Environmental Testing Of High Temperature Materials and Coatings

Craig Robinson
Chief, Environmental Effects and Coatings Branch
NASA Glenn Research Center, Cleveland OH 44135

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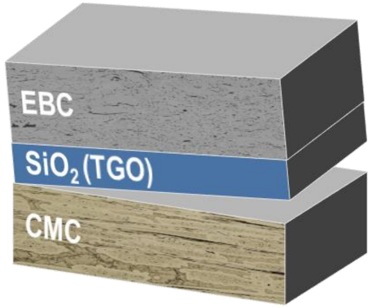
Introduction

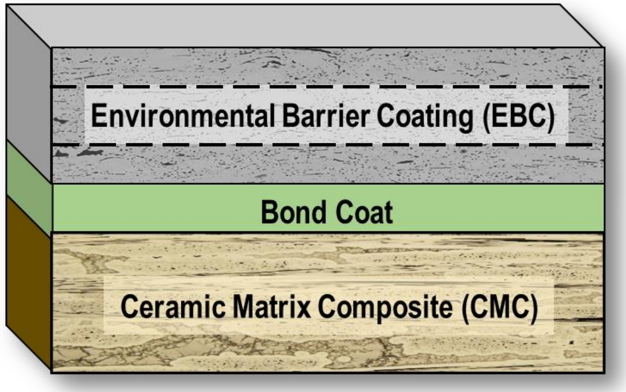
- Today′s materials for extreme environments have unique challenges and design requirements that require multi-faceted evaluations.

- Thermo−mechanical behavior is obviously important, but environmental effects are often the limiting factor and root cause of failure of materials in service.
  - Degradation modes such as oxidation, recession, erosion, foreign object damage, and chemical corrosion can negatively impact the materials thermo-mechanical behavior.
  - Evaluation of materials systems under relevant conditions are critical…..start by identifying mechanisms and develop corresponding test methods.
  - Comprehensive modeling begins with thermo-mechanical behavior, then considers environmental degradation modes that provide a “knock down” of that behavior.

Environmental Barrier Coating & Failure Modes

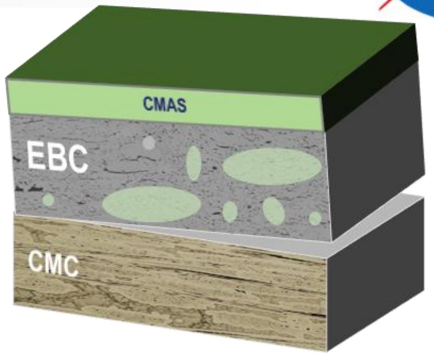


Steam Oxidation



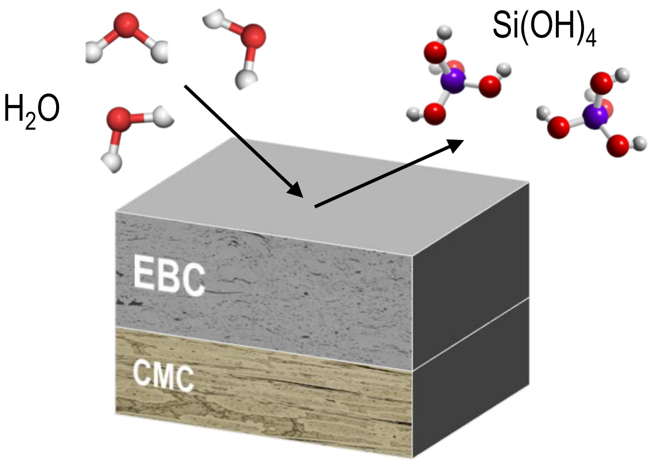
TC: Barrier from environment

BC: Bonding, adhesion, oxidation resistance

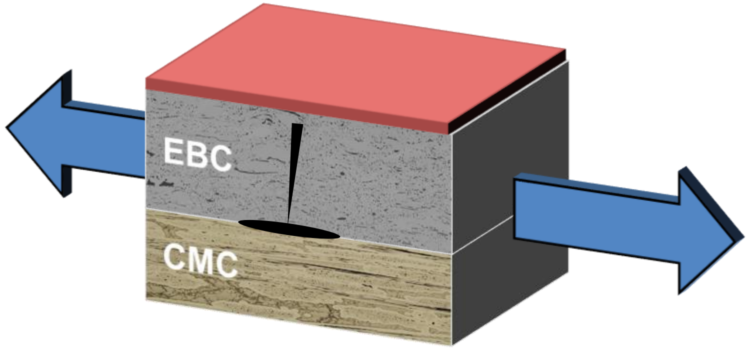


CMAS Attack & Infiltration
(Calcium-Magnesium-Alumino-Silicate)

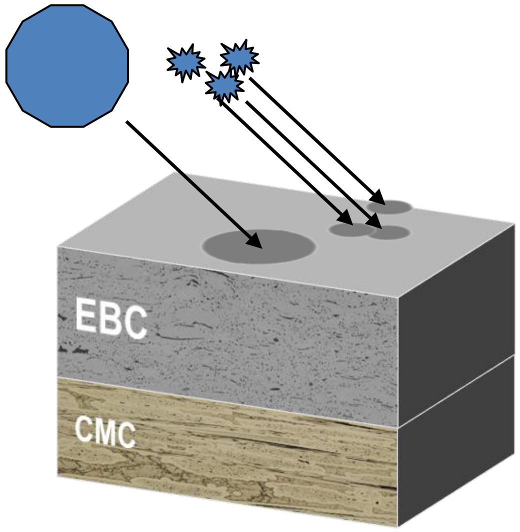
Individual mechanisms must be well understood before evaluating combined effects
Synergies between failure modes determine EBC lifetime and design requirements



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)

