( $\sim 1.8 \text{ mm}$ )

SiC Recession: Fuel-rich mixture -  $P_{total} \sim 6 \text{ atm}$ ,  $v \sim 20 \text{ m/s}$

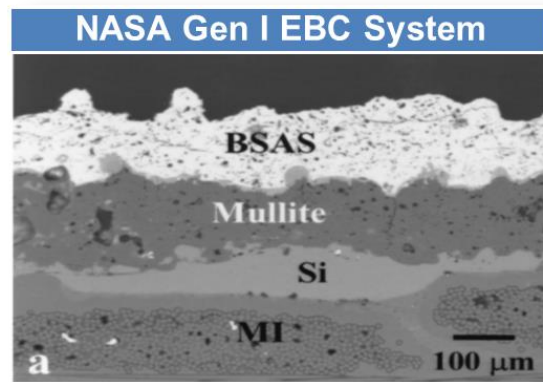
Time [hours]	1,200°C [ $\mu\text{m}/\text{side}$ ]	1,300°C [ $\mu\text{m}/\text{side}$ ]	1,400°C [ $\mu\text{m}/\text{side}$ ]
100	10.67	25.94	56.69
1,000	106.7	259.4	566.9
10,000	1067 ( $\sim 1.1 \text{ mm}$ )	2594 ( $\sim 2.6 \text{ mm}$ )	5669 ( $\sim 5.7 \text{ mm}$ )

- Current engines require materials to have durability  $>10,000$  hours
- Supersonic turbine engines require 6,000 – 9,000 hours at max temperature



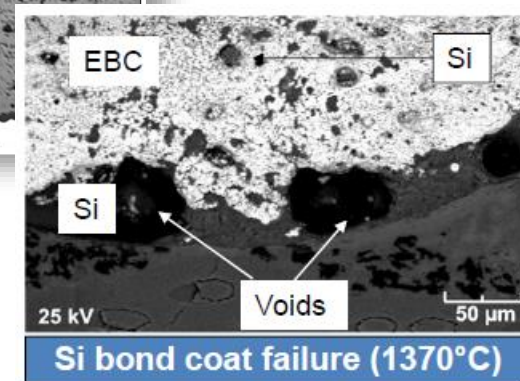
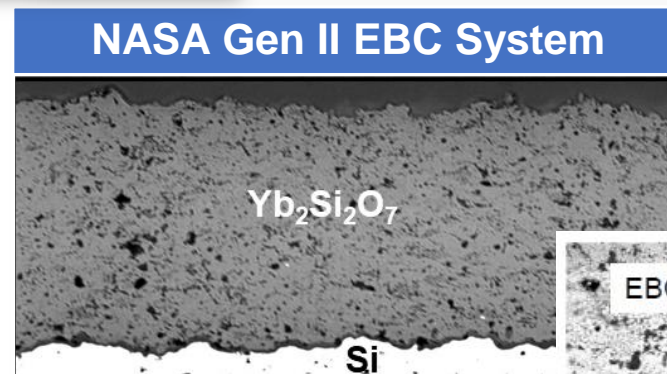
# EBC Development History

- Gen I EBC developed at NASA GRC in collaboration with GE and P&W
- BSAS/Mullite/Silicon multilayer
  - Low  $\text{SiO}_2$  activity
  - Good CTE match
- Silicon (Si) bond coat added to improve adhesion and oxidation resistance
- Gen II EBC developed at NASA GRC in early 2000s
  - Rare earth silicate-based system
  - $\text{RE}_2\text{SiO}_5$ ,  $\text{RE}_2\text{Si}_2\text{O}_7$
  - $\text{RE} = \text{Y}, \text{Yb}, \text{Sc}, \text{Lu}, \text{etc.}$
  - Higher thermodynamic stability compared to Gen I EBC systems
  - Upper use temperature limited by Si bond coat ( $T_m \sim 1410^\circ\text{C}$ )



Mullite:  $(3\text{Al}_2\text{O}_3:2\text{SiO}_2)$

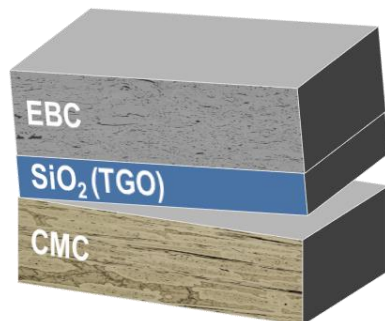
BSAS:  $1-x\text{BaO} \cdot x\text{SrO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ,  $0 < x < 1$



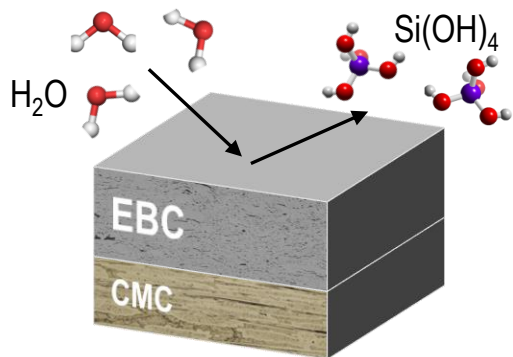




# Environmental Barrier Coating Failure Modes



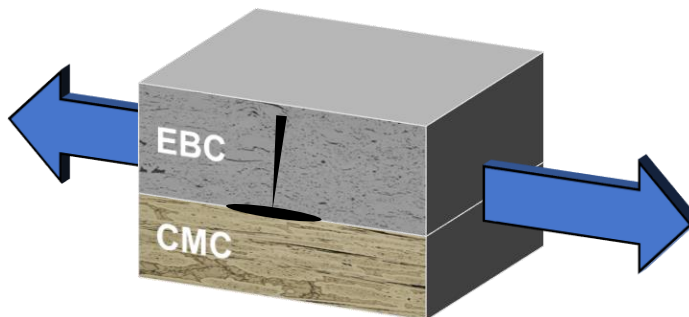
Steam Oxidation



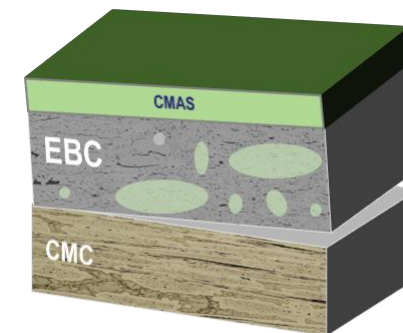
Hydroxide Formation/Recession

**Testing of EBC systems is critical**  
**Individual mechanisms must be well understood before evaluating combinatorial effects**

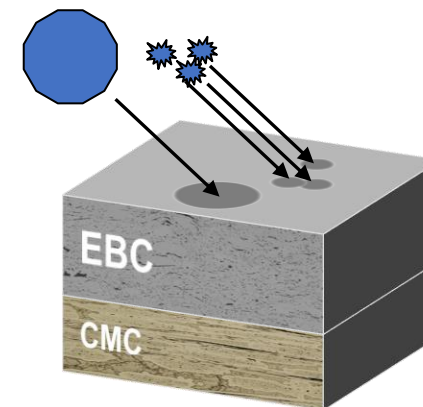
**Synergies between extrinsic failure modes determine EBC lifetime and design requirements**



Thermomechanical Durability



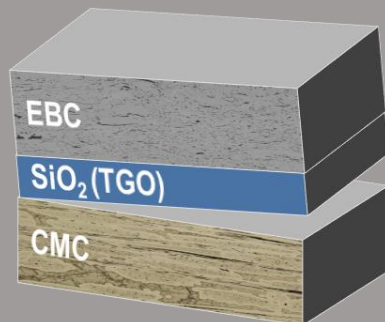
Molten Silicate Attack and Infiltration



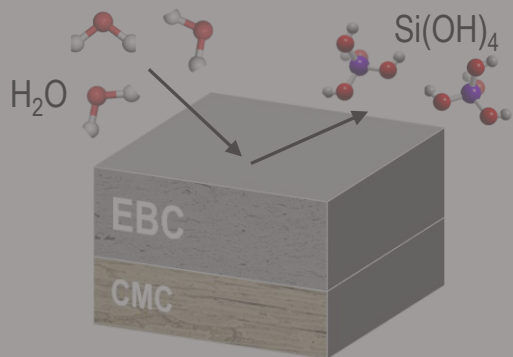
Erosion and FOD



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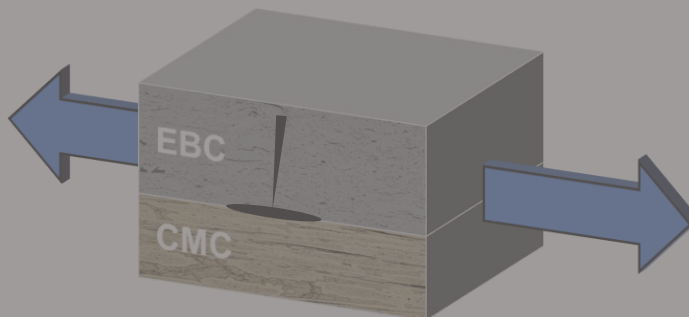
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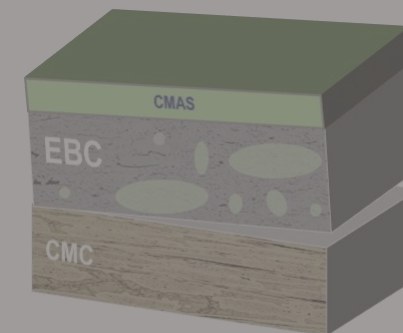
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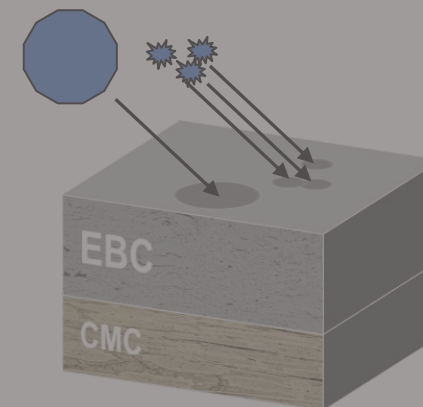
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Thermomechanical Durability



