



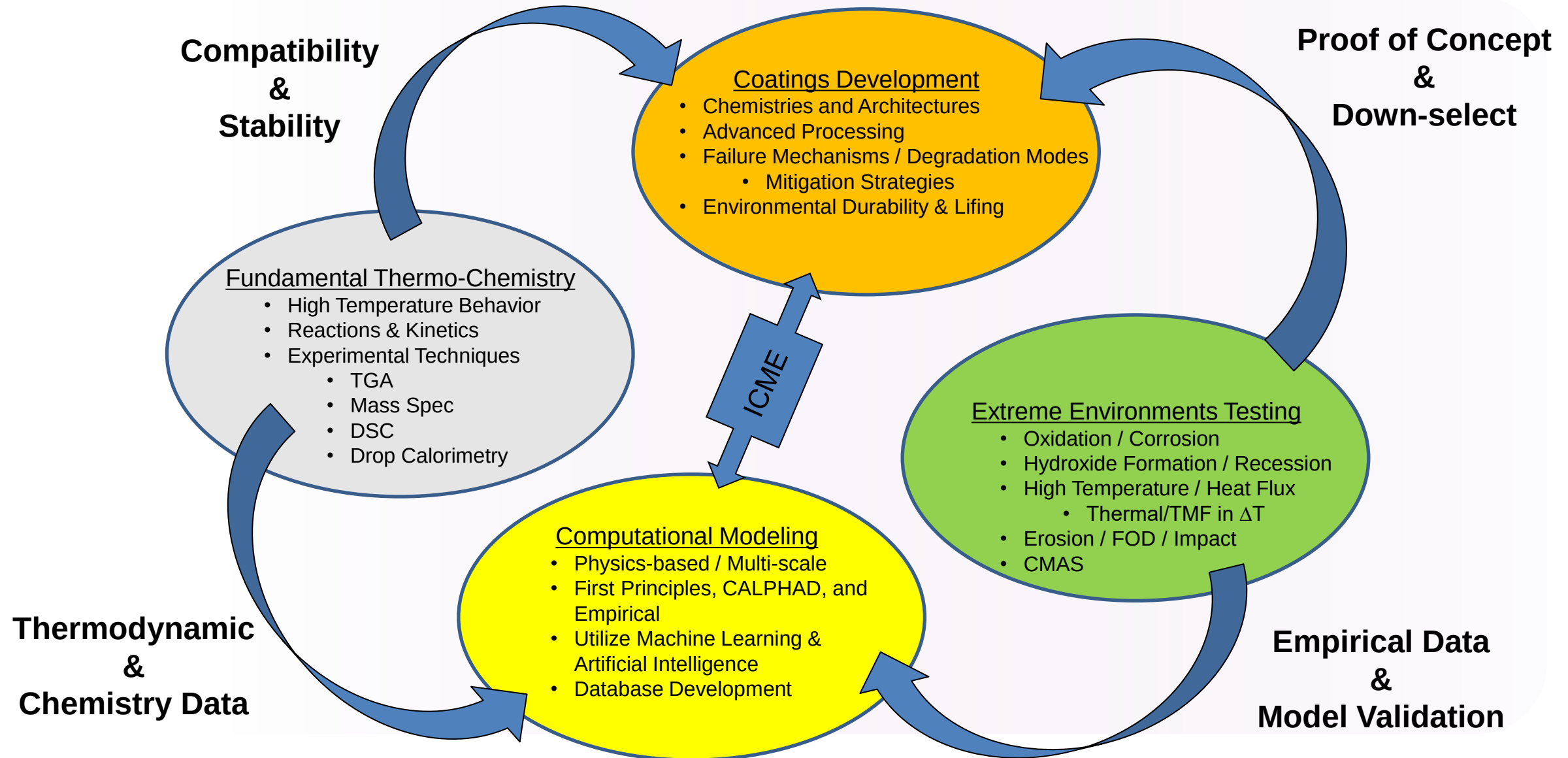
GRC High Temperature Environmental Effects and Coatings - Current Thrusts, Capabilities, Gaps

Craig Robinson

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

NASA GRC / AFRL Tech Info Mtng
April 18-19, 2024

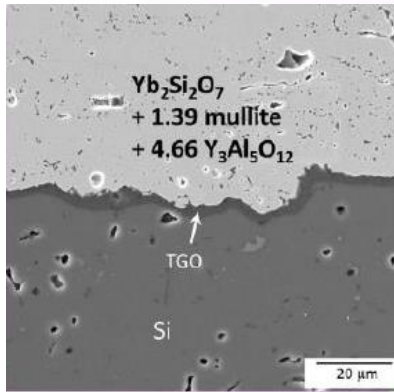
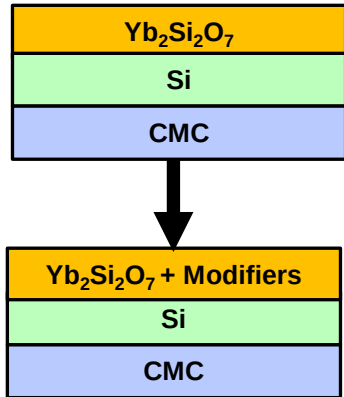
Introduction – Environmental Effects and Coatings



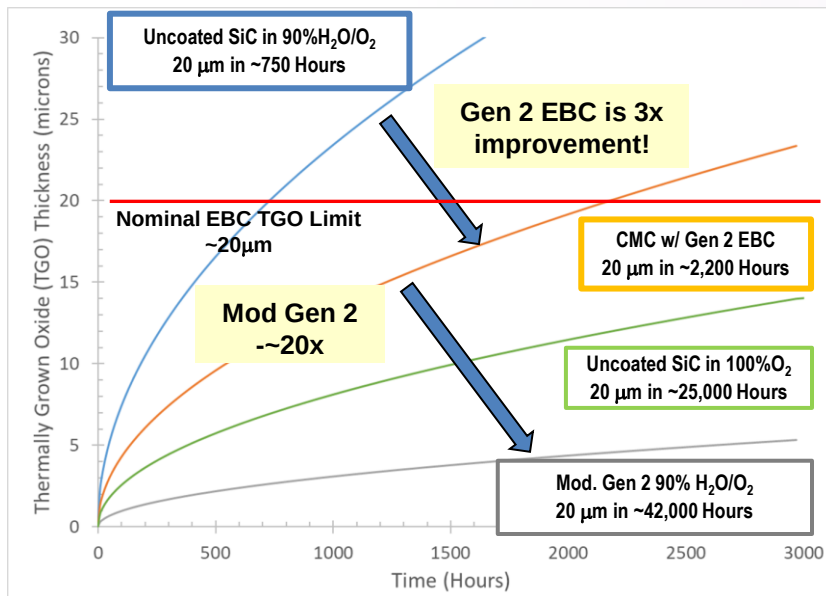
Current Thrusts – Environmental Barrier Coatings (EBCs)



Mod Gen 2 – 2400 F EBC



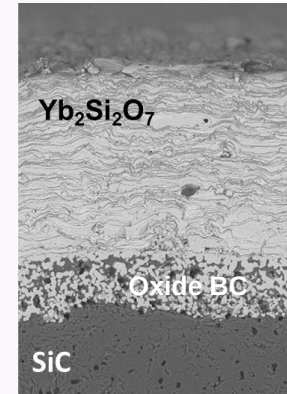
- 2400 F (1316 C) EBCs with Si bond coats – air plasma sprayed (APS)
- Modified coating chemistry (doping of YbDS w/ Al_2O_3) improved oxidation performance by reducing the TGO ~80%