Roadmap for EBC failure mechanism modeling

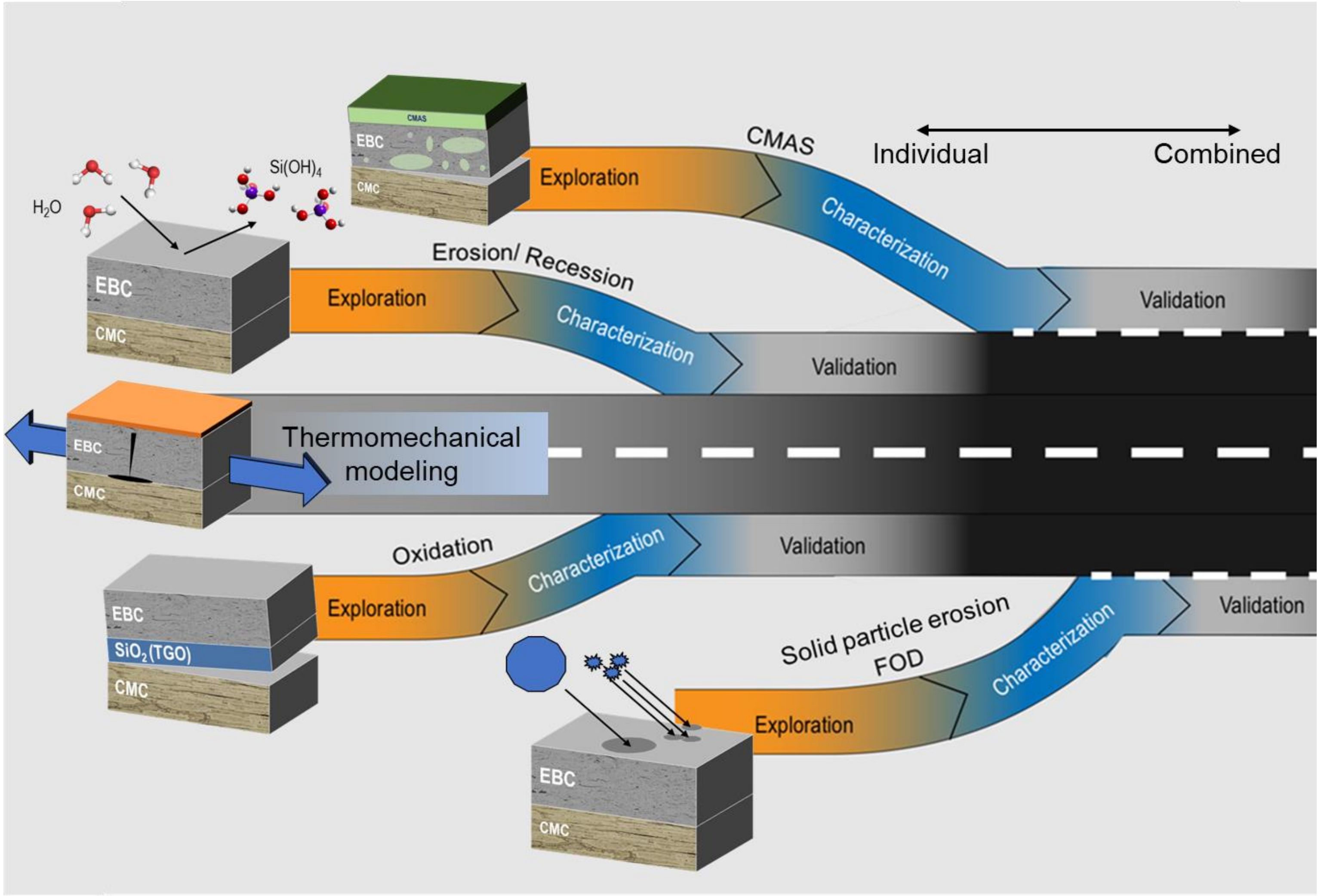
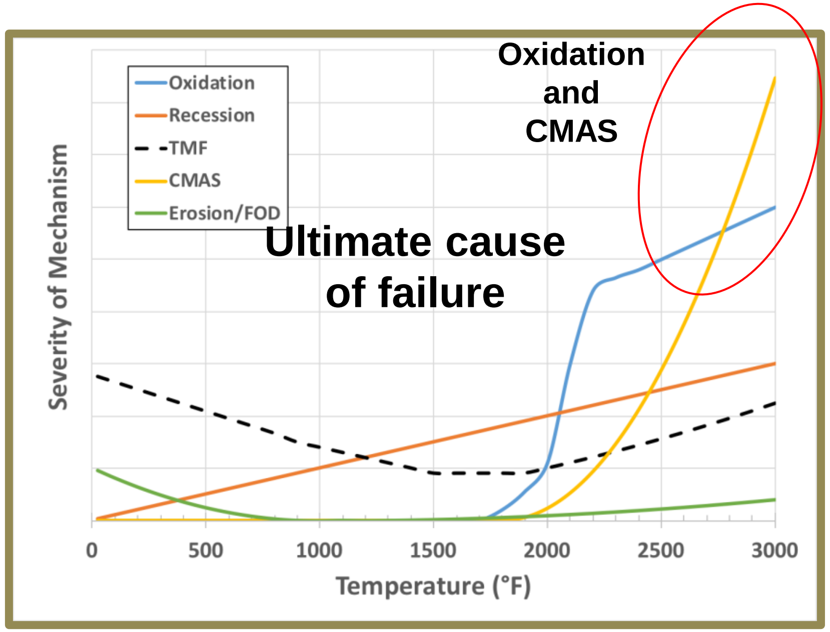
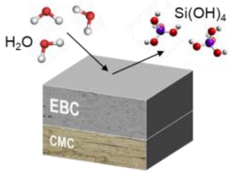


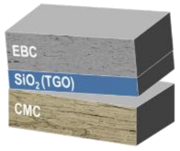
Illustration of EBC Mechanism map

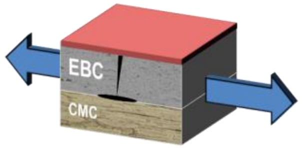


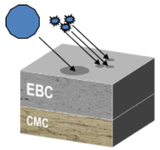
Exploration requires testing

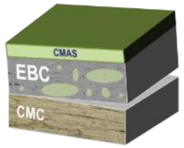
Environmental Testing Summary

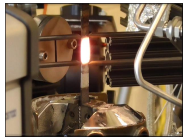
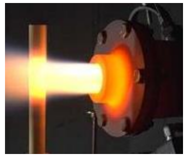
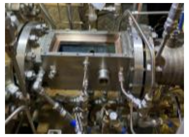








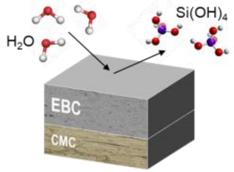


		Recession	Oxidation	Thermomechanical	Erosion/FOD	CMAS
	**Thermochemistry** $P(H_2O)$ = N/A V = N/A P_{total} = N/A	✓ (green)				✓ (yellow)
	**Steam Cycling** $P(H_2O)$ = 0.9atm V = 10 cm/s P_{total} = 1 atm	✓ (yellow)	✓ (green)			✓ (yellow)
	**High Heat Flux Laser** $P(H_2O)$ = 0.1atm V = none P_{total} = 1 atm		✓ (yellow)	✓ (green)	✓ (green)	✓ (yellow)
	**Jet-A Burner Rig** $P(H_2O)$ = 0.1 atm V ~ 100 - 340 m/s P_{total} = 1 atm	✓ (yellow)	✓ (green)	✓ (green)	✓ (green)	✓ (yellow)
	**NG/O_2 Burner Rig** $P(H_2O)$ = 0.1 - 0.5 atm V ~ 100 – 250 (est.) m/s P_{total} = 1 atm	✓ (green)	✓ (green)	✓ (yellow)	✓ (green)	✓ (yellow)
	**Combustion Rig** $P(H_2O)$ = >6 atm V ~ 30 m/s P_{total} = 10 - 30 atm	✓ (green)	✓ (green)	✓ (yellow)	✓ (yellow)	