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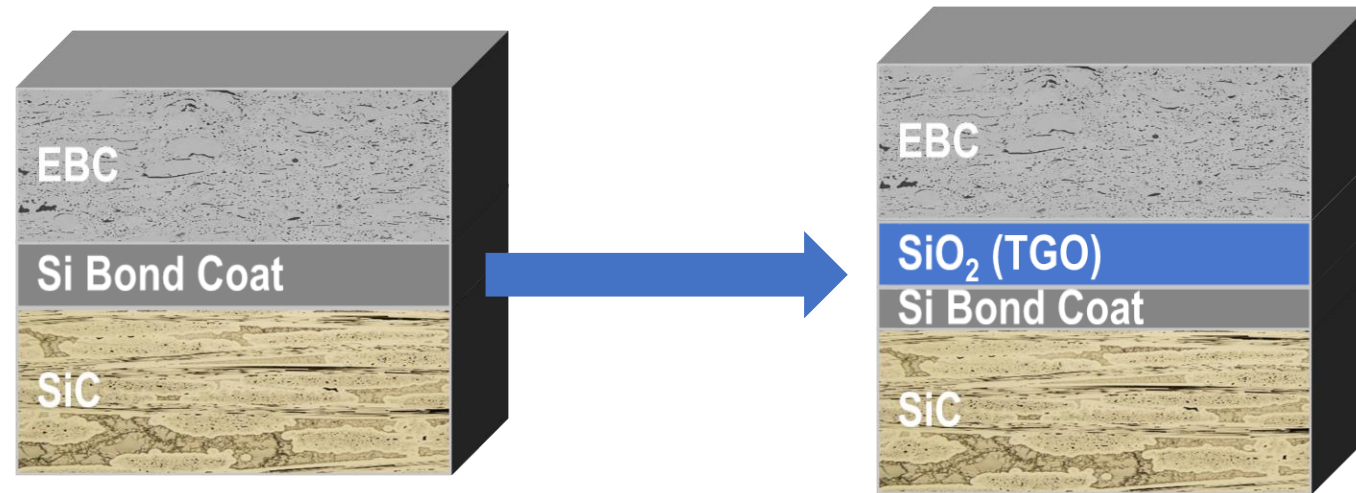
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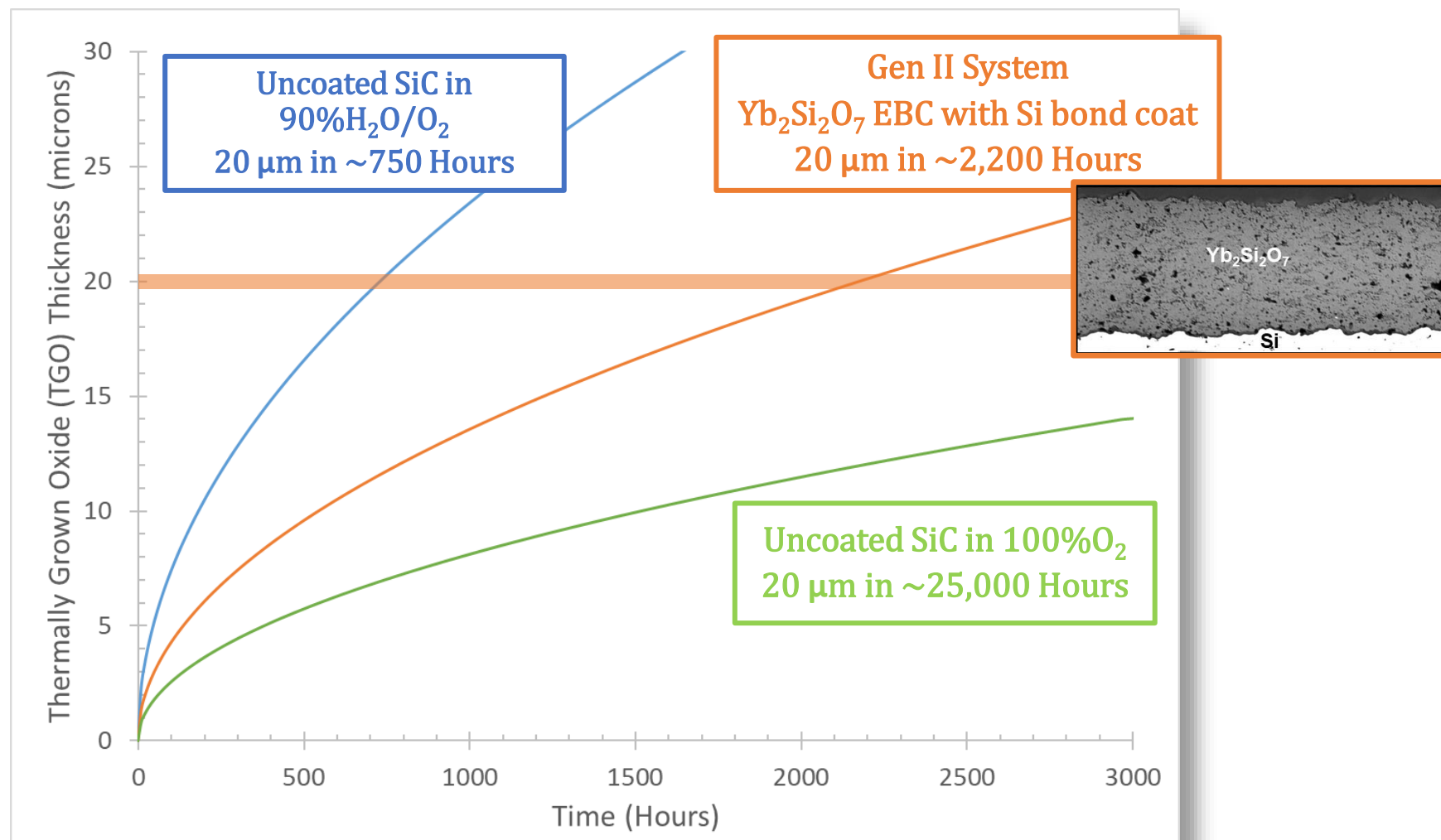
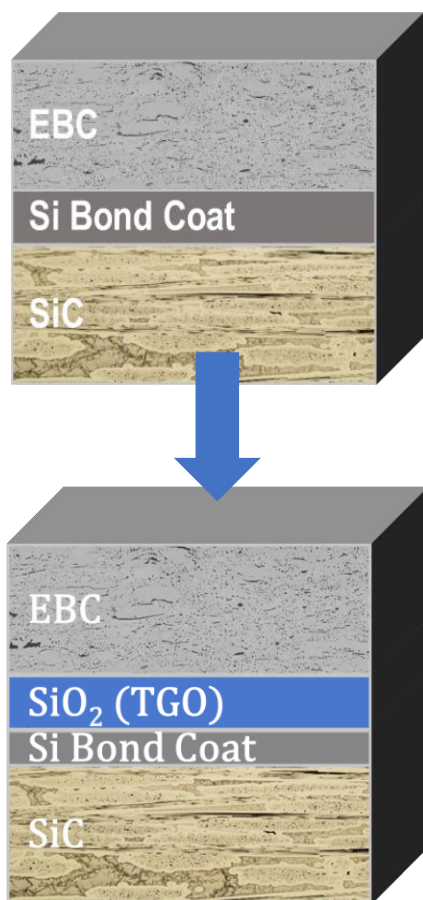
# Steam Oxidation

- Limiting the formation and growth of  $\text{SiO}_2$  layer is critical to long-life and durability requirements
- Thermally grown oxide (TGO) layer is the weak link in coating system due to CTE mismatch and phase transformation
- Oxidation of Si-based ceramics (including Si) is an order of magnitude or more in steam versus dry air





# Steam Oxidation







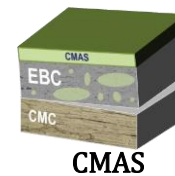
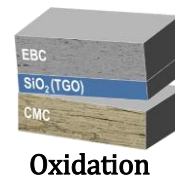
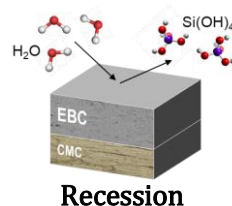
# Steam Oxidation

## Steam Cycling

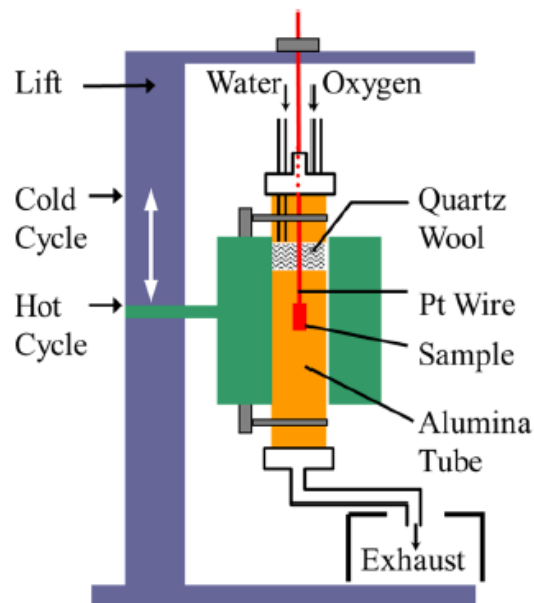
$$P(\text{H}_2\text{O}) = 0.9\text{atm}$$

$$V = 10 \text{ cm/s}$$

$$P_{\text{total}} = 1 \text{ atm}$$



## Steam Cycling Schematic



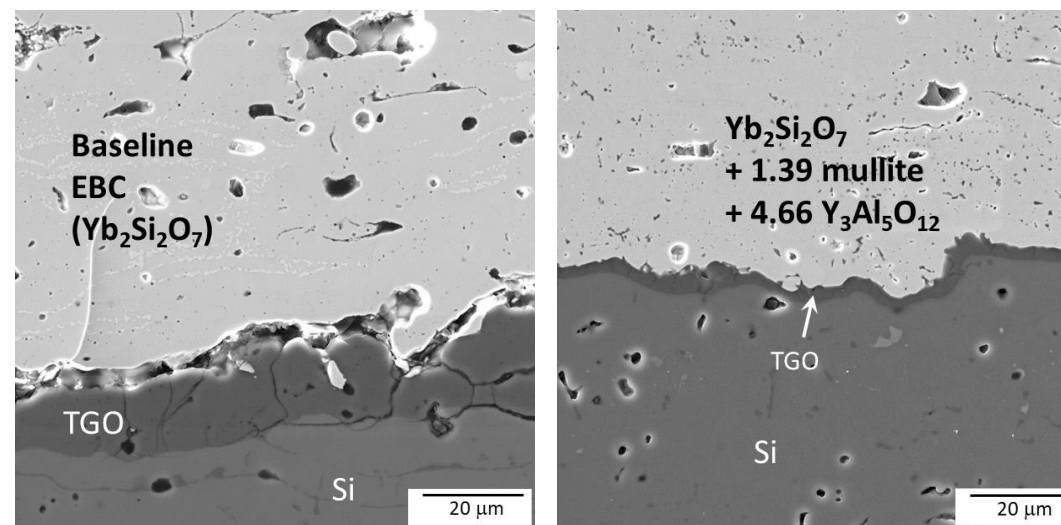
- Continuous cycling of samples at temperature forms thermally grown oxide (TGO) –  $\text{SiO}_2$ .
  - TGO is the weak interface
  - Life-limiting factor
- Steam oxidation provides 10x increase in TGO thickness versus dry air
- Cycling in 90%  $\text{H}_2\text{O}/\text{O}_2$  is akin to 9 atm combustion environment



# Steam Oxidation

- K. Lee (NASA) modified a  $\text{Yb}_2\text{Si}_2\text{O}_7$  EBC with  $\text{Al}_2\text{O}_3$ , mullite,  $\text{Y}_3\text{Al}_5\text{O}_{12}$ , and  $\text{TiO}_2$ 
  - Published in 2019
- APS 2-layer system consisting of a Si bond coat and the modified EBC topcoat
- Samples were cycled for up to 1000 hours in 90%  $\text{H}_2\text{O}$  / 10%  $\text{O}_2$  at 1316°C
- $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ -containing modifiers showed the largest reduction in TGO growth rate
  - TGO was shown to contain  $\text{Al}_2\text{O}_3$
- Reduction in the TGO thickness translates to potentially **>20x** life improvement over baseline systems