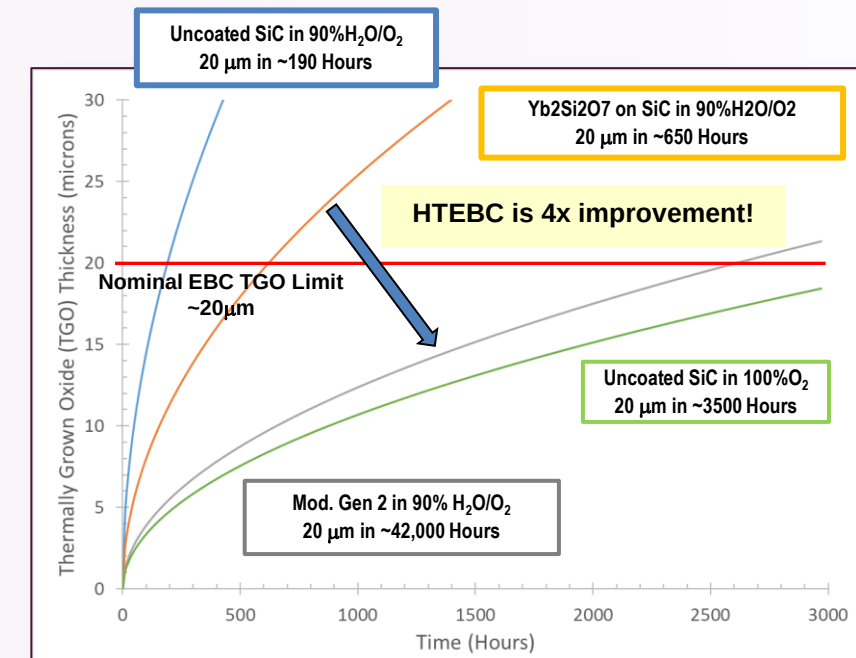
HTEBC – 2700 F

TRL 5 Validation

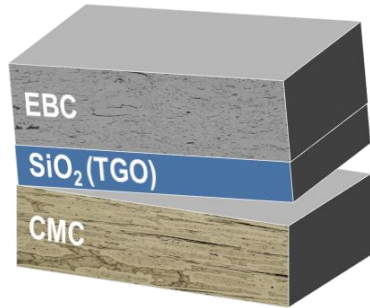
- 1000 hrs steam ox.
- 250 hrs NG/O₂ @ 3000/2700 F EBC/CMC
- 1000 cycles JETS @ 3000/2700 F EBC/CMC
- 9hrs CE-5 combustion rig



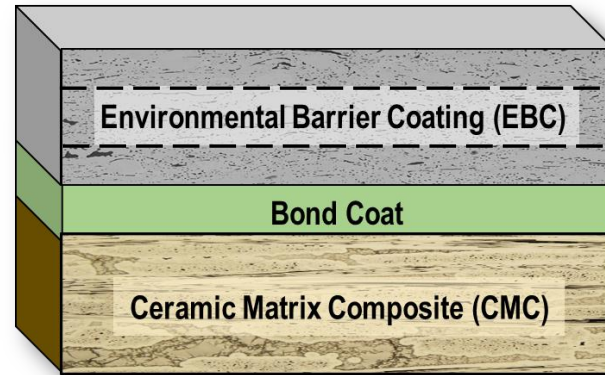
- 2700 F (1482 C) EBCs being developed with oxide-based high temperature bond coats (non Si) – slurry coating process
- Thermal barrier topcoats being developed for 2700 F (1842 C) EBC systems – PS-PVD deposition



Current Thrusts – Modeling EBC Failure Modes

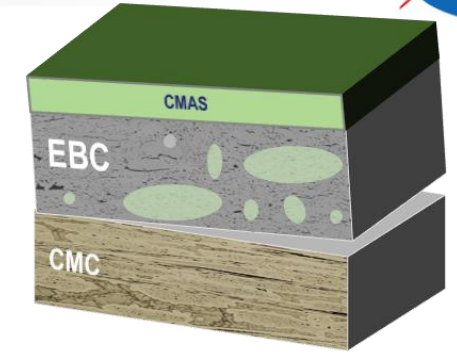


Steam Oxidation

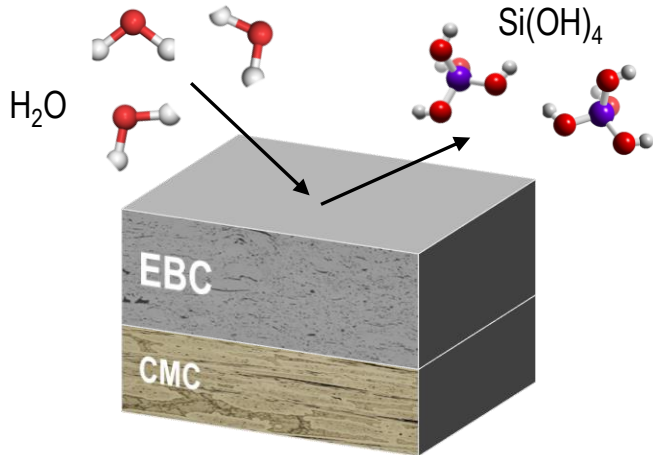


TC: Barrier from environment

BC: Bonding, adhesion, oxidation resistance

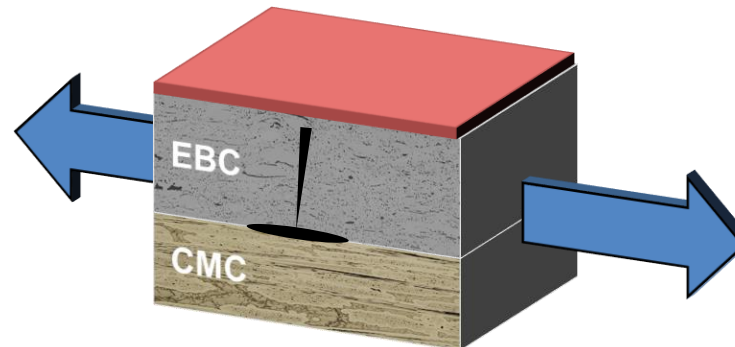


CMAS Attack & Infiltration
(**C**alcium-**M**agnesium-**A**lumino-**S**ilicate)