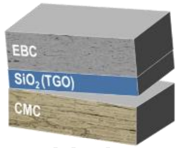
Environmental Testing - Thermochemistry

Thermochemistry

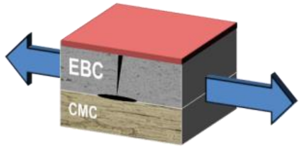
$P(H_2O)$ = N/A
V = N/A
P_{total} = N/A



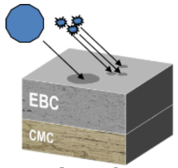
Recession



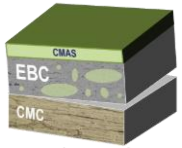
Oxidation



Thermomechanical



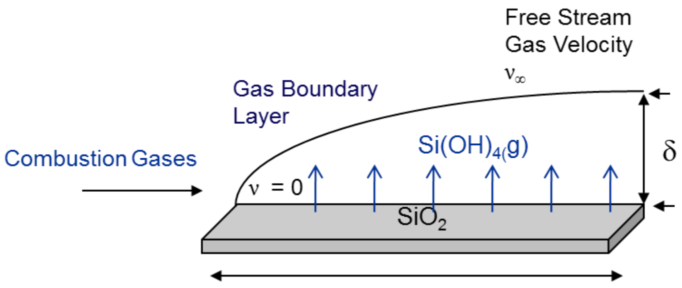
Erosion / FOD



CMAS

- Fundamental high temperature material behavior from thermodynamics, chemistry, and kinetics.
- Experimental / computational methods.
- Comprehensive suite of instruments
- Measure thermodynamic properties and identify potential gaseous species

Instrument	Measurements
KEMS Mass Spectrometry (2000˚C)	Products, activities, vapor pressure, enthalpy of vaporization
TGA (1650˚C air, 3000˚C vacuum)	Wt. change, oxidation, reduction, vaporization
DTA, DSC (2400˚C)	Enthalpy of fusion, heat capacity
Drop Calorimetry (>3000˚C)	Enthalpy of formation, reaction, and mixing
XRD, EDS, Raman (1600˚C)	Crystal structure, phase, composition, bonding



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


KEMS

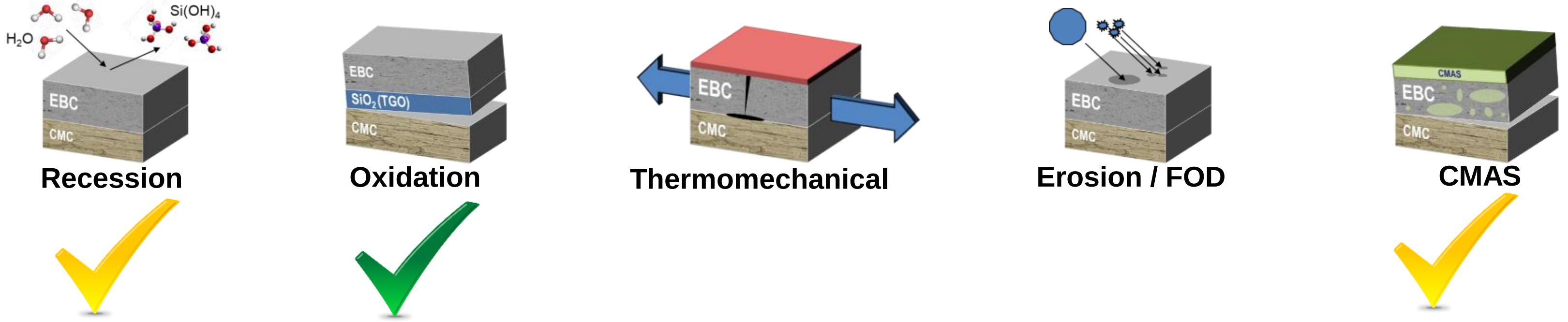

Thermo-gravimetric Analysis (air/water/vacuum)