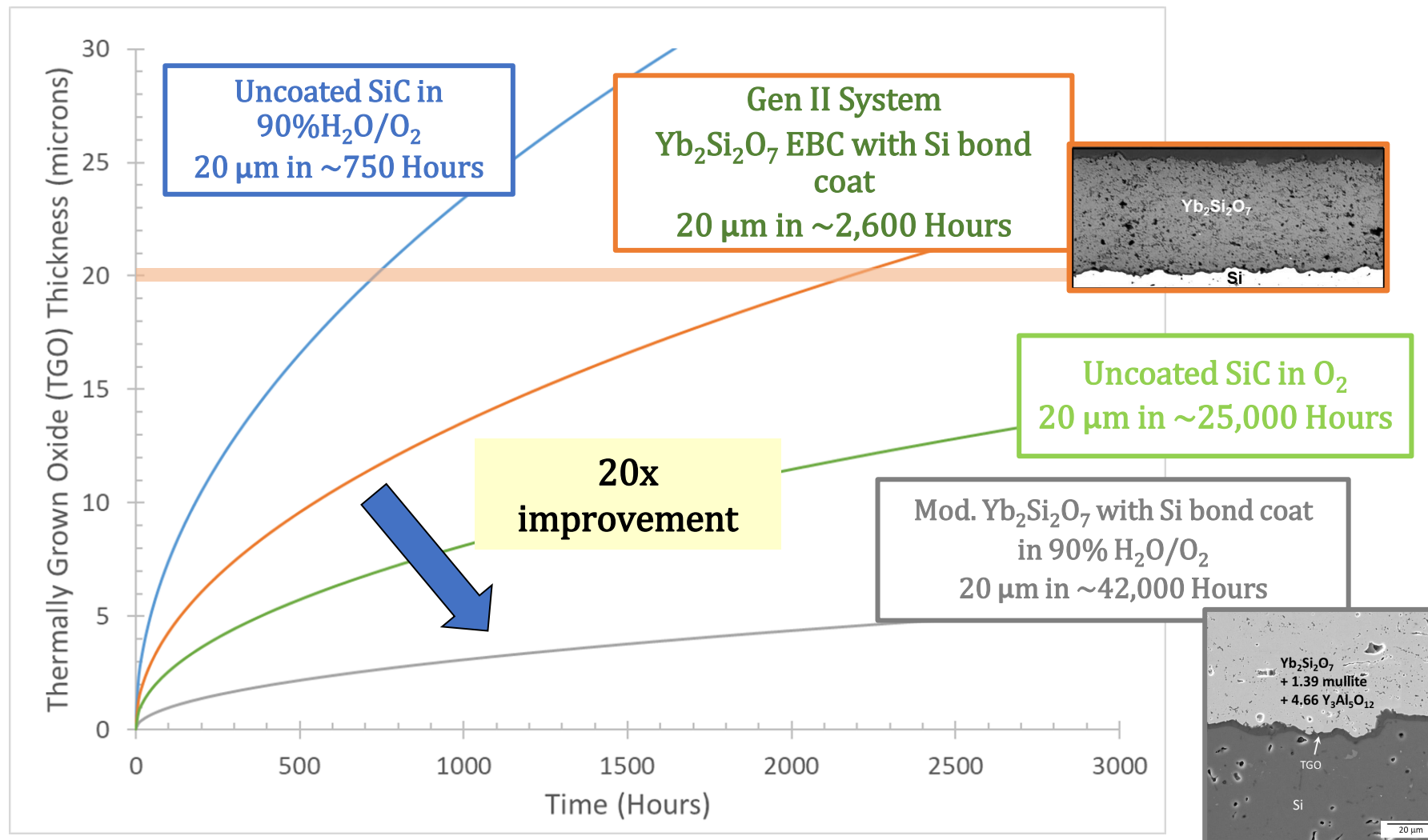
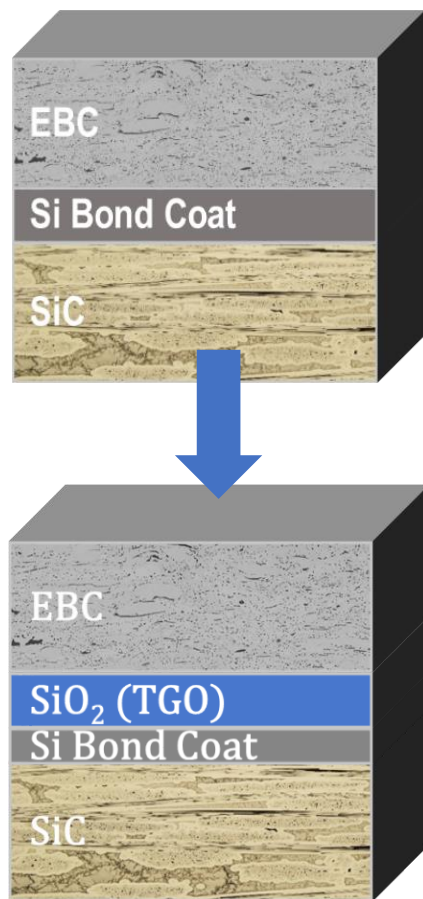
1 hour hot / 1000 cycles at 1316°C





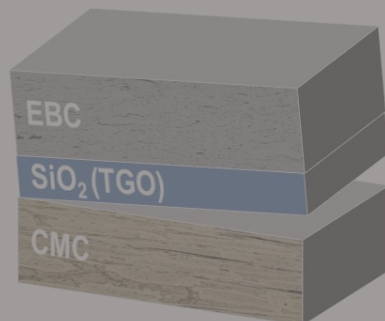


# Steam Oxidation





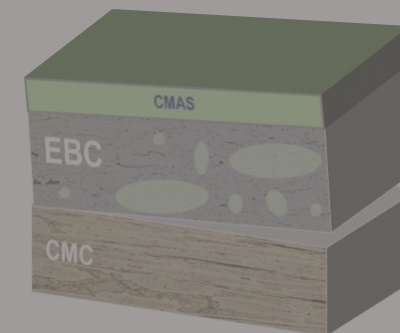
# Environmental Barrier Coating Failure Modes



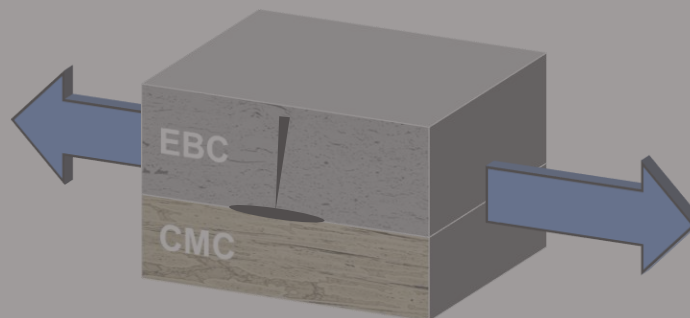
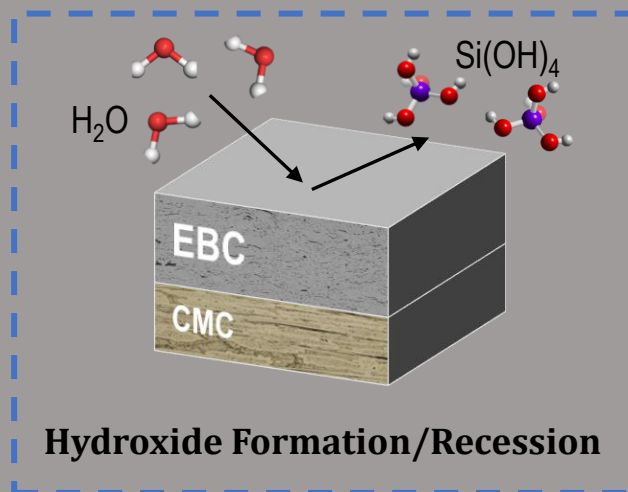
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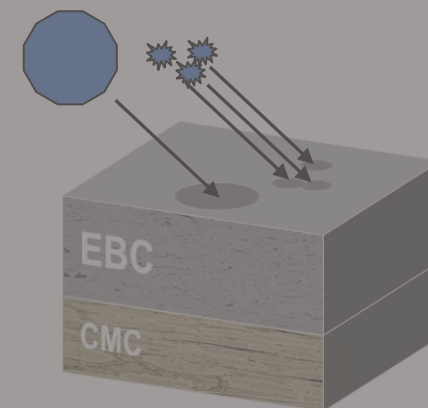
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Molten Silicate Attack and Infiltration



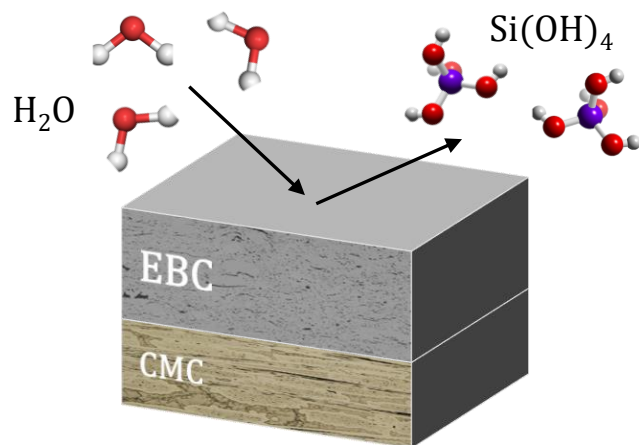
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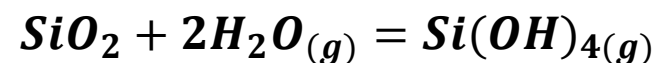
Erosion and FOD



# Hydroxide Formation/Recession



Hydroxide Formation/Recession



CTE too high  
( $>10 \times 10^{-6}/^\circ\text{C}$ )

CTE too high or  
anisotropic

Currently used  
CTE  $\sim 4\text{--}6 \times 10^{-6}/^\circ\text{C}$

Not protective

Most Water Vapor Resistant

HfO<sub>2</sub>

ZrO<sub>2</sub> (YSZ)

2(Y<sub>2</sub>O<sub>3</sub>) • 3(ZrO<sub>2</sub>)

3(Y<sub>2</sub>O<sub>3</sub>) • 5(Al<sub>2</sub>O<sub>3</sub>) (YAG)

Yb<sub>2</sub>O<sub>3</sub> • SiO<sub>2</sub>

Y<sub>2</sub>O<sub>3</sub> • SiO<sub>2</sub>

Al<sub>2</sub>O<sub>3</sub> • TiO<sub>2</sub>

Yb<sub>2</sub>O<sub>3</sub> • 2(SiO<sub>2</sub>)

Y<sub>2</sub>O<sub>3</sub> • 2(SiO<sub>2</sub>)

Ba(Sr)O • Al<sub>2</sub>O<sub>3</sub> • 2(SiO<sub>2</sub>) (BSAS)

Al<sub>2</sub>O<sub>3</sub>

TiO<sub>2</sub>

SiO<sub>2</sub>

Cr<sub>2</sub>O<sub>3</sub>

Least Water Vapor Resistant



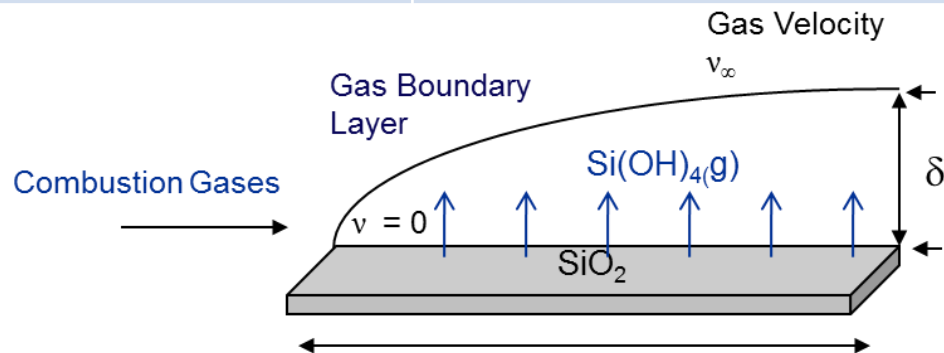


# Hydroxide Formation/Recession

## Fundamental Thermochemistry

Instrument	Measurements
Mass Spectrometry (2000°C)	Products, activities, vapor pressure, enthalpy of vaporization
TGA (1650°C air, 2400°C vacuum)	Wt. change, oxidation, reduction, vaporization
Drop Solution Calorimeter	Enthalpy of formation, reaction, and mixing

- Develop an understanding of high temperature material behavior from fundamental thermodynamics, chemistry, and kinetics.
- Experimental and computational methods.
- Modeling can be used to determine potential gaseous species



NASA GRC identified  $\text{Si(OH)}_4$  product for reaction of SiC with moisture – reaction is life limiting to SiC/SiC durability in turbine engines.