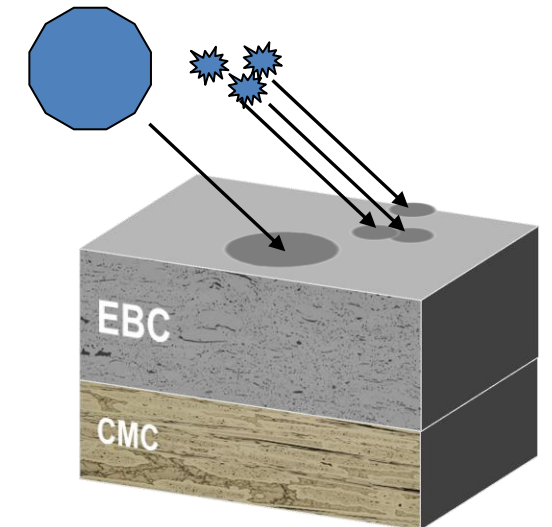


Hydroxide Formation/Recession

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



Thermomechanical Durability



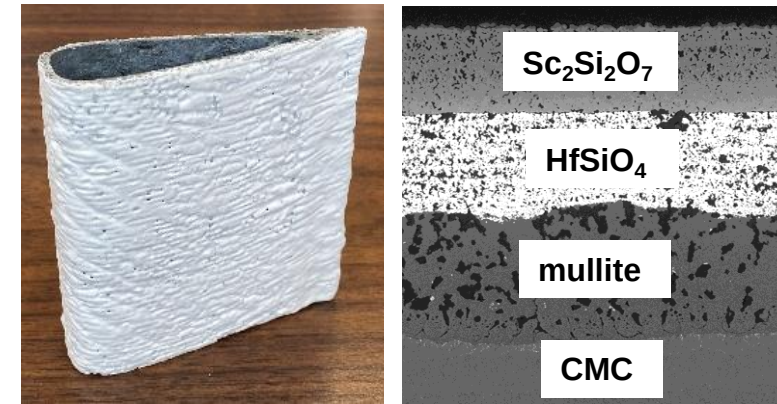
Erosion and FOD

Capabilities – EBC Processing

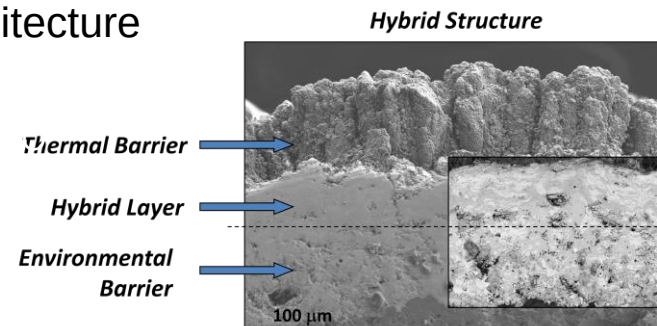
Advanced processing methods used to generate chemistries and architectures

- Commercial APS
- Slurry processing
 - Non-line of sight / complex shapes
 - Material/chemistry flexible
 - Airbrush or dip/spin coating
 - Must be sintered above use temp
- Plasma Spray- Physical Vapor Deposition (PS-PVD)...1 of 2 in U.S.
 - Variable microstructure and architecture
 - via liquid and/or vapor
 - Material flexible
 - Multilayer coatings
 - Non-line of sight

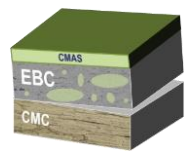
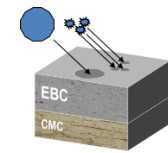
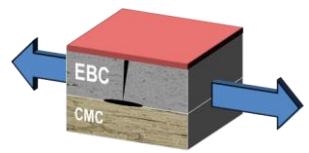
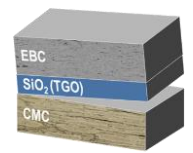
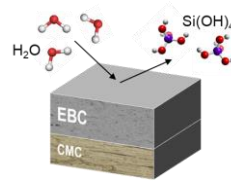
Slurry deposition of EBCs



PS-PVD processing



Capabilities - Environmental Testing



Recession

Oxidation

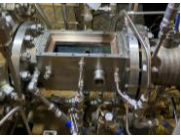
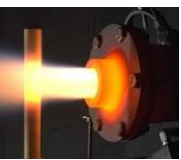
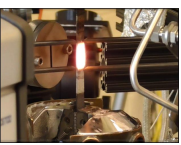
Thermomechanical

Erosion/FOD

CMAS

- Thermochemistry**
 $P(H_2O) = N/A$
 $V = N/A$
 $P_{total} = N/A$
- Steam Cycling**
 $P(H_2O) = 0.9atm$
 $V = 10\text{ cm/s}$
 $P_{total} = 1\text{ atm}$
- High Heat Flux Laser**
 $P(H_2O) = 0.1atm$
 $V = \text{none}$
 $P_{total} = 1\text{ atm}$
- Jet-A Burner Rig**
 $P(H_2O) = 0.1\text{ atm}$
 $V \sim 100 - 340\text{ m/s}$
 $P_{total} = 1\text{ atm}$
- NG/O₂ Burner Rig**
 $P(H_2O) = 0.1 - 0.5\text{ atm}$
 $V \sim 100 - 250\text{ (est.) m/s}$
 $P_{total} = 1\text{ atm}$
- Combustion Rig**
 $P(H_2O) = >6\text{ atm}$
 $V \sim 30\text{ m/s}$
 $P_{total} = 10 - 30\text{ atm}$

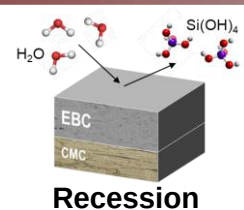
✓				✓
✓	✓			✓
	✓	✓	✓	✓
✓	✓	✓	✓	✓
✓	✓	✓	✓	✓
✓	✓	✓	✓	



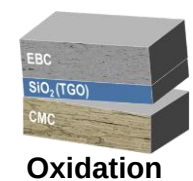
Environmental Testing - Thermochemistry

Thermochemistry

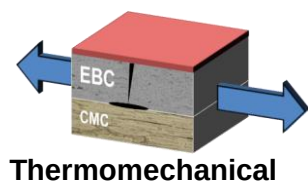
$P(\text{H}_2\text{O}) = \text{N/A}$
 $V = \text{N/A}$
 $P_{\text{total}} = \text{N/A}$



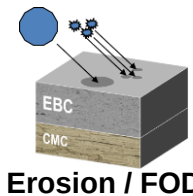
Recession



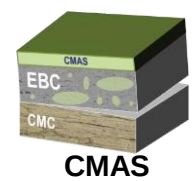
Oxidation



Thermomechanical



Erosion / FOD

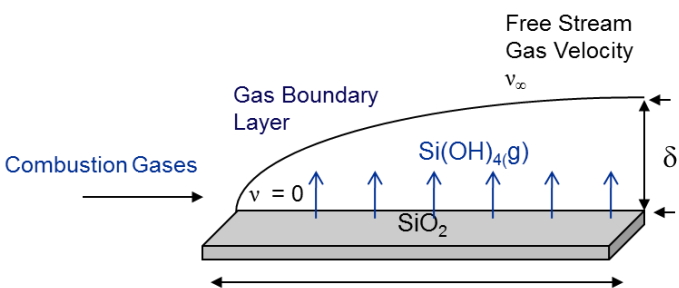


CMAS



- 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)

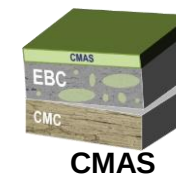
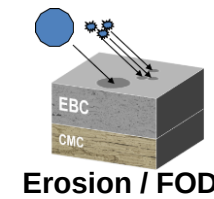
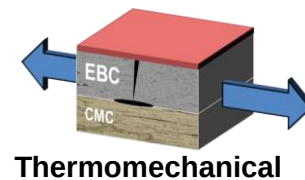
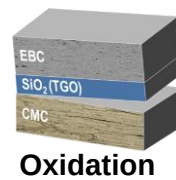
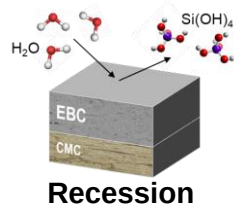