Calorimeter

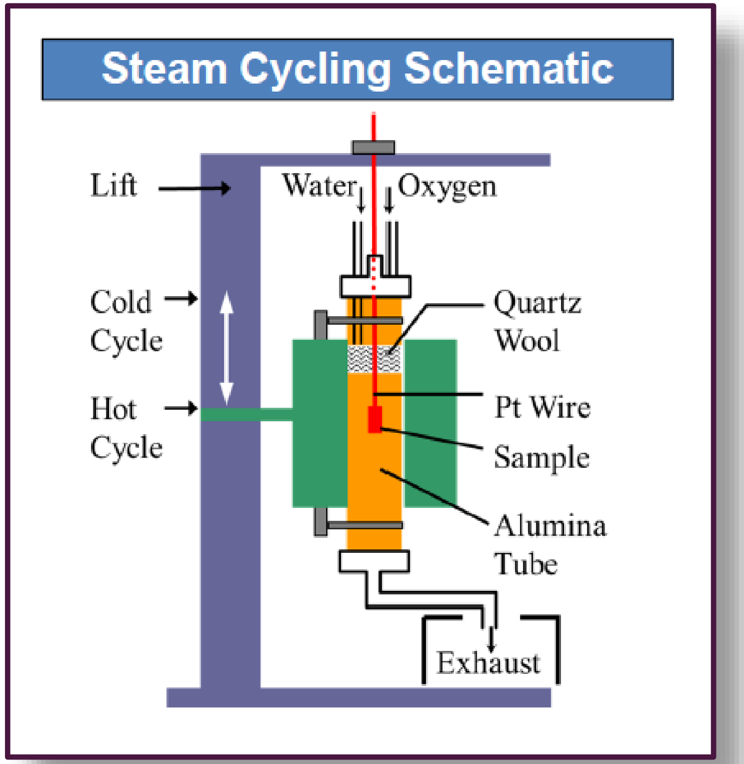
Environmental Testing – Steam Cycling

Steam Cycling

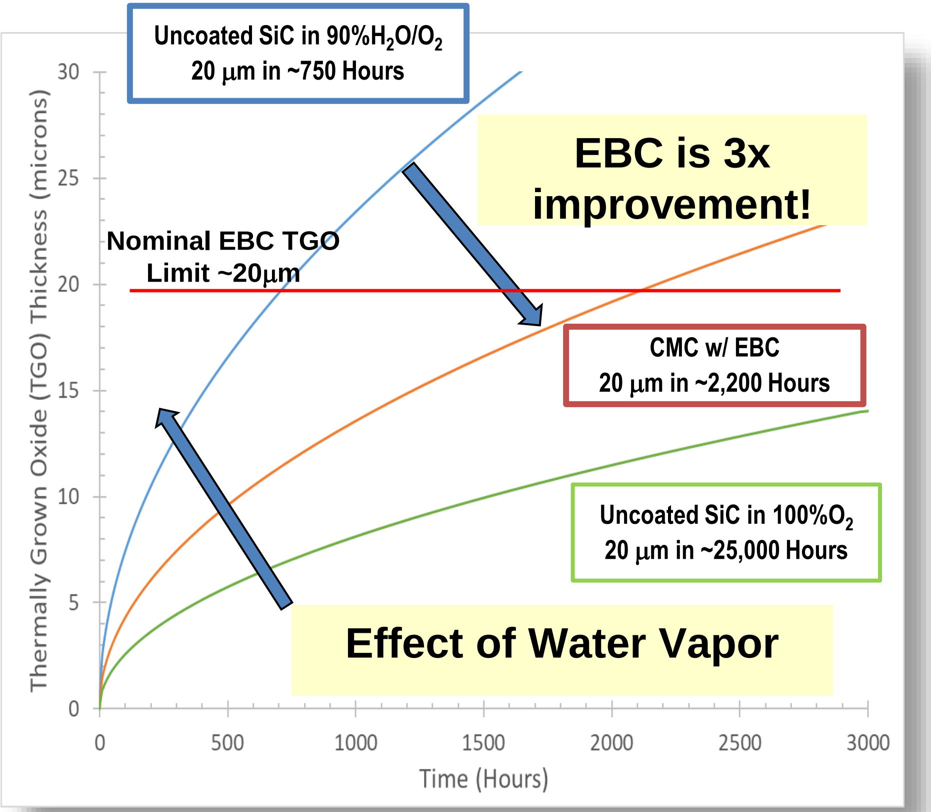
$P(H_2O) = 0.9$atm
V = 10 cm/s
$P_{total} = 1$ atm



$$x_{TGO} = \sqrt{k_p t}$$

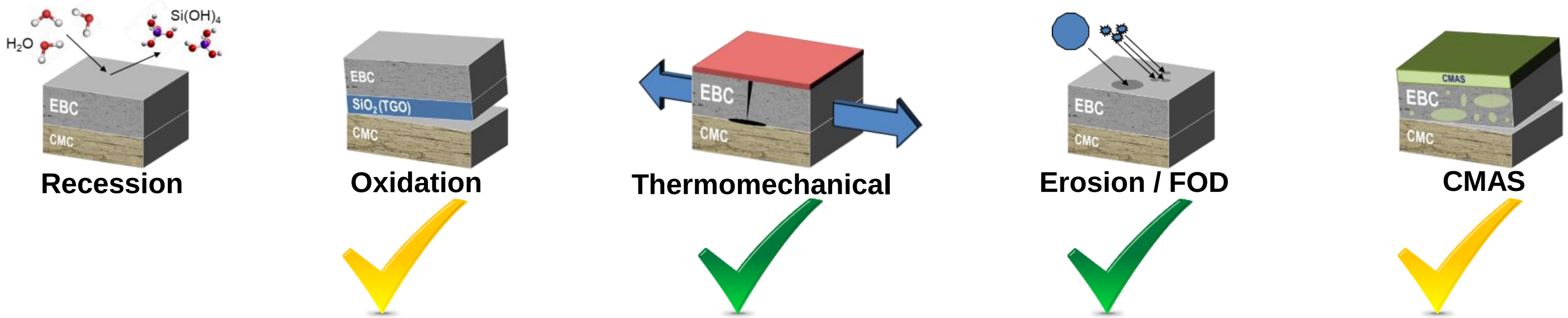


- Continuous cycling of samples at temperature forms thermally grown oxides (TGO) like SiO_2.
  - TGO is the weak EBC interface/life-limiting factor!
- Cycling in 90% H_2O/O_2 is akin to 9atm combustion environment!
- Steam oxidation provides 10x increase in TGO versus air.

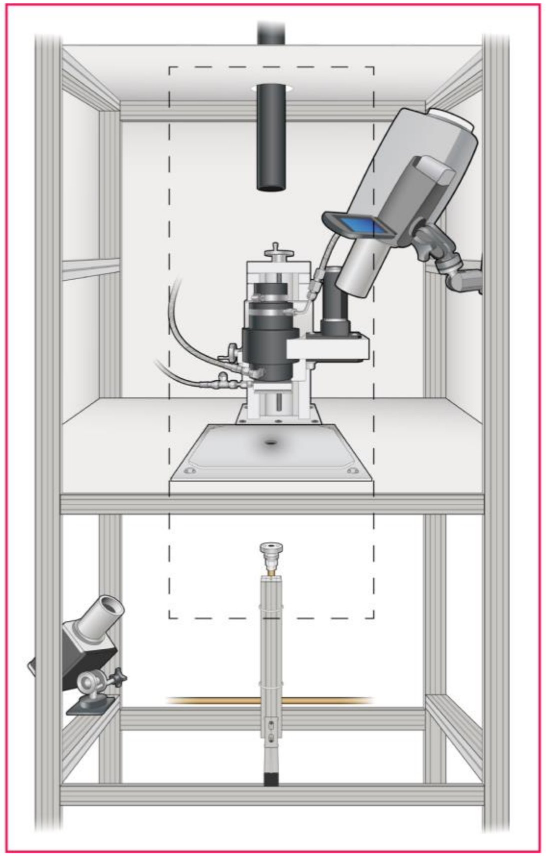


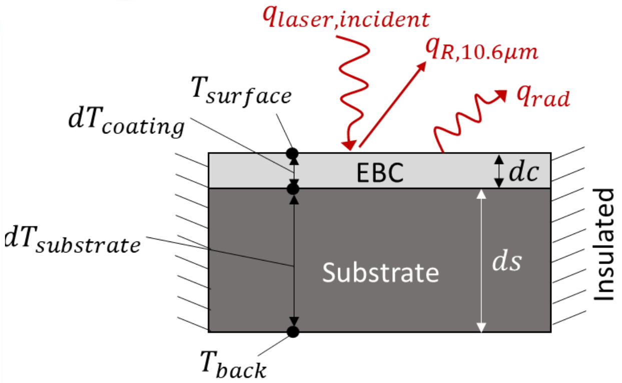
Environmental Testing – High Heat Flux Laser

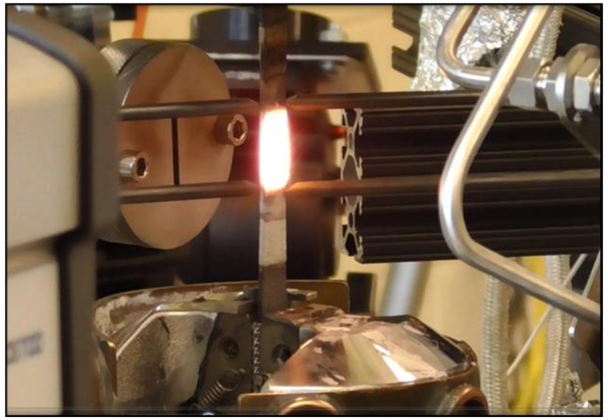
High Heat Flux Laser
- $P(H_2O)$ = 0.1atm
- V = none
- P_{total} = 1 atm



