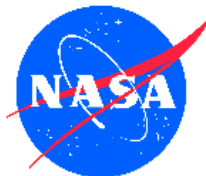


# **Advanced Environmental Barrier Coating Development for SiC-SiC Ceramic Matrix Composite Components**

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**12th Pacific Rim Conference on Ceramic and Glass Technology (PACRIM 12)  
Waikoloa, Hawaii  
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# Durable Thermal and Environmental Barrier Coating Systems for Ceramic Matrix Composites (CMCs):

Enabling Technology for Low Emission, High Efficiency and Light-Weight Propulsion

## — NASA Environmental barrier coatings (EBCs) development objectives

- Help achieve future engine temperature and performance goals
- Ensure system durability – towards prime reliant coatings
- Establish database, design tools and coating lifing methodologies
- Improve technology readiness



Fixed Wing Subsonic Aircraft