Calorimeter

Environmental Testing – Steam Cycling

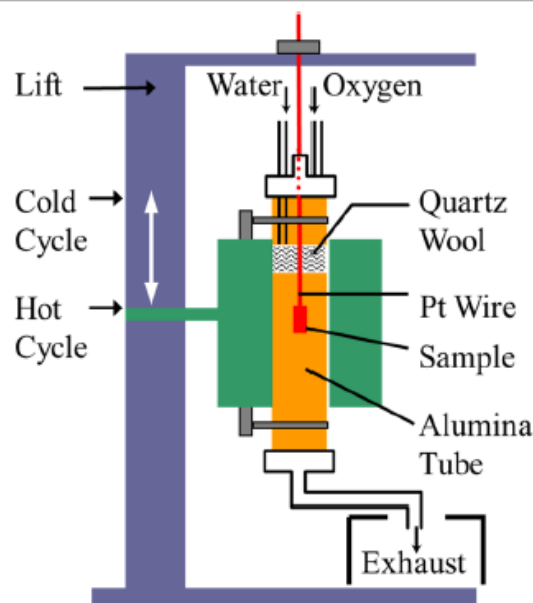
Steam Cycling

$P(\text{H}_2\text{O}) = 0.9\text{atm}$
 $V = 10\text{ cm/s}$
 $P_{\text{total}} = 1\text{ atm}$

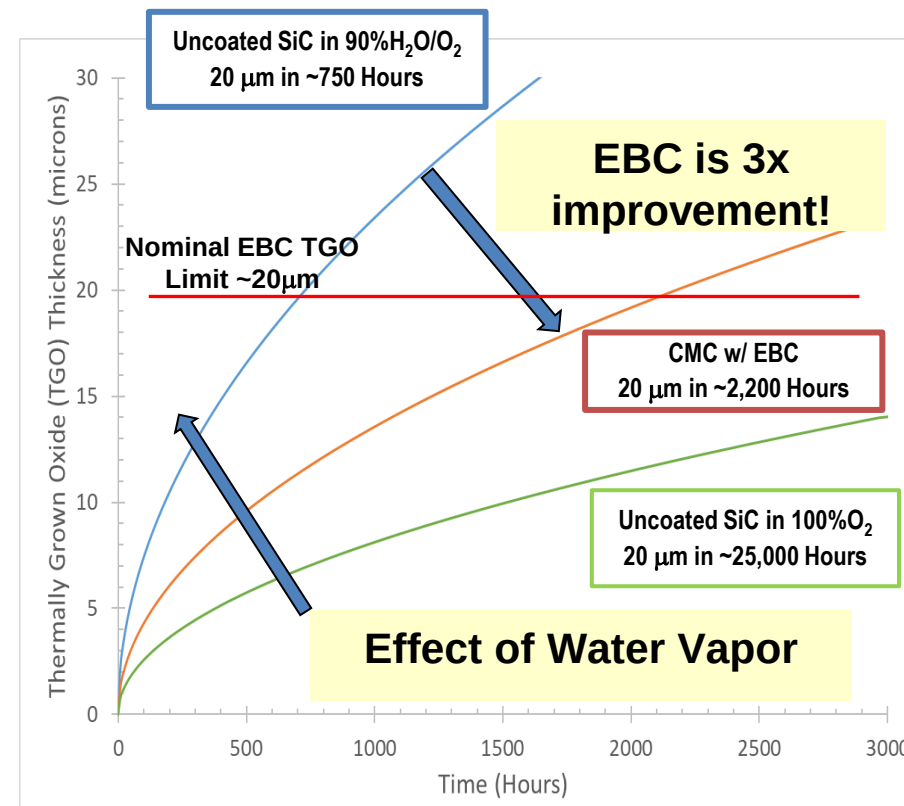


$$x_{TGO} = \sqrt{k_p t}$$

Steam Cycling Schematic



- Continuous cycling of samples at temperature forms thermally grown oxides (TGO) like SiO_2 .
 - TGO is the weak EBC interface/life-limiting factor!
- Up to 2700 F (1482C), ~0.1 m/sec, pH₂O ~0.9 atm, 1 atm total press, 1 hr hot/30 min cool
- Cycling in 90% H₂O/O₂ is akin to 9atm combustion environment!
- Steam oxidation provides 10x increase in TGO versus air.



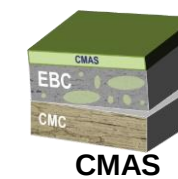
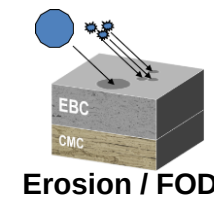
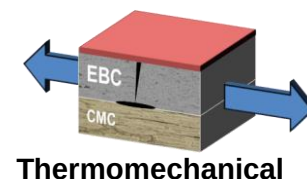
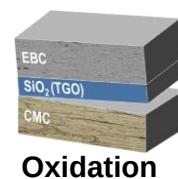
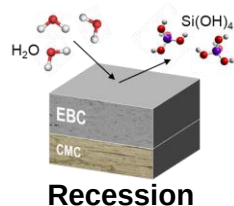
Environmental Testing – High Heat Flux Laser

High Heat Flux Laser

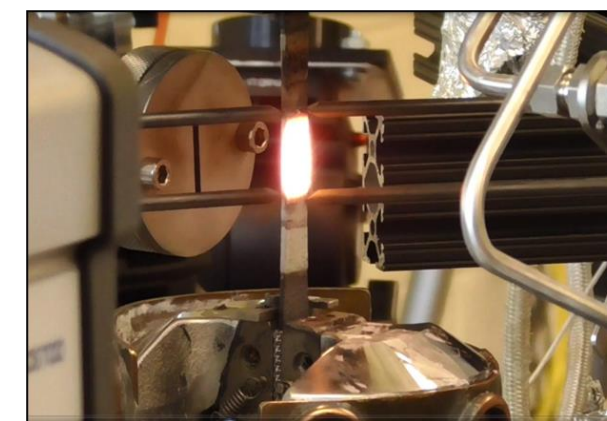
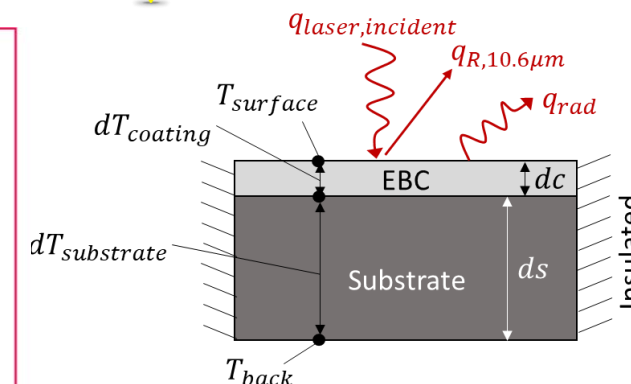
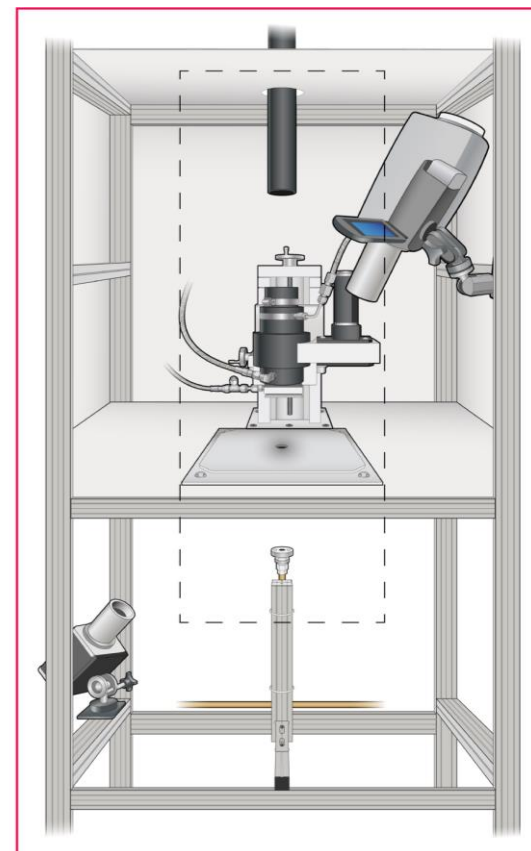
$P(\text{H}_2\text{O}) = 0.1\text{atm}$