**KEMS**



**Thermo-gravimetric Analysis** (air/water/vacuum)



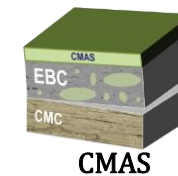
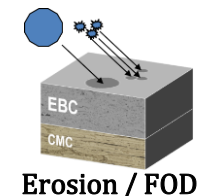
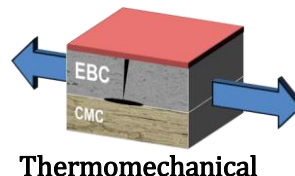
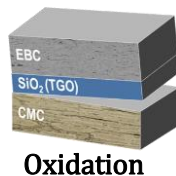
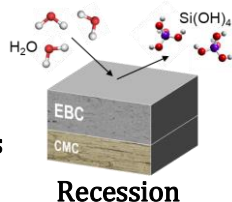
**Calorimeter**



# Hydroxide Formation/Recession

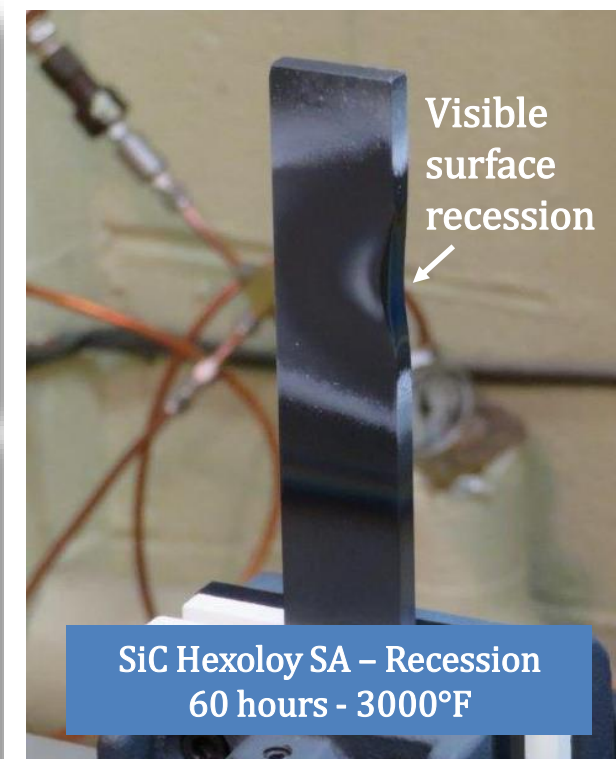
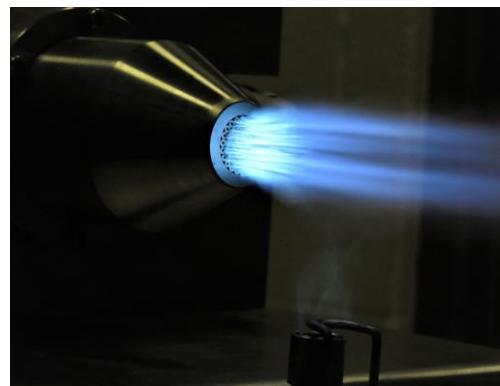
## NG/O<sub>2</sub> Burner Rig

$P(\text{H}_2\text{O}) = 0.1 - 0.5 \text{ atm}$   
 $V \sim 100 - 250 \text{ (est.) m/s}$   
 $P_{\text{total}} = 1 \text{ atm}$



## Chemical Equilibrium Analysis (CEA)

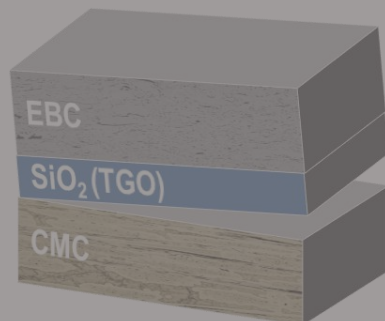
- Peak adiabatic flame temperature – 4,986°F (~2,750°C)
- $P(\text{H}_2\text{O}) \sim 0.1 - 0.4 \text{ atm} \rightarrow P(\text{total}) = 1 \text{ atm}$
- Gas velocity estimated:  $\sim 100 - 300 \text{ m/s}$
- High heat flux (up to  $\sim 200 \text{ W/cm}^2$ )



SiC Hexoloy SA – Recession  
60 hours - 3000°F



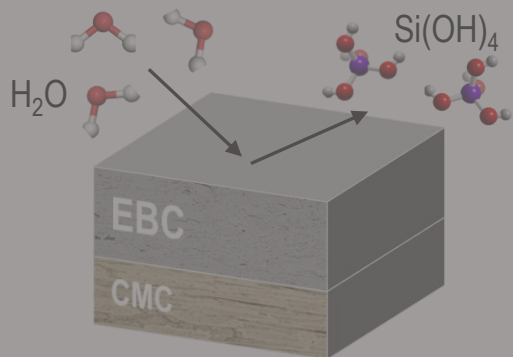
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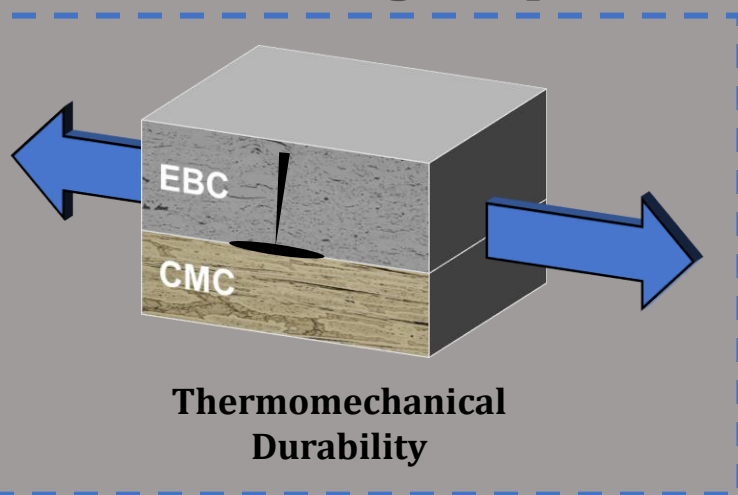
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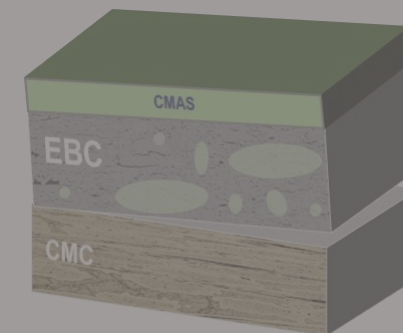
Synergies between extrinsic failure modes determine EBC lifetime and design requirements



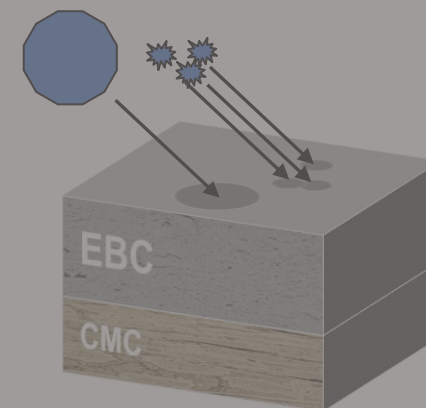
Hydroxide Formation/Recession



Thermomechanical Durability



Molten Silicate Attack and Infiltration



Erosion and FOD





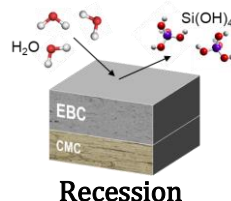
# Thermomechanical Durability

## High Heat Flux Laser

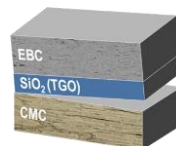
$P(\text{H}_2\text{O}) = \text{none}$

$V = \text{none}$

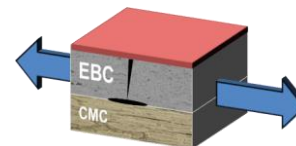
$P_{\text{total}} = 1 \text{ atm}$



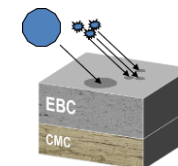
Recession



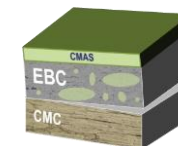
Oxidation



Thermomechanical

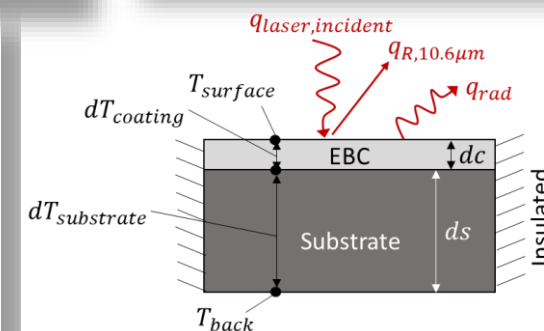
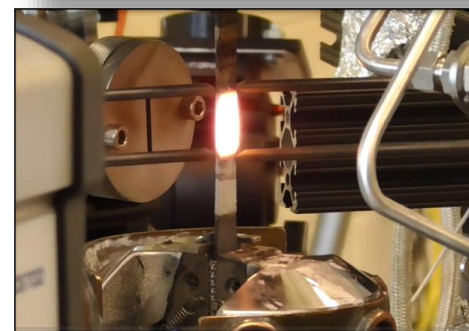
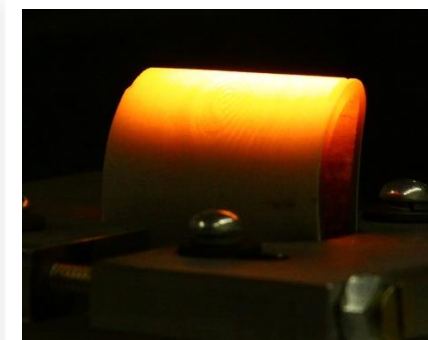
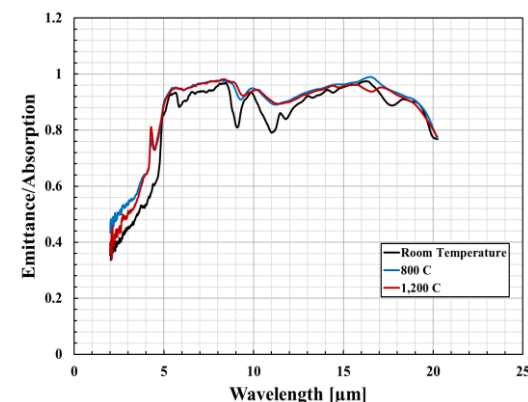


Erosion / FOD



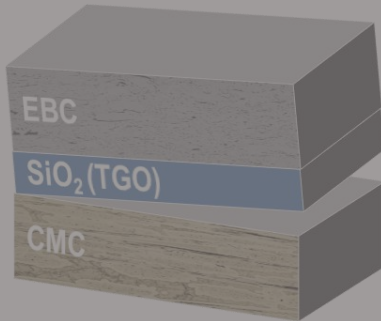
CMAS

- High-powered Carbon Dioxide ( $\text{CO}_2$ ) lasers
  - 10.6  $\mu\text{m}$  wavelength ideal for EBC candidate materials
  - High absorption
- Thermal or combined thermal-mechanical testing capabilities
- Surface temperatures  $> 3,000^\circ\text{F}$  ( $1650^\circ\text{C}$ ) are achievable
- Incident heat flux up to  $300 \text{ W}/\text{cm}^2$  (assuming 1 inch diameter spot size)

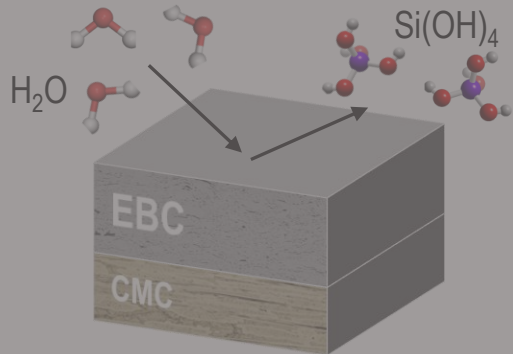




# Environmental Barrier Coating Failure Modes



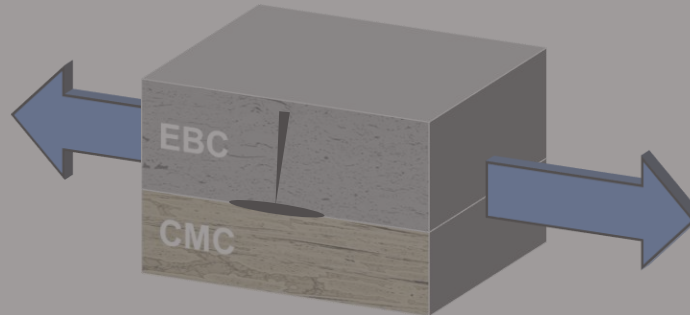
Steam Oxidation



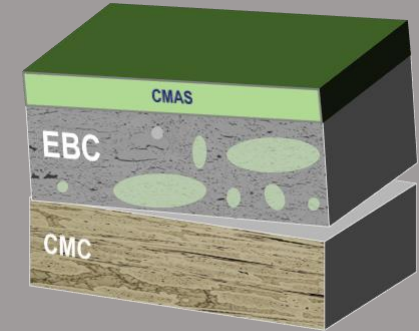
Hydroxide Formation/Recession

Testing of EBC systems is critical  
Individual mechanisms must be well understood before evaluating combinatorial effects