- High-powered Carbon Dioxide (CO_2) lasers
  - – 10.6µm wavelength = high absorption EBC materials
- Thermal or combined thermal-mechanical testing capabilities, backside cooling for ΔT testing
- Surface temperatures > 3,000˚ F (1650˚ C) are achievable
- Incident heat flux up to 300 W/cm² (assuming 1 inch diameter spot size)
- Heat transfer properties (thermal conductivity)



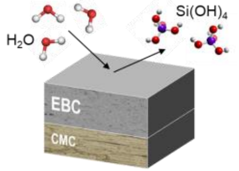




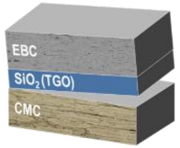
Environmental Testing – Jet-A Burner Rig

Jet-A Burner Rig

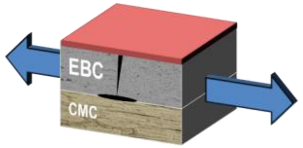
$P(H_2O) = 0.1$ atm
V ~ 100 - 340 m/s
P_{total} = 1 atm



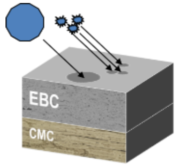
Recession



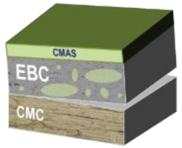
Oxidation



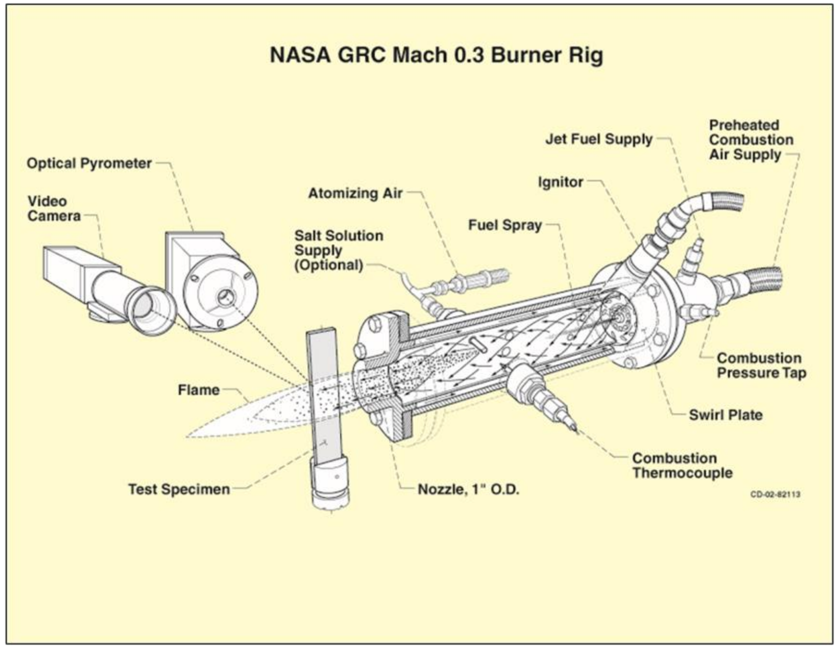
Thermomechanical



Erosion / FOD



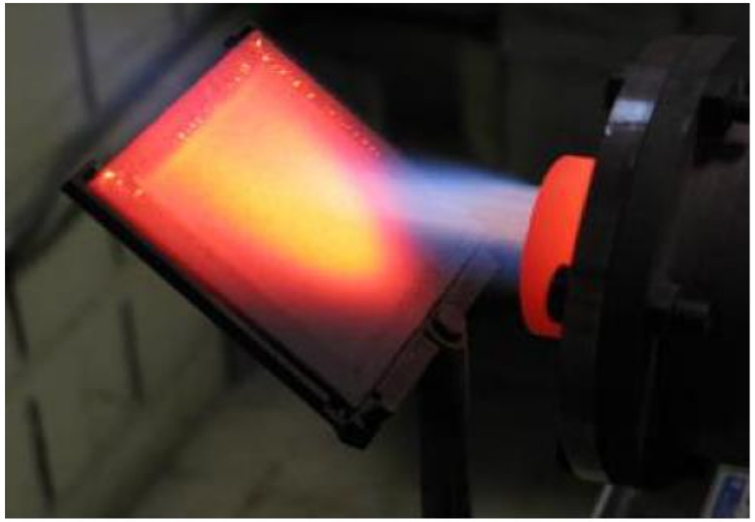
CMAS



- Simulates a variety of gas-turbine conditions
- Jet-A/pre-heated air burner rig
- Coupon to sub-component scale testing
- Multiple mechanisms can be studied simultaneously

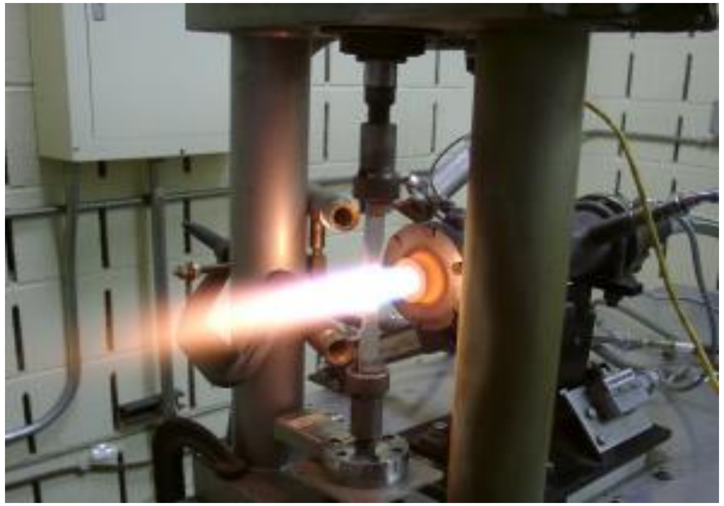
Jet-A Burner Rig – Oxidation, Corrosion, Thermomechanical