$V = \text{none}$

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



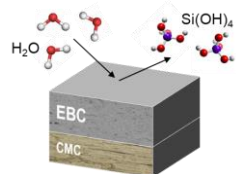
- High-powered Carbon Dioxide (CO_2) lasers
 - $10.6\mu\text{m}$ wavelength = high absorption EBC materials
- Thermal or combined thermal-mechanical testing capabilities, backside cooling for ΔT testing
- Surface temperatures $> 3,000^\circ\text{ F}$ (1650° C) are achievable
- Incident heat flux up to 300 W/cm^2 (assuming 1 inch diameter spot size)
- Heat transfer properties (thermal conductivity)



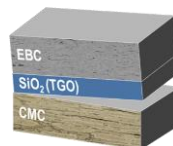
Environmental Testing – Jet-A Burner Rig

Jet-A Burner Rig

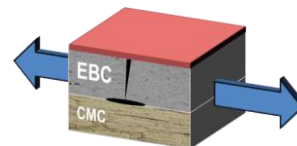
$P(\text{H}_2\text{O}) = 0.1 \text{ atm}$
 $V \sim 100 - 340 \text{ m/s}$
 $P_{\text{total}} = 1 \text{ atm}$



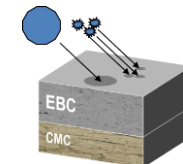
Recession



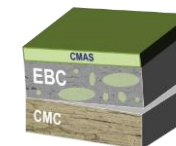
Oxidation



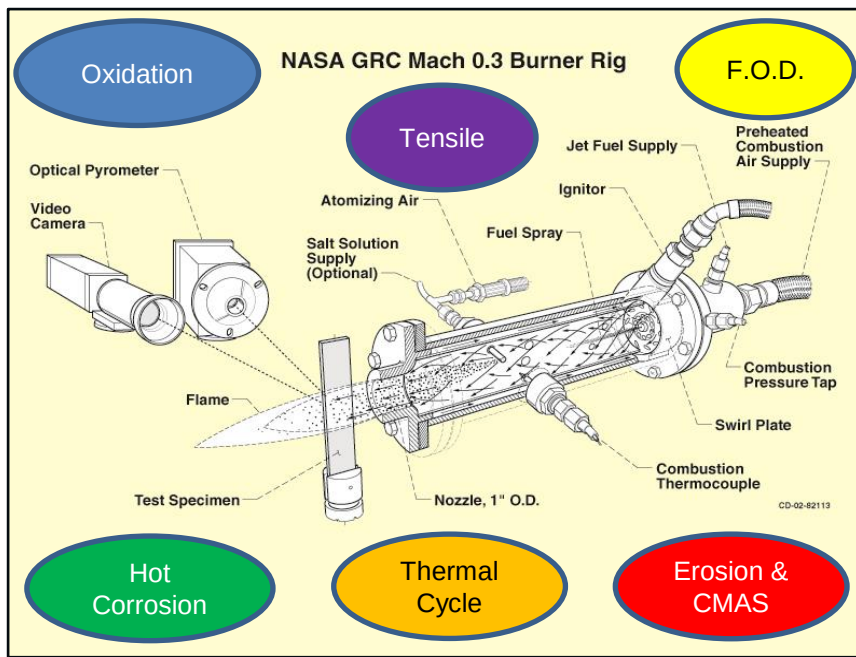
Thermomechanical



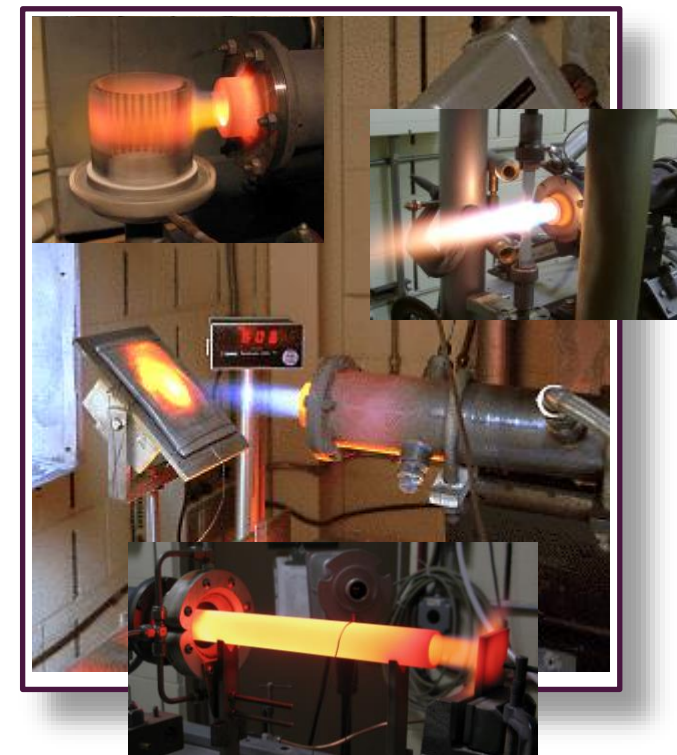
Erosion / FOD



CMAS



- 8 computer-controlled atmospheric, jet-fueled combustors
- Material test temperatures from 600° to 2700°F, flame temperatures to 3000°F
- Mach 0.3 to 1.0 (100-340 m/s)
- Multiple or single samples tested using rotating carousels
- Thermal cycling
- ~10% water vapor



Environmental Testing - Natural Gas / Oxygen Burner Rig

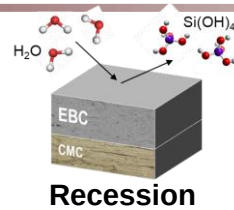
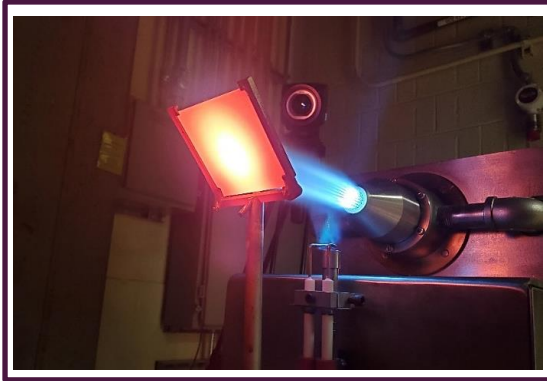


NG/O₂ Burner Rig

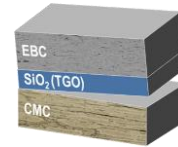
$P(\text{H}_2\text{O}) = 0.1 - 0.5 \text{ atm}$