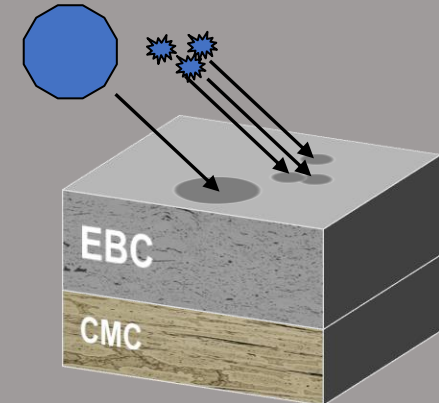
Synergies between extrinsic failure modes determine EBC lifetime and design requirements



Thermomechanical Durability



Molten Silicate Attack and Infiltration



Erosion and FOD



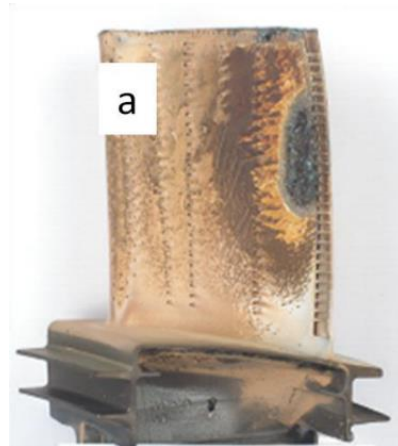
# Degradation by Particulate Ingestion

## CMAS

- Particulates (i.e. sand, volcanic ash) ingested by engine melt into **Calcium-Magnesium-Alumino-Silicate (CMAS)** deposits above 1200°C
- Molten CMAS degrades T/EBCs (chemical + mechanical)
  - CMAS infiltration of T/EBC due to lowered CMAS viscosity at elevated temperatures → CTE mismatch
  - Thermochemical interactions of CMAS with T/EBC → spallation



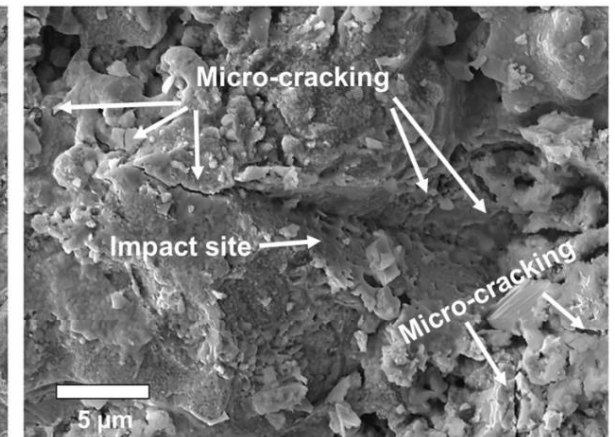
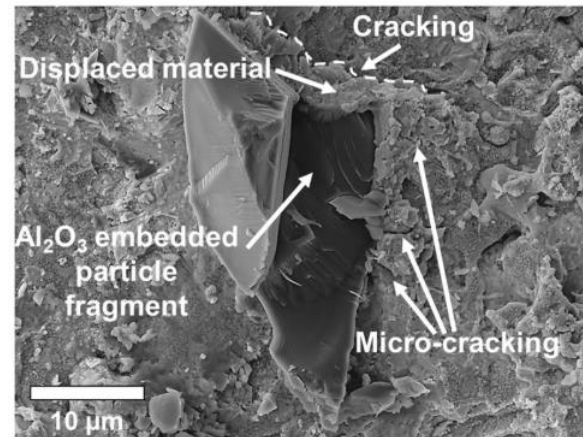
*Eyjafjallajökull volcano eruption in Iceland (2010)*



*Damage on a turbine blade caused by CMAS >1200°C*

## Solid Particle Erosion

- Particulates (i.e. sand, volcanic ash) ingested by engine can **mechanically erode** T/EBCs at higher temperatures
- Brittle fracture dominated erosion response of T/EBCs at high temperature
  - Coating microstructure affects durability



Presby et al., *Ceramics International* **47** (2021)

Presby et al., *Coatings* **13** (2023)





# Degradation by Particulate Ingestion

**Relative Composition (mol%) of Sources and Deposits\***

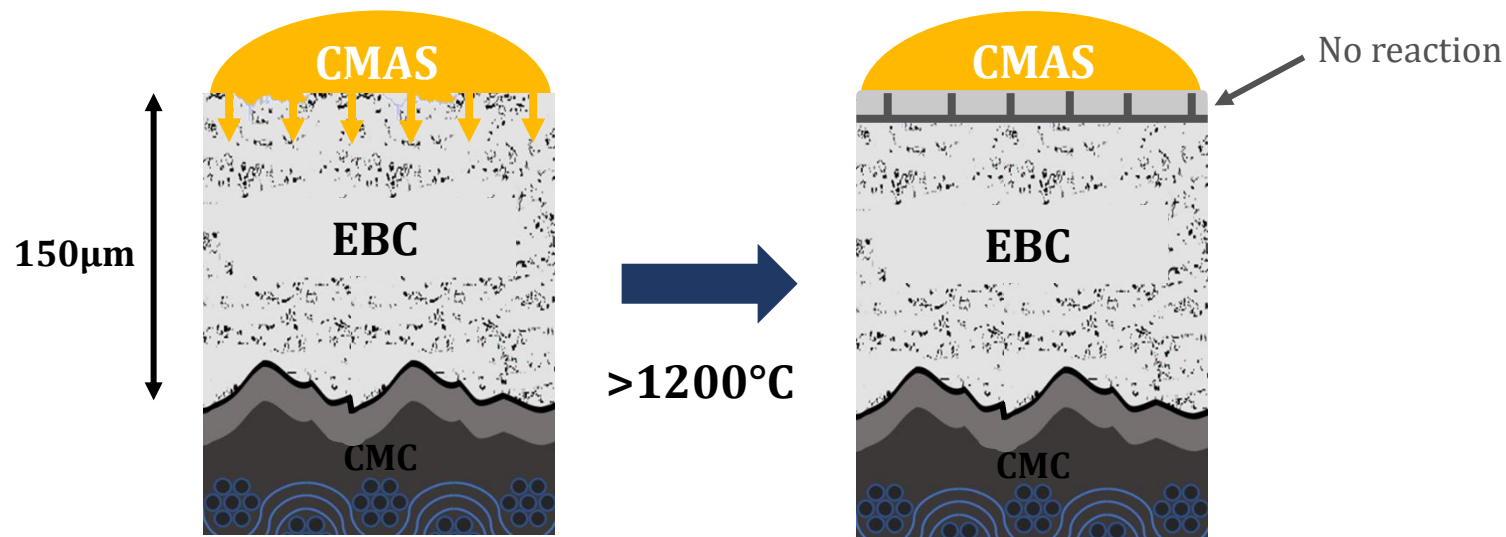
	SiO <sub>2</sub>	CaO	MgO	AlO <sub>1.5</sub>	FeO	CaO/SiO <sub>2</sub>
Earth's Crust	65	6	6	10	4	0.093
Saudi Sand	93	1	< 1	4	< 1	0.011
Airport Runway Dust	75	5	2	15	4	0.067
Volcanic Ash	65	5	4	18	5	0.077
Fly Ash	40	5-20	5	20	5-20	0.125-0.5
<b>Engine Deposits</b>	<b>25-40</b>	<b>20-35</b>	<b>7-15</b>	<b>10-15</b>	<b>7-15</b>	<b>0.5-1.43</b>

**Engine Deposits have a wide  
composition range!**



# CMAS Mitigation Strategies

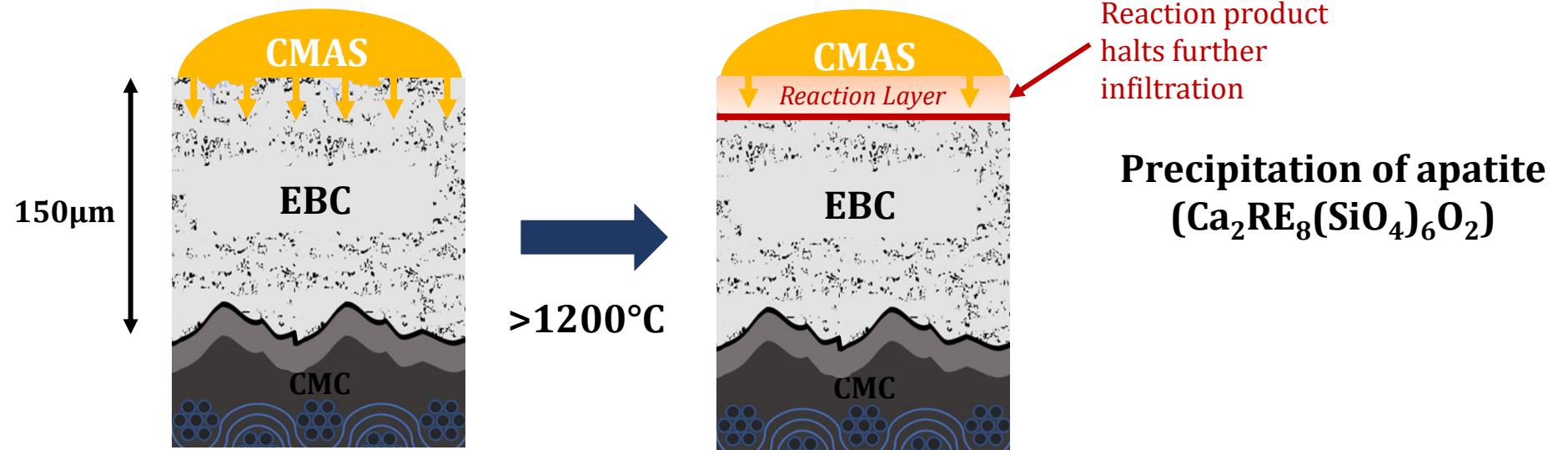
- *Minimize* reactivity of coating material with molten CMAS
  - Thermodynamic stability over reaction products





# CMAS Mitigation Strategies

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



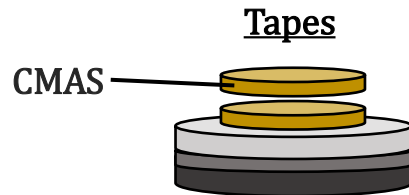
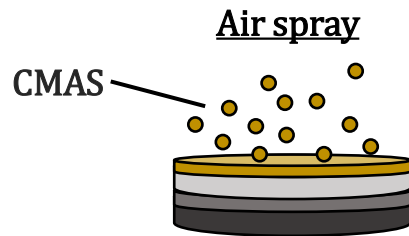




# Degradation by Particulate Ingestion

## Testing Methods

### Static CMAS Testing



Heat treatment in box furnace → thermodynamic assessment of reactivity

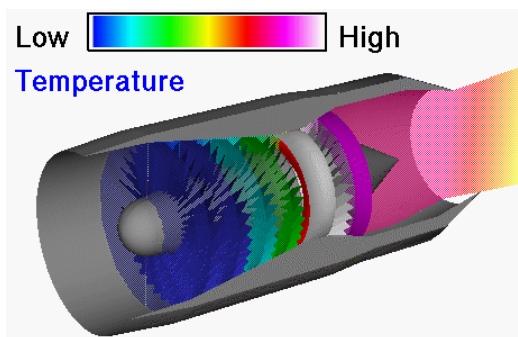


# Degradation by Particulate Ingestion

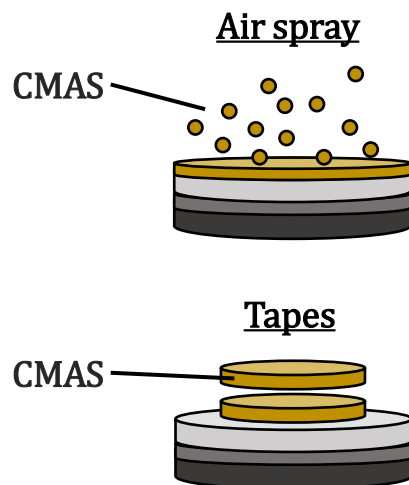
## Testing Methods

Engine Environment

Dynamic, Non-equilibrium

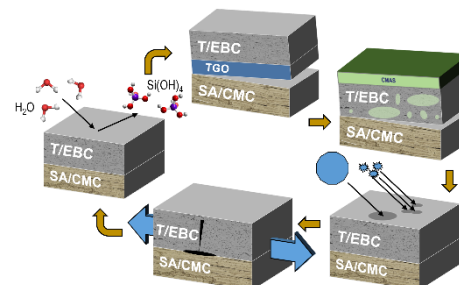


Static CMAS Testing



Heat treatment in box furnace → thermodynamic assessment of reactivity

Synergistic → Thermal cycling, gradients, water vapor combined will all affect coating durability