3” x 3” plate - Oxidation



Sea Salt Corrosion

Tensile Load Testing

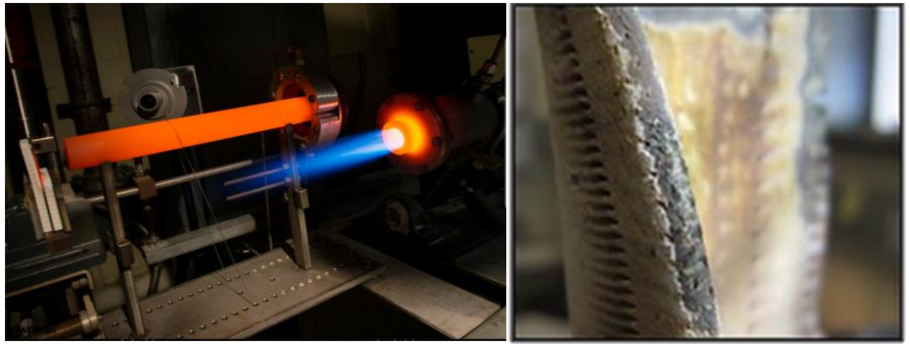


<u>Jet-A Burner Rigs</u>

- Sample temperature: ~600 - 2500˚ F
- Mach 0.3 – Mach 1.0 gas velocity (~100 – 340 m/s)
- ~10% water vapor (atmospheric pressure – 1atm)

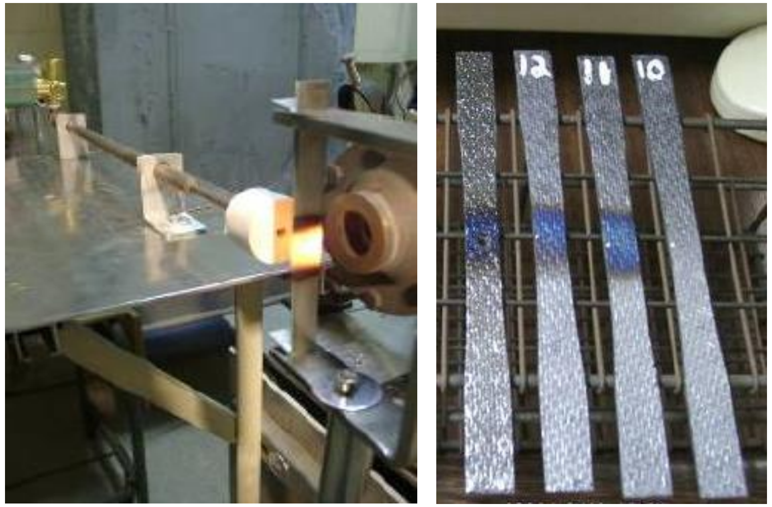
Jet-A Burner Rig – Erosion / Foreign object Damage

Solid Particle Erosion Rig **Erosion in Field**



Presby, et. al. J. Eng. Gas Turbine Power – 2020

- TBC erosion well characterized, but limited research on EBCs and CMCs.
- Rig damage observed like in-service components.



Fox, et. al. NASA/TM – 2011-216986

Erosion Testing

- Particle velocities: ~100 – 400 m/s
- Particle sizes: ~27 – 150 µm
- Eroding media: Al_2O_3
- Temperature: 1500 - 2400 ˚ F
- Impingement angle: 30 - 90˚

- Limited research on FOD for EBCs.
- EBCs protect CMCs from impact damage.

FOD Testing

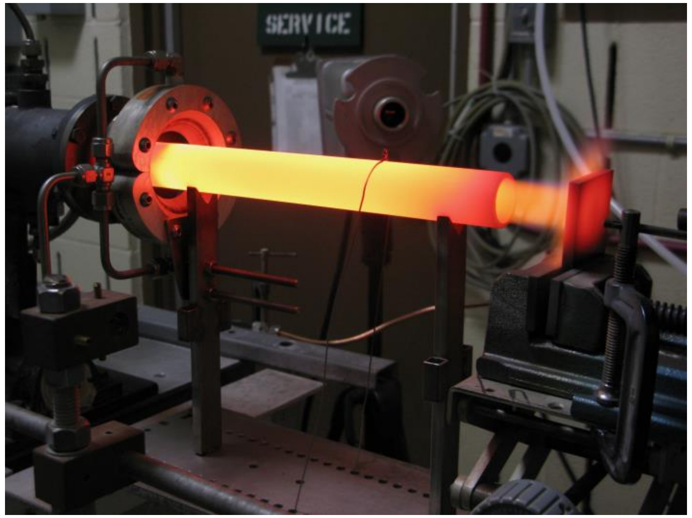
- 1/16-in. steel ball projectile
- Ambient and high temperature
- Velocity = ~100 – 400 m/s
- Impingement angle: ~10 - 90˚

Jet-A Burner Rig Facility – CMAS Attack and Infiltration

Costa, et. al. J. Am. Ceram. Soc. – 2018

- Ingested particles melt into **C**alcium-**M**agnesium-**A**lumino **S**ilicate (**CMAS**) glass at temperatures >1200ºC.
- Thermomechanical - Dissimilar CTEs and densification produce stresses.
- Thermochemical - Interactions with coatings form unwanted phases.
- Most testing uses static loadings (tapes, air-spray, etc.), better test methods needed.

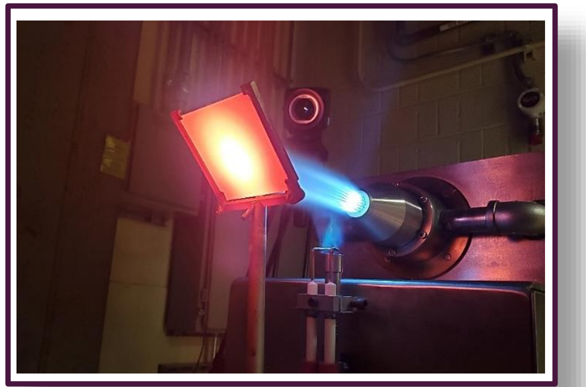
- Burner rigs a more ‘realistic’ test method for CMAS research compared to applying CMAS and melting/reacting in an isothermal environment (furnace).
- Complicated analysis – mass CMAS injected may not always equal mass CMAS deposited.
- Combined SPE and CMAS damage likely, transitions need identified.