$V \sim 100 - 300 \text{ (est.) m/s}$

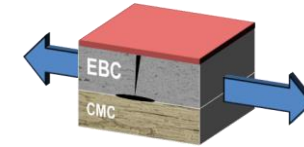
$P_{\text{total}} = 1 \text{ atm}$



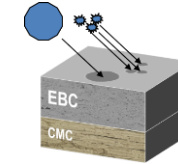
Recession



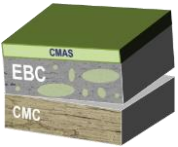
Oxidation



Thermomechanical



Erosion / FOD

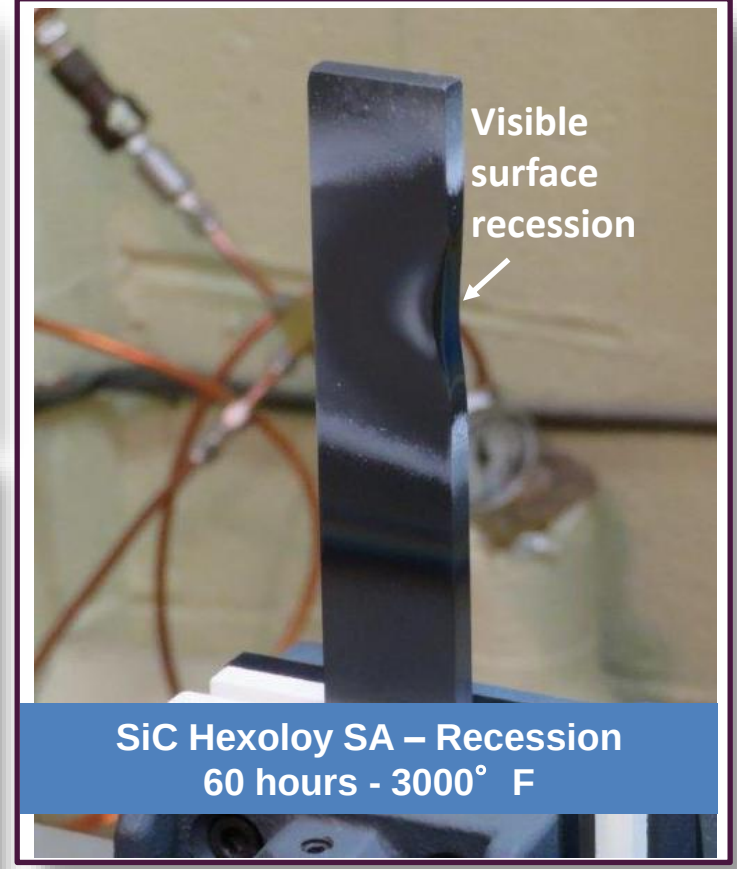
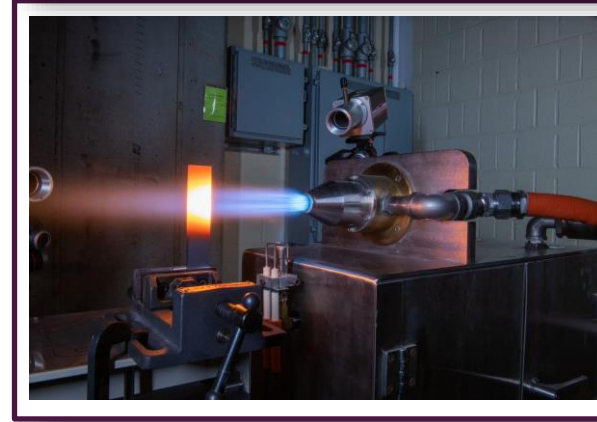
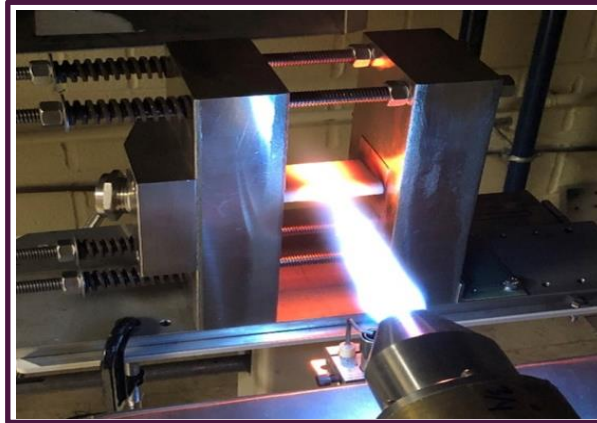


CMAS



Chemical Equilibrium Analysis (CEA)

- Peak flame temperature – 4,986° F
- $P(\text{H}_2\text{O}) \sim 0.1 - 0.4 \text{ atm} \rightarrow P(\text{total}) = 1 \text{ atm}$
- Gas velocity estimated: $\sim 100 - 300 \text{ m/s}$
- High heat flux (up to $\sim 200 \text{ W/cm}^2$)
- 1000's of hrs. at 3000F completed

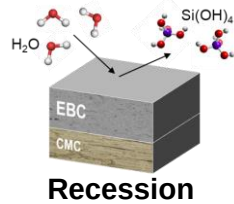


SiC Hexoloy SA – Recession
60 hours - 3000° F

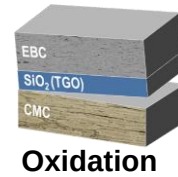
Environmental Testing – NASA Combustion Rig

Combustion Rig

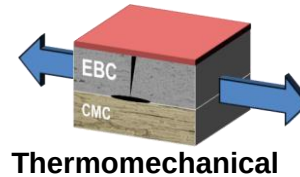
$P(\text{H}_2\text{O}) = >6 \text{ atm}$
 $V \sim 30 \text{ m/s}$
 $P_{\text{total}} = 10 - 30 \text{ atm}$