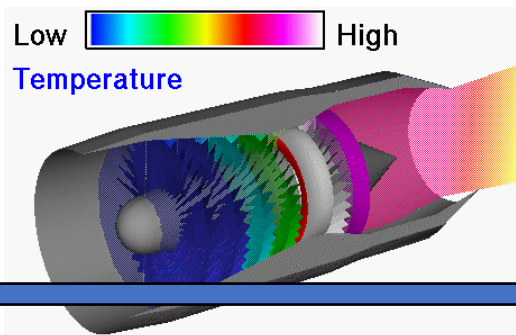


# Degradation by Particulate Ingestion

## Testing Methods

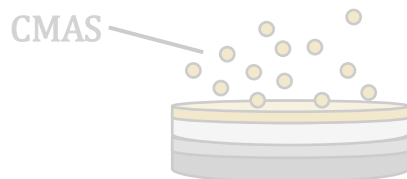
Engine Environment

Dynamic, Non-equilibrium

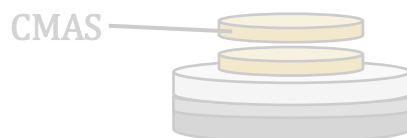


Static CMAS Testing

Air spray

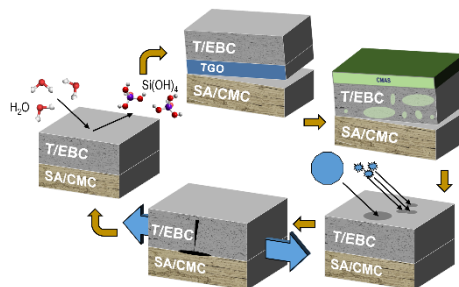


Tapes



Heat treatment in box furnace → thermodynamic assessment of reactivity

Synergistic → Thermal cycling, gradients, water vapor combined will all affect coating durability



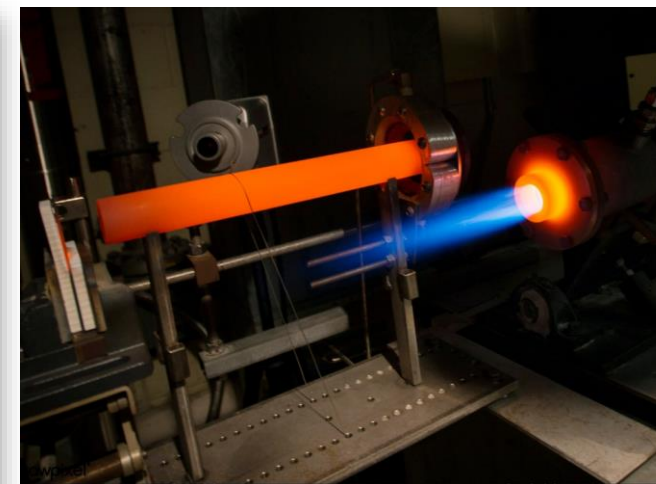
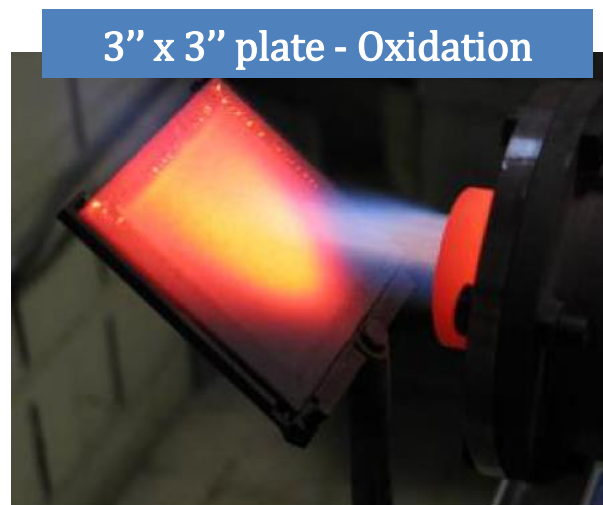
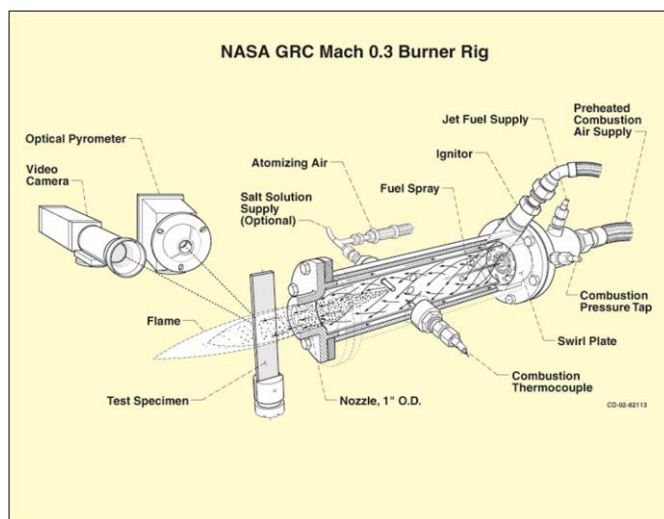
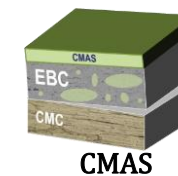
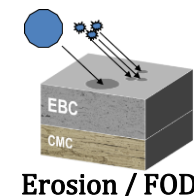
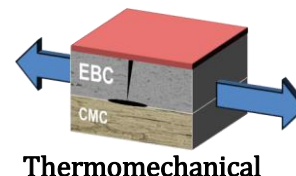
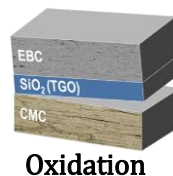
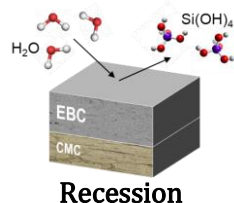
- Laboratory scale testing in relevant environments
  - Induce coating damage observed in flight hardware
  - Standardize testing procedures for high temperature evaluation of particle interactions



# Mach 0.3-1 Jet-A Burner Rig Facility

## Jet-A Burner Rig

$P(\text{H}_2\text{O}) = 0.1 \text{ atm}$   
 $V \sim 100 - 340 \text{ m/s}$   
 $P_{\text{total}} = 1 \text{ atm}$



- Sample temperature:  $\sim 600 - 2700^\circ\text{F}$  ( $\sim 315 - 1482^\circ\text{C}$ )
- Mach 0.3 – Mach 1.0 gas velocity ( $\sim 100 - 340 \text{ m/s}$ )
- $\sim 10\%$  water vapor (atmospheric pressure – 1atm)

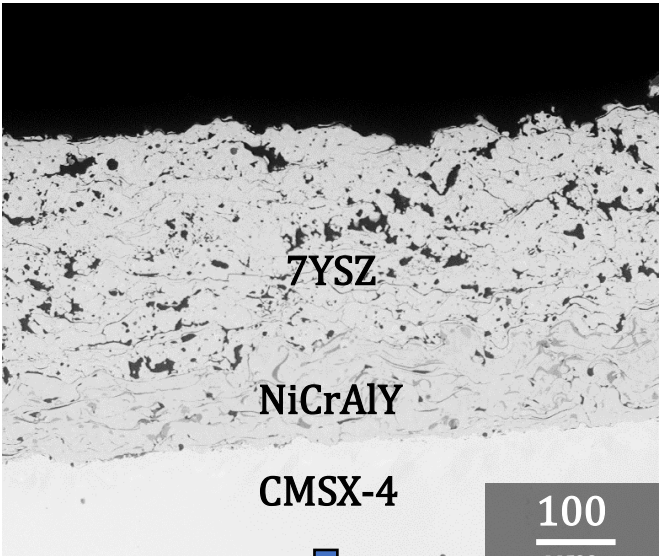
- Jet-A/pre-heated air burner rig
- Coupon to sub-component scale testing
- Multiple mechanisms can be studied simultaneously





# Degradation by Particulate Ingestion

## 7 wt.% Ytria-Stabilized Zirconia TBC

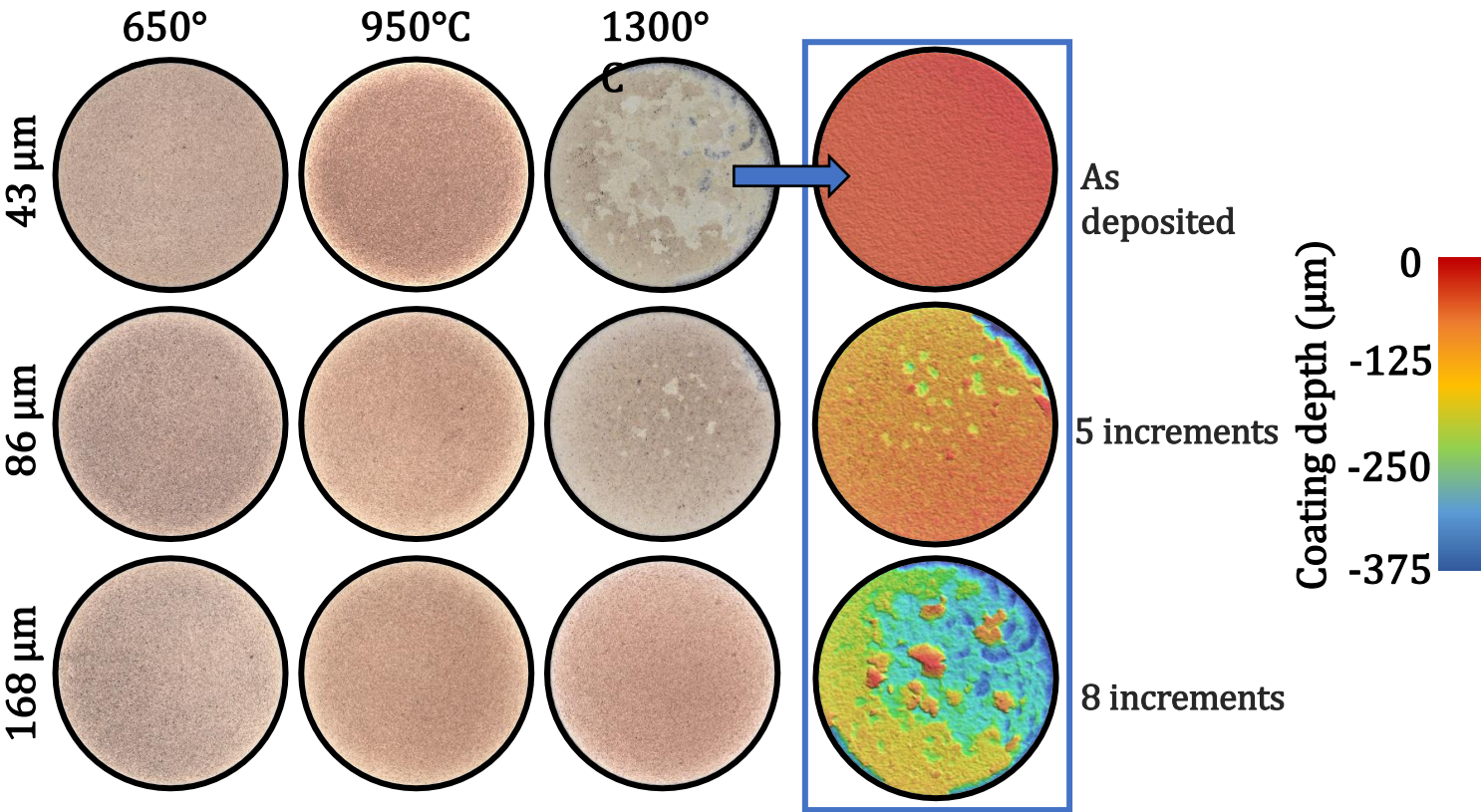


### Erosion Burner Rig Facility

- Jet-A combustor
- Variable fuel air ratio to achieve different temperatures
- 30.67CaO-8.25MgO-12.81AlO<sub>1.5</sub>-48.27SiO<sub>2</sub> (mol.%) glass frit melts at ~1240°C