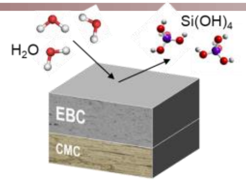


Environmental Testing - Natural Gas / Oxygen Burner Rig

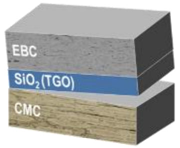
NG/O_2 Burner Rig

$P(H_2O)$ = 0.1 - 0.5 atm
V ~ 100 – 300 (est.) m/s
P_{total} = 1 atm

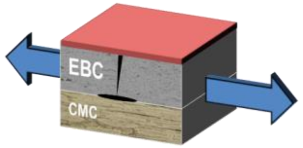




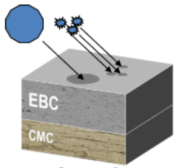
Recession



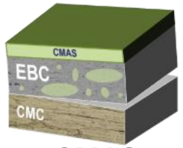
Oxidation



Thermomechanical



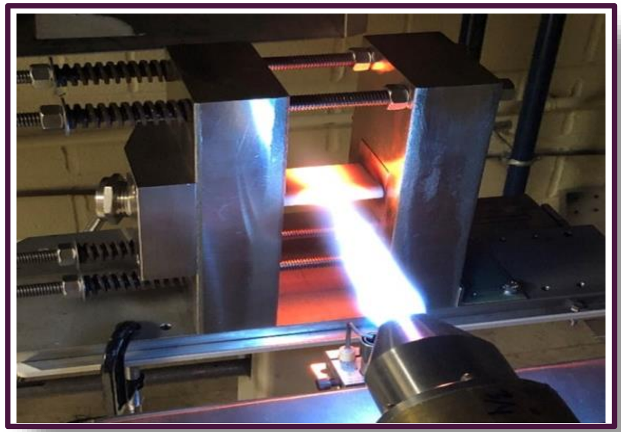
Erosion / FOD

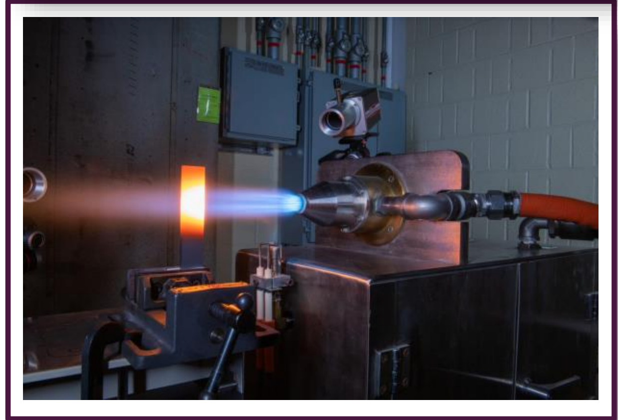


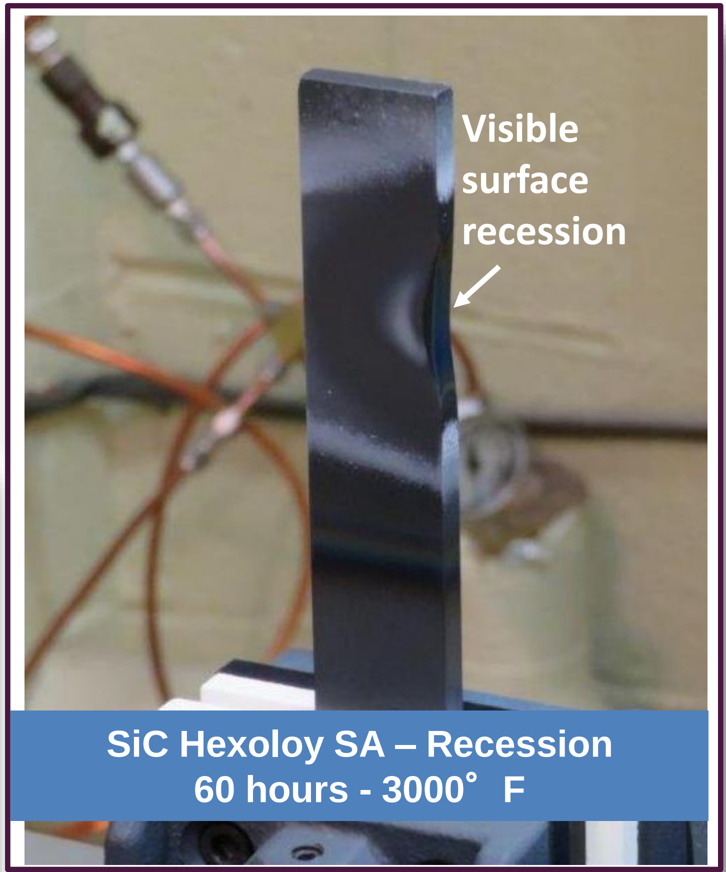
CMAS

Chemical Equilibrium Analysis (CEA)

- Peak flame temperature – 4,986˚ F
- $P(H_2O)$ ~ 0.1 – 0.4 atm → P(total) = 1atm
- Gas velocity estimated: ~100 - 300 m/s
- High heat flux (up to ~200 W/cm²)
- 1000's of hrs. at 3000F completed





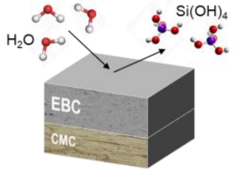


SiC Hexoloy SA – Recession
60 hours - 3000˚ F

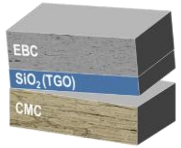
Environmental Testing – NASA Combustion Rig

Combustion Rig

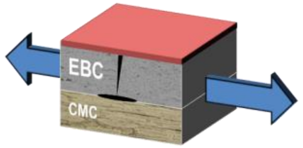
$P(H_2O)$ = >6 atm
V ~ 30 m/s
P_{total} = 10 - 30 atm