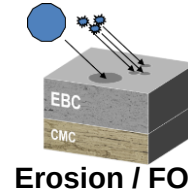
Recession



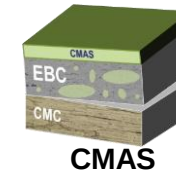
Oxidation



Thermomechanical

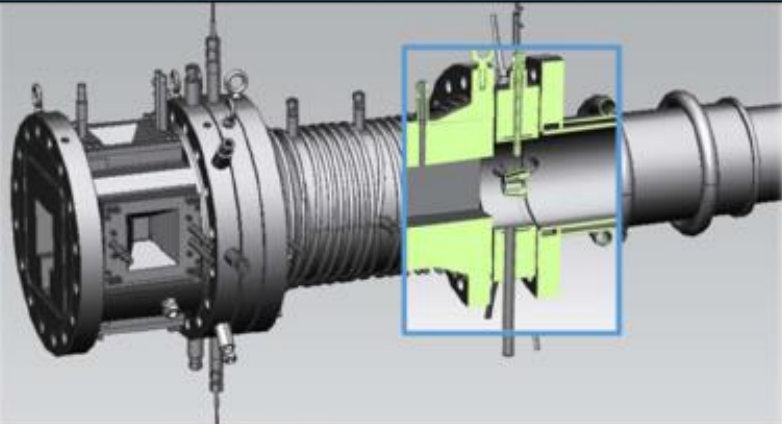


Erosion / FOD



CMAS

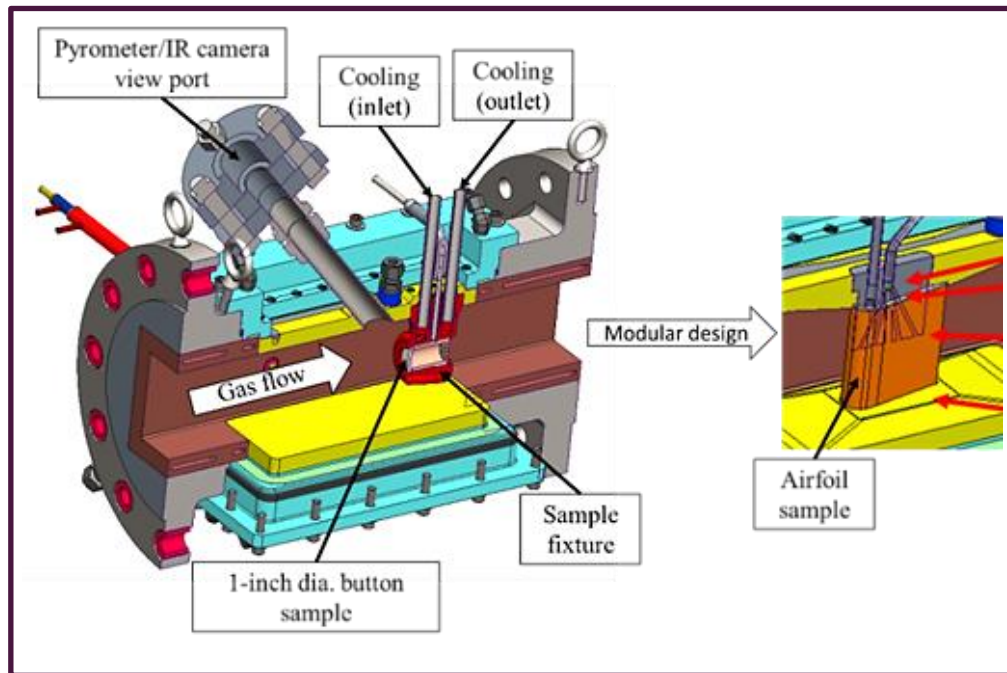
NASA GRC CE-5 Test Stand



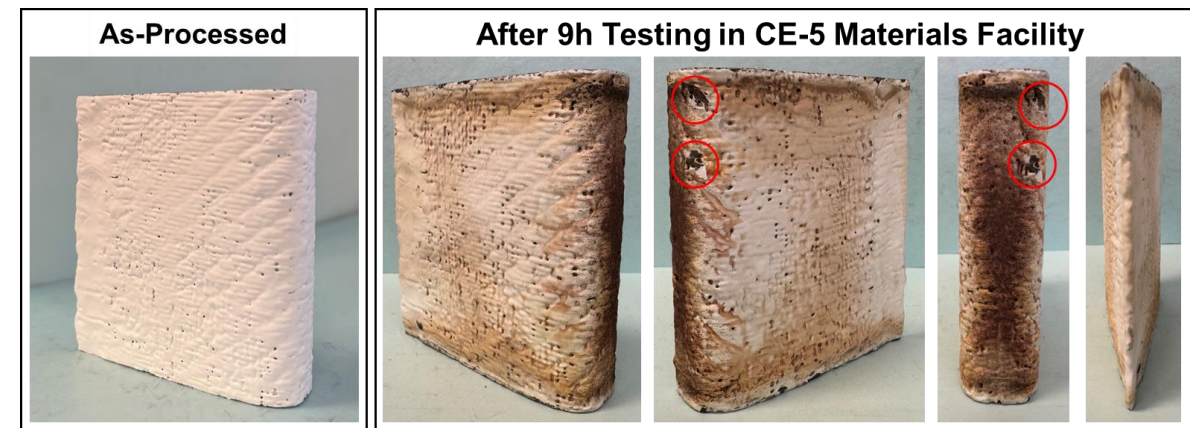
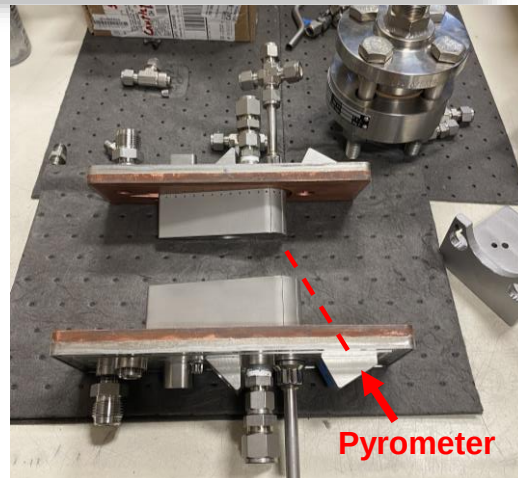
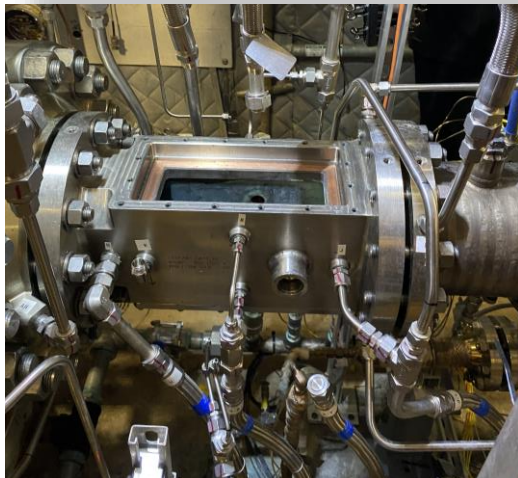
30 atm pressure
>1650°C gas temperature
Maximum 20 lb/s Mass Flow

- 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+ °F
 - Pressure / Velocity: 270 psi / ~40 m/s
 - Backside cooling temperature: 900 °F
 - 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.



Gaps – Hypersonic test facilities



Variable Plasma Environmental Test Rig (VaPER)

- 100kW Thermal Spray Torch
- Low pressure (variable chamber range: 0.75 – 40 mbar)
- Velocity of ~Mach 2...potentially higher with new nozzle design
- Continuous Ar/He plasma
- Test gases – air, N₂, N₂/O₂ mixtures, CO/Ar or CO/N₂ mixtures