### SOA Air Plasma Sprayed ~250 μm 7YSZ TBC

- CMSX-4 superalloy (25.4 mm diameter)
- Particle degradation well documented in literature



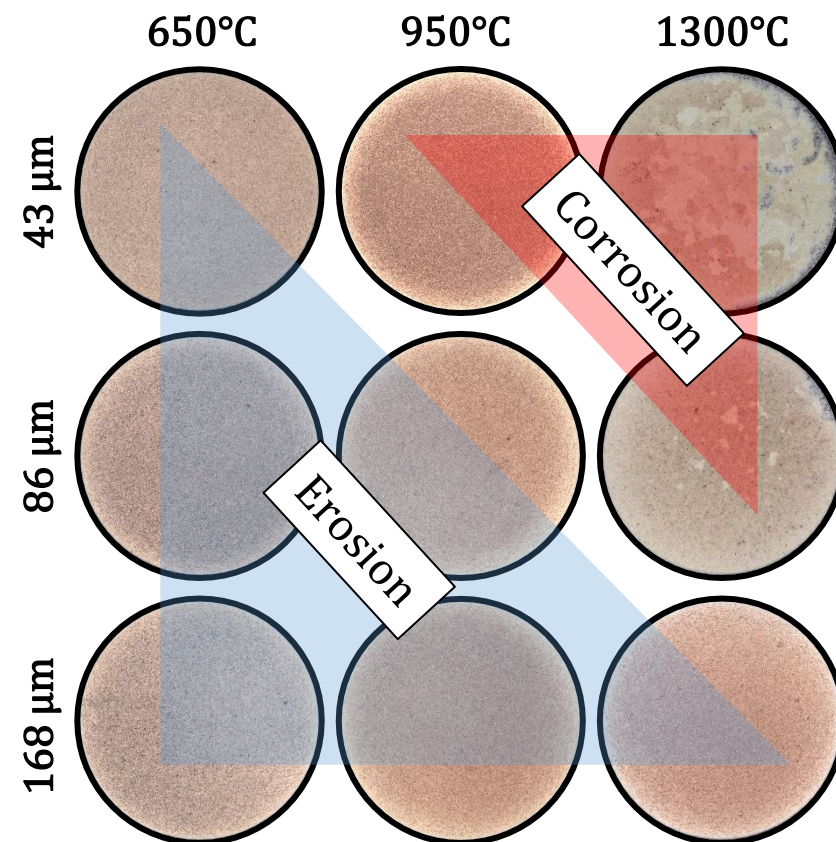


# Degradation by Particulate Ingestion

## Erosive vs Corrosive Behavior

### Effects of Erodent Material

- Define erosion and corrosion as dominant regimes within a set of conditions used for testing.
  - All 168  $\mu\text{m}$  samples exhibited erosion-dominated behavior (i.e. consistent mass loss).
  - Flow through the burner at 1300°C was rapid enough to not produce fully molten particles at this particle size range.
- 43  $\mu\text{m}$  particles became fully molten at 1300°C, resulting in deposition and reaction with 7YSZ
- Varying CMAS compositions will result in shift of regimes



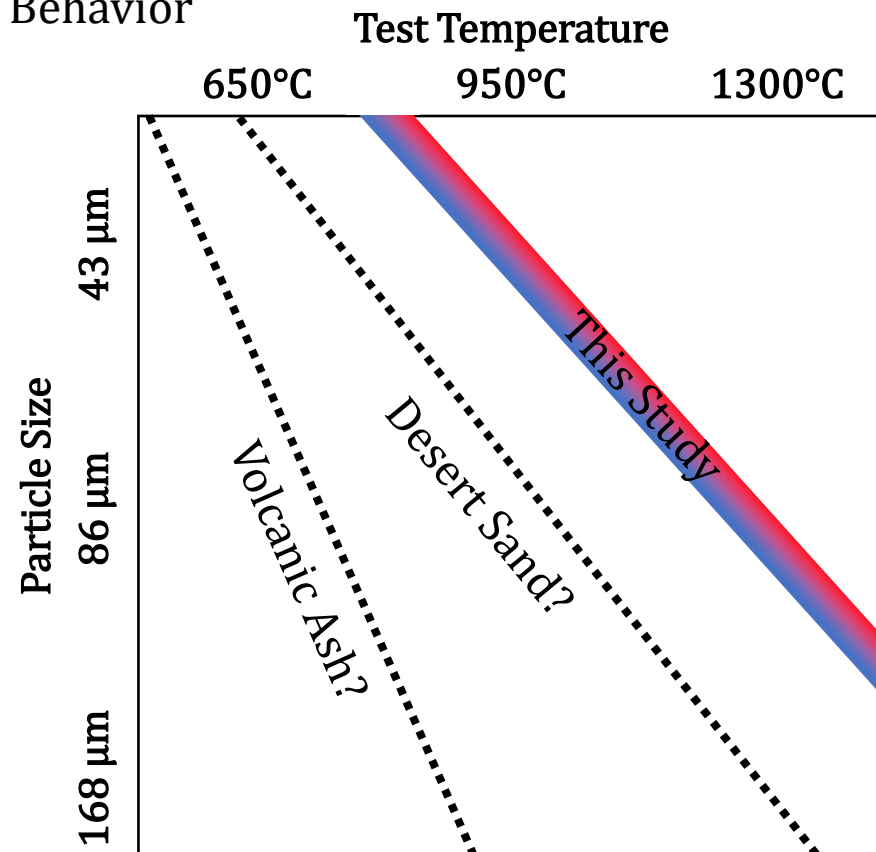


# Degradation by Particulate Ingestion

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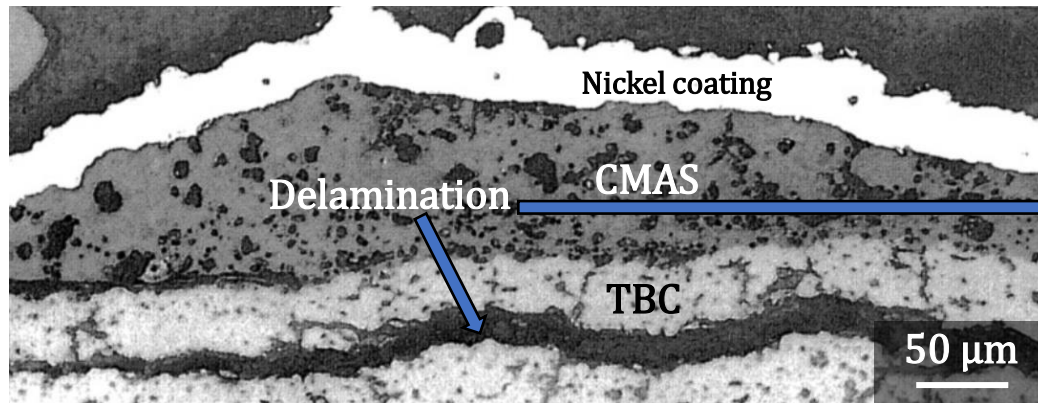
# Degradation by Particulate Ingestion

## Mechanisms of Degradation

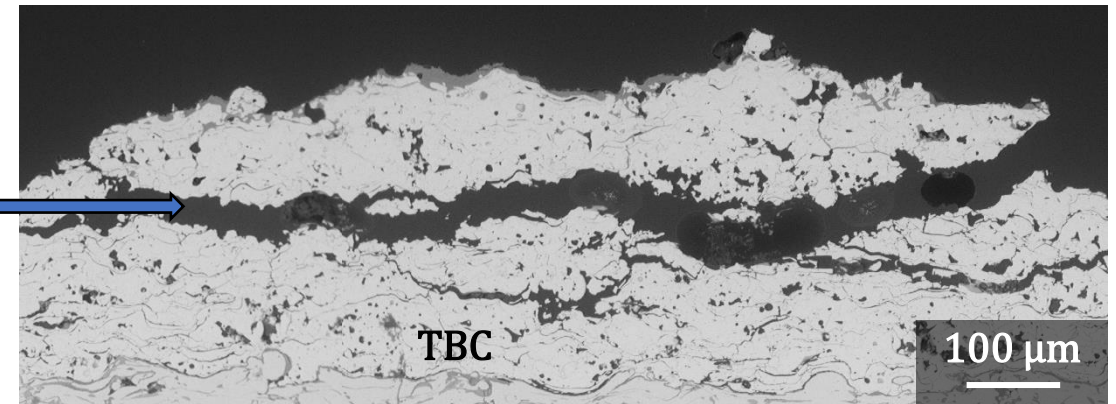
### Burner Rig Testing: Comparison to Furnace Tests and In-Service Components

- In static furnace testing, CMAS is observed to completely infiltrate YSZ coatings
- Limited CMAS infiltration was observed in burner rig testing in remaining coating
- Thermal gradient and thermal cycling cause progressive exfoliation of layers of YSZ upon cooling

#### In Service Component



#### Burner Rig



Borom et al., *Surface and Coatings Technology* 86-87 (1996)





# Conclusions

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- Incorporation of new material systems such as ceramic matrix composites (CMCs) offer potential for higher efficiencies and reduced emissions but require environmental barrier coatings for use in next generation turbine engines.
  - High temperature corrosion via the oxidation of the silicon bond coat or SiC substrate is a critical mechanism that needs to be slowed for long-life EBCs and will be of even more important at temperatures  $>2400^{\circ}\text{F}$  ( $1316^{\circ}\text{C}$ ).
  - Recession via the formation of  $\text{Si}(\text{OH})_4$  needs to be suppressed as much as possible for long-life / durability.
  - CMAS may be 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.
  - Overall, design of EBCs requires a knowledge of both chemistry and physics to design for long-life and durability in the extreme environment of a turbine engine.
-

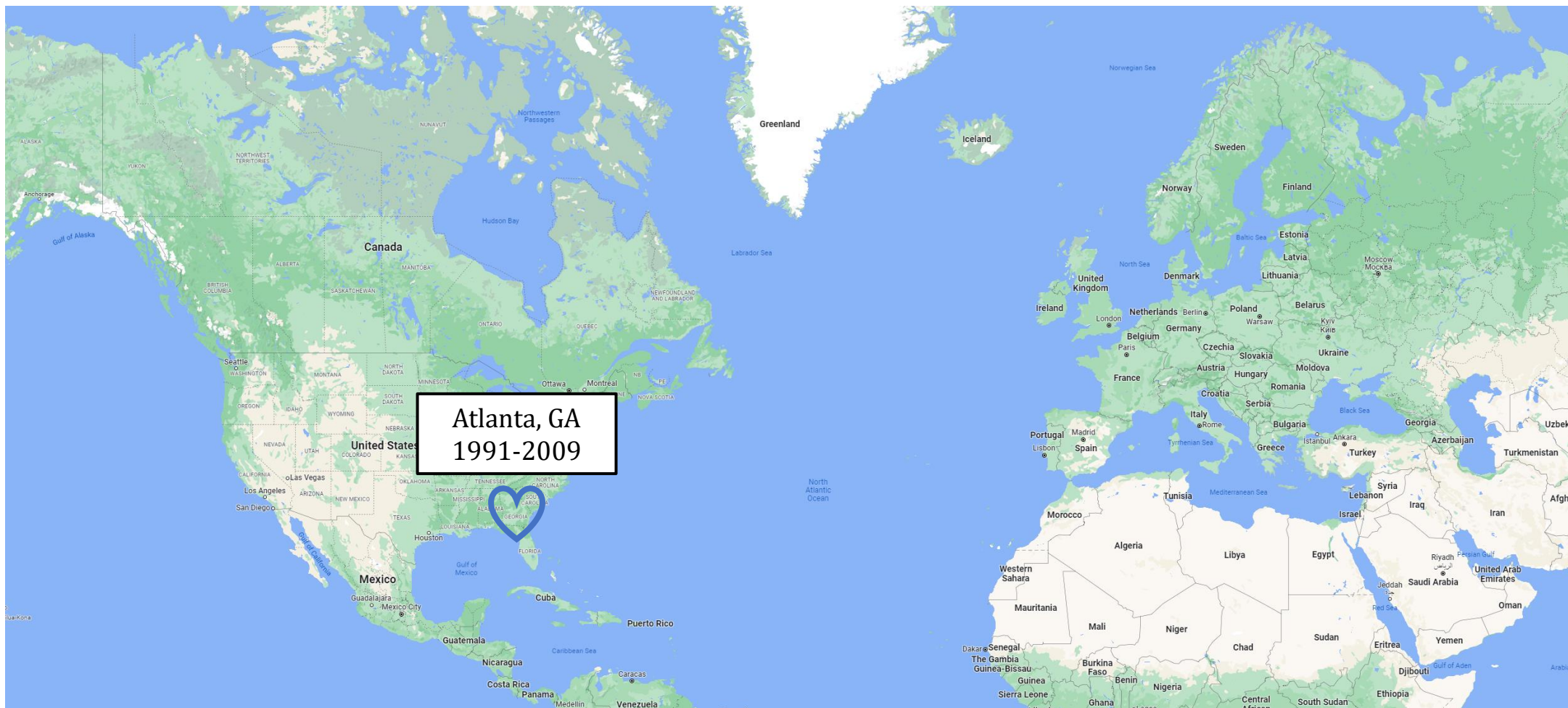


*[intern.nasa.gov](https://intern.nasa.gov) – Internships, fellowships and scholarship opportunities*