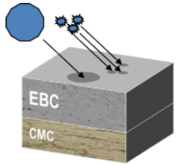
Recession



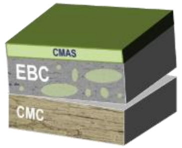
Oxidation



Thermomechanical

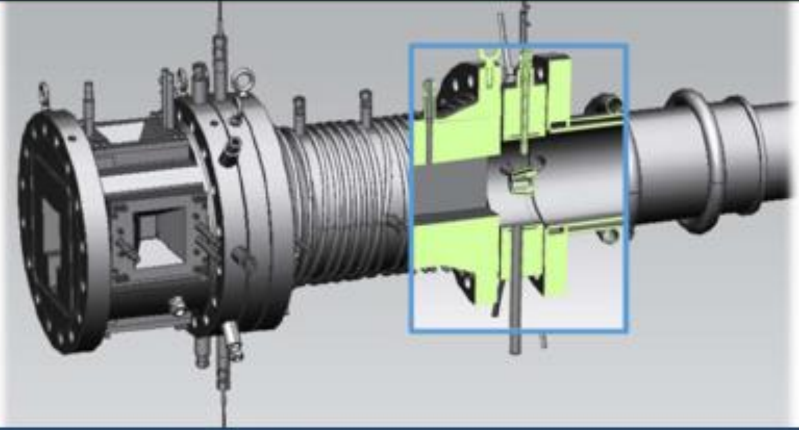


Erosion / FOD



CMAS

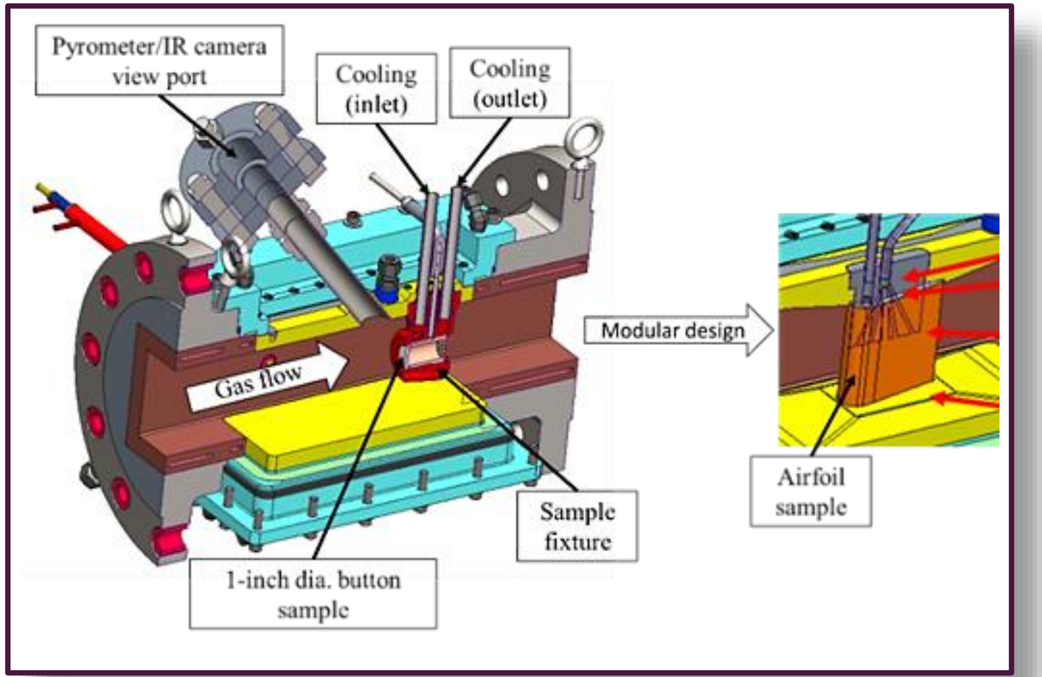
NASA GRC CE-5 Test Stand



30 atm pressure
>1650°C gas temperature
Maximum 20 lb/s Mass Flow

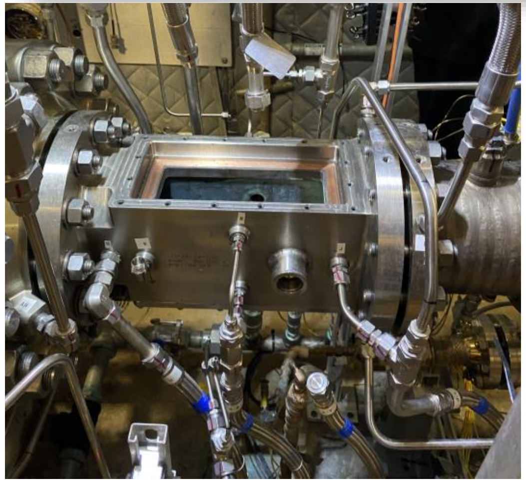
- GRC asset….collaborate with Propulsion.
- Best test for material performance with actual engine / combustion environment.
- CE-5 provides realistic environment with temperature, pressure, etc.
- Fixtures to scale testing from coupon to component level test articles.
- Expensive! … but necessary!

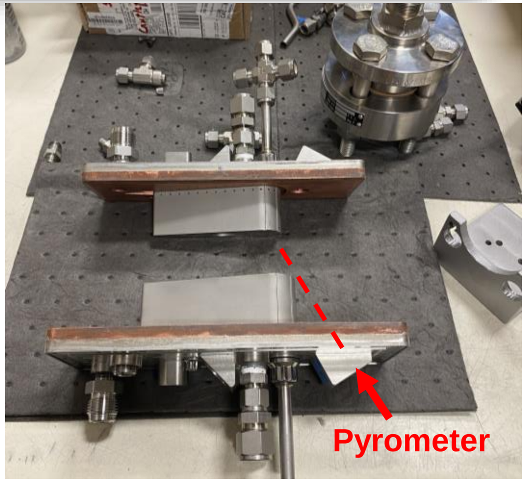
NASA Combustion Rig – Materials Test Section

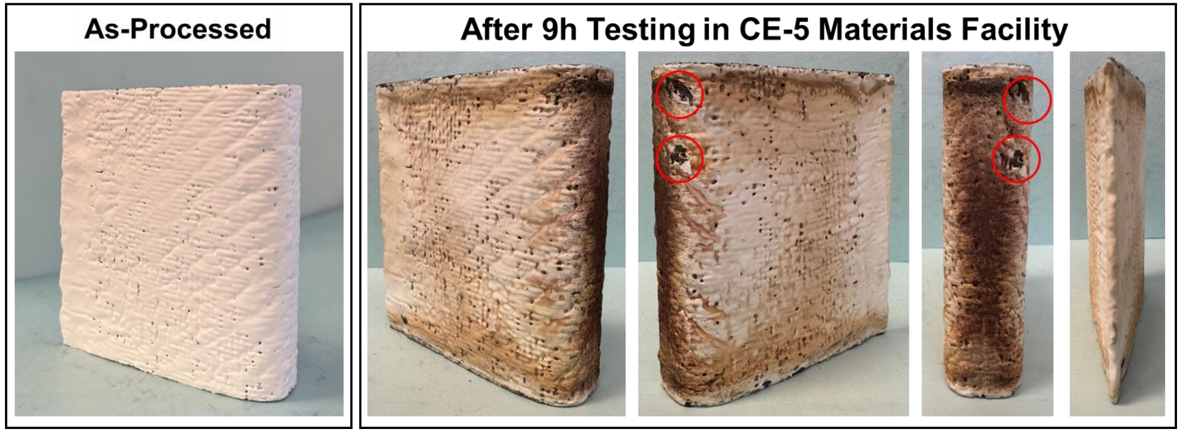


- Test Conditions
  - Combustion gas temperature: 3000^{+} ºF
  - Pressure / Velocity: 270 psi / ~40 m/s
  - Backside cooling temperature: 900 ºF
  - LE /backside/sidewall TCs, pyrometers, and IR camera

- NASA EBC/CMC systems tested at 1,650˚C (3,000˚F) EBC surface temperature, and 1,482˚C (2,700˚F) interface temperature.







Summary

- Advanced materials, such at those in ARPA-E ULTIMATE, have challenges that include thermal, mechanical, AND environmental effects.

- It is critical to identify the potential failure mechanisms for materials systems and test under relevant conditions.

- Individual mechanisms must be well understood before evaluating synergies between extrinsic failure modes that determine material lifetime and design requirements.

- NASA GRC has a complete suite of environmental test capabilities for today’s most common failure mechanisms, and open to external collaborations thru NASA Space Act Agreements.