Test Conditions

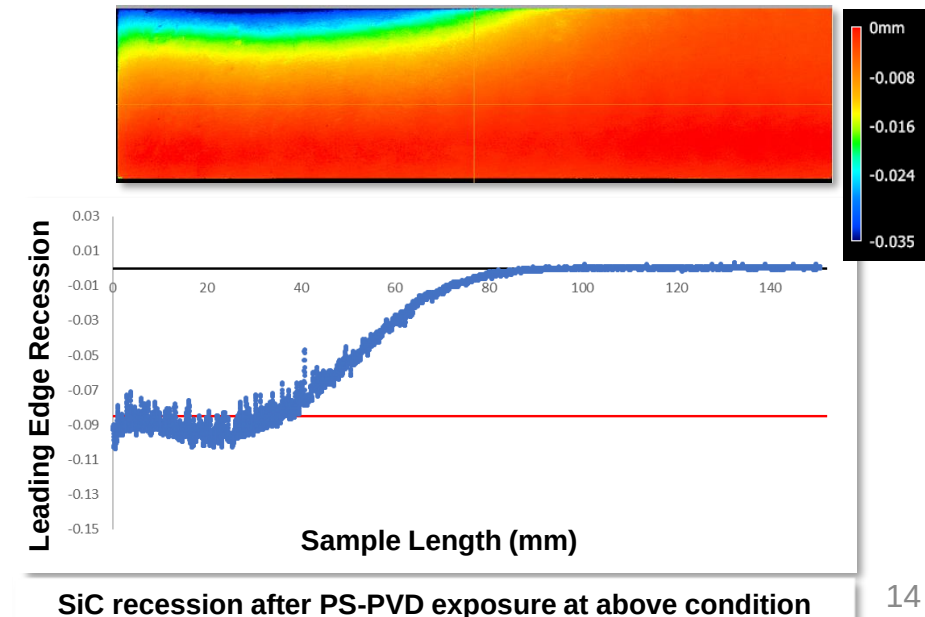
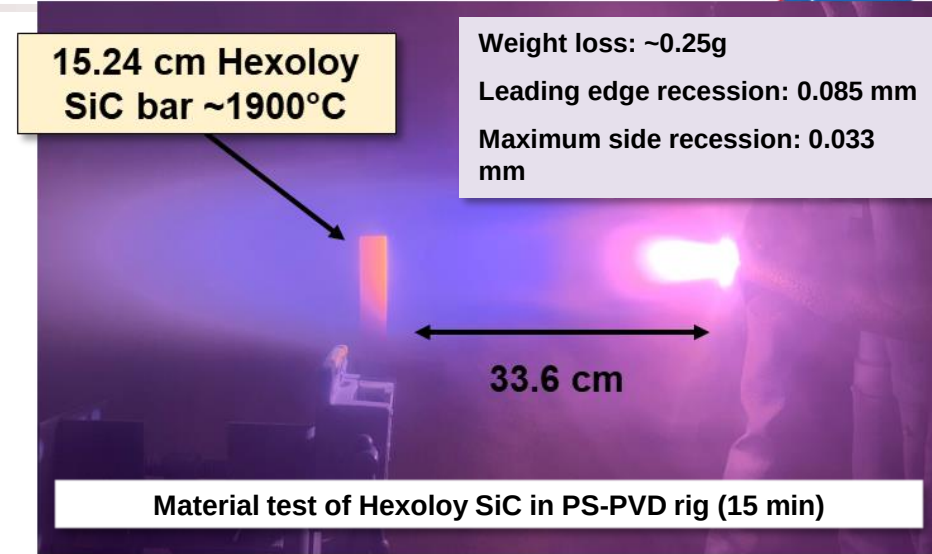
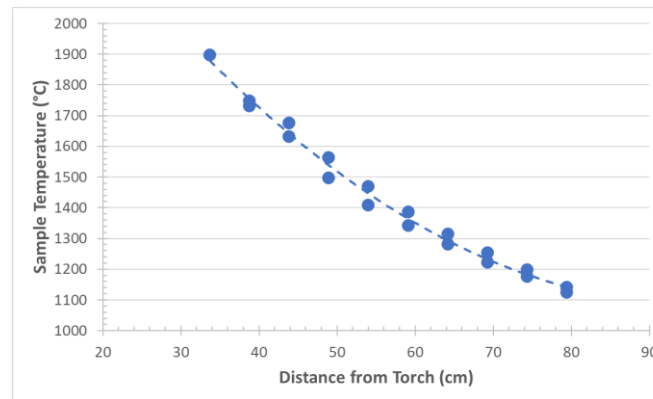
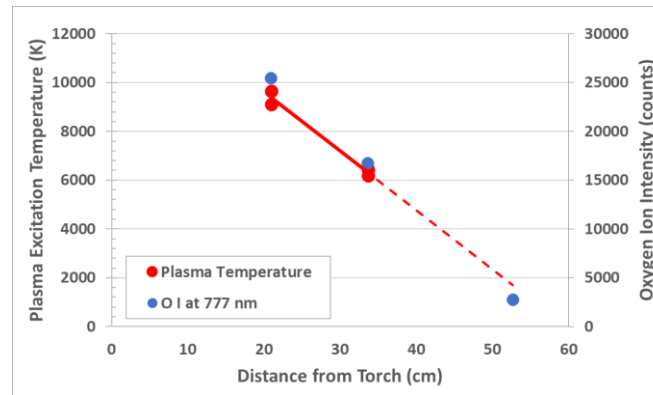
- Plasma excitation temperature: ~10,000K
- Heat flux: TBD, expected >200 W/cm²
- Plasma diameter: variable, 25-75mm
- Chamber: 1.5m with capability for water/instrumentation connections
- Test time range: minutes to hours (if needed)

Instrumentation

- Optical Spectrometer to determine excitation levels of Ar, He, O, N, etc.
- Pyrometers, thermocouples, thermal imaging

Future Plans

- Integrate pitot tube and slug calorimeter to determine heat flux

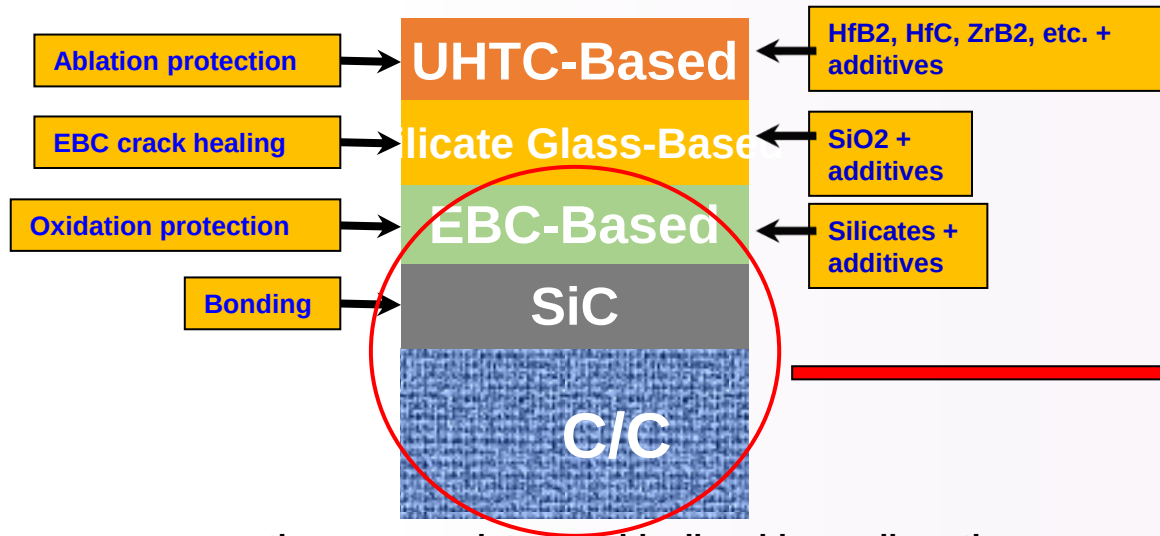


Gaps – Coatings for Hypersonics



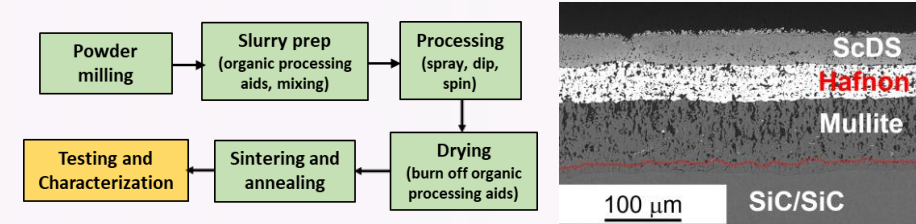
Multi-layer Coatings Concept

UTHCs, Silicate glasses, and SiC technologies all part of SOA. Key is to successfully integrate the various layers and add EBCs for oxidation.



- Multilayer coatings approach to individually address all coating requirements
- Technology for each layer exists, but no coatings technology combining all four technologies exist
- Leverages NASA's expertise in EBCs, slurry process, high temperature materials chemistry, and environmental testing

Center Innovation Fund (CIF) Project



Project Vision/Goals

- Protective oxidation coating with innovative dense silica oxidation barrier and self-healing coating microstructure protects C/C and C/SiC to help enable realization of hypersonic vehicles.
- Goal is to apply NASA-developed mullite-based coating to C/C and C/SiC composites to meet oxidation life goal of these composites in hypersonic environments at temperatures of up to 3000F.
- Oxidation goals based on DARPA/Air Force Falcon Program

Project Plans and Milestones

- Eutectic temperature vs. chemistry map (Jan 2024)
- Procurement of C/C and C/SiC test coupons (Dec 2023)
- Down-selection of self-healing and dense silica forming coating chemistries (Jul 2024)
- Validate oxidation protection capability via furnace, laser, and burner rig tests (Sep 2024)

Questions