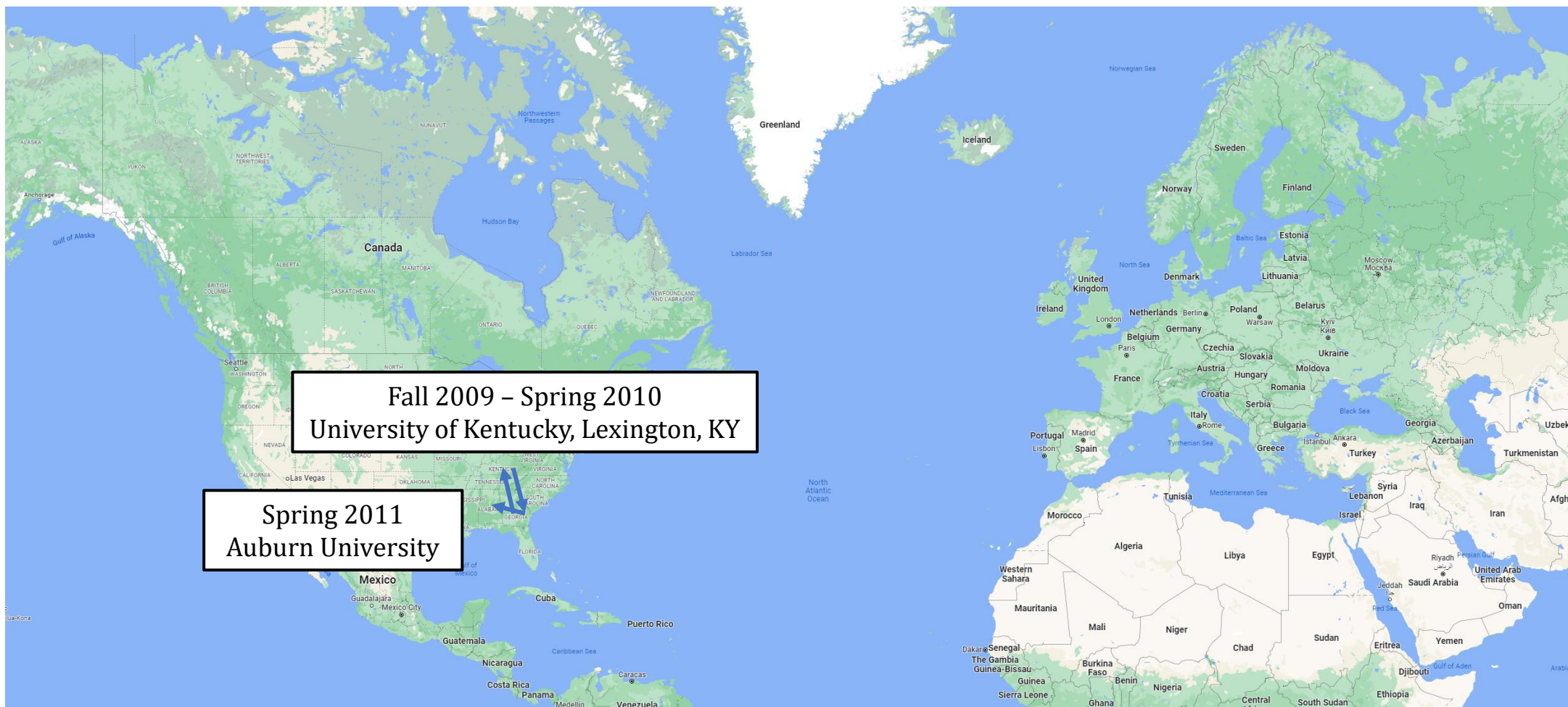


# About Me!





# About Me!





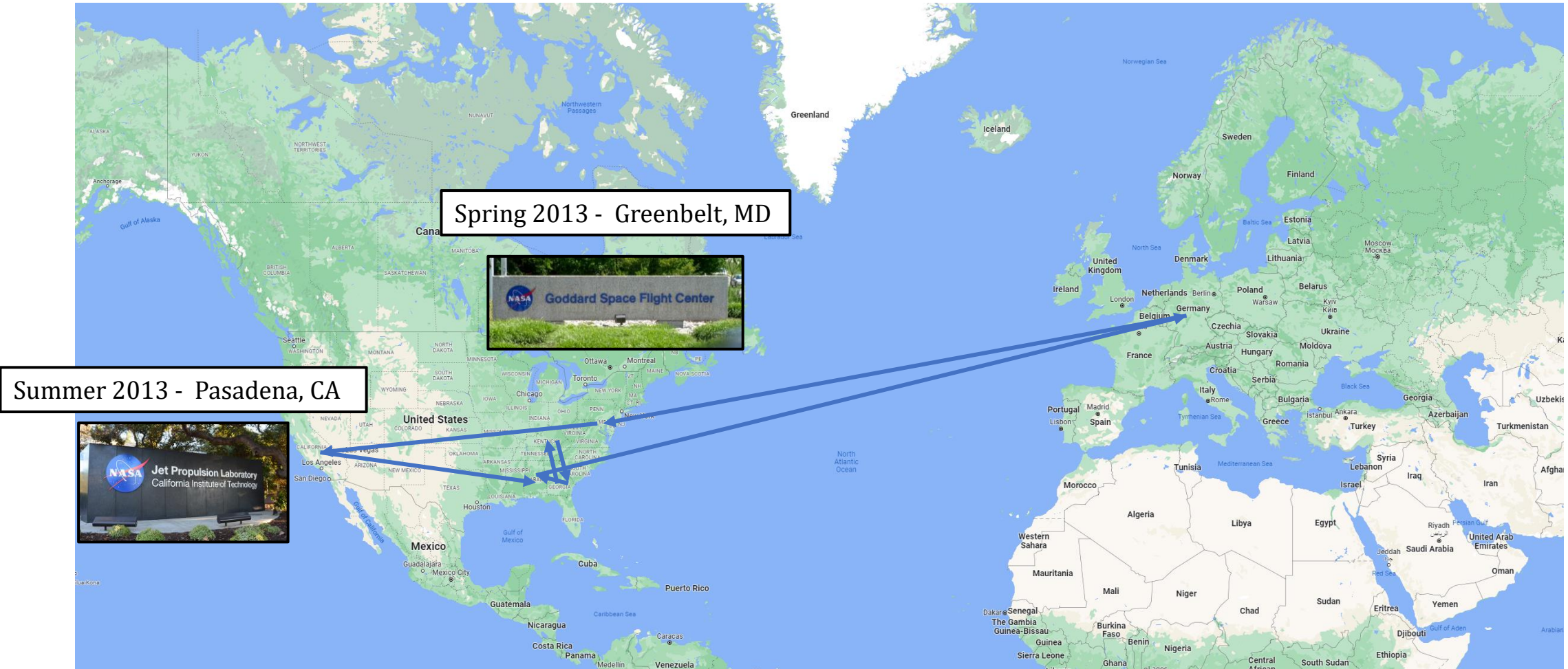


# About Me!





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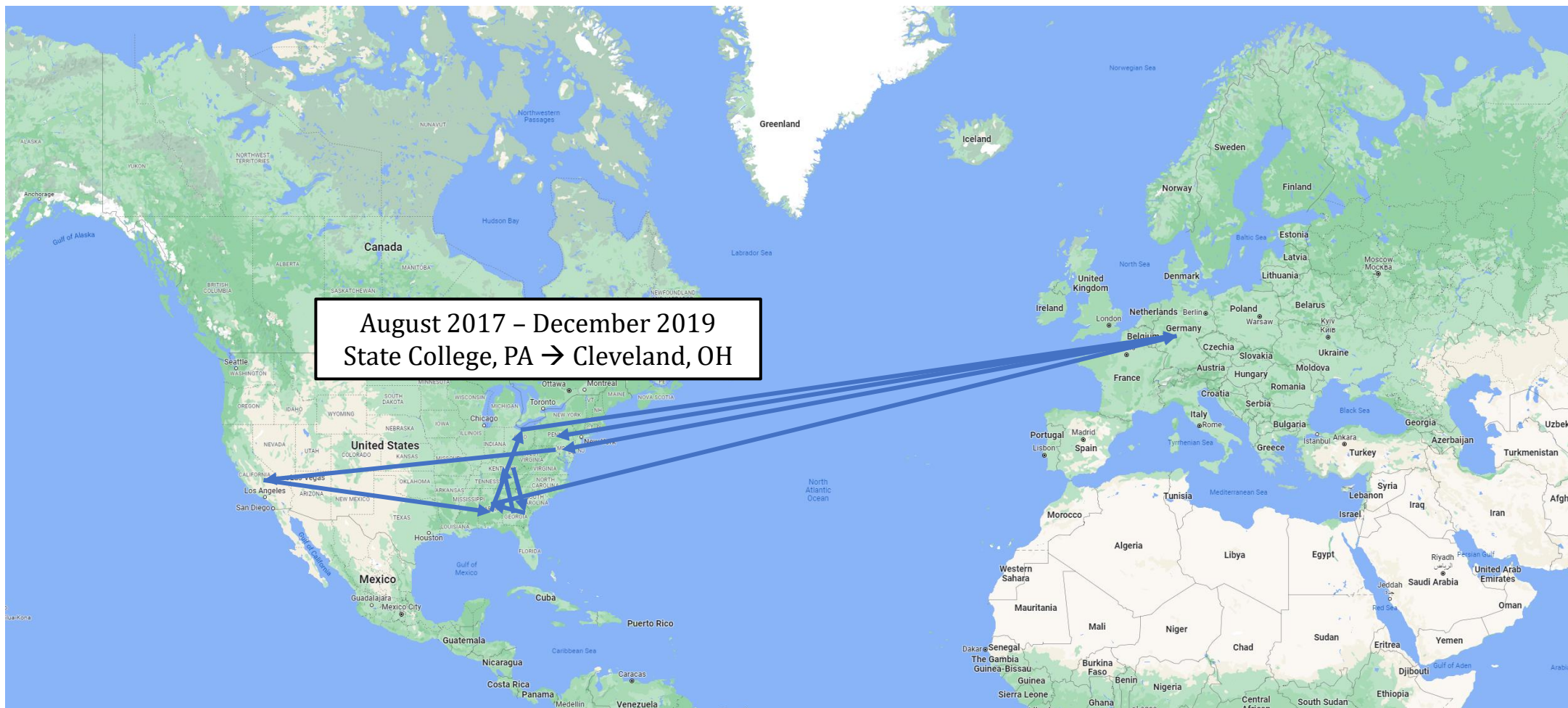
# About Me!

August 2015 – December 2019  
State College, PA





# About Me!







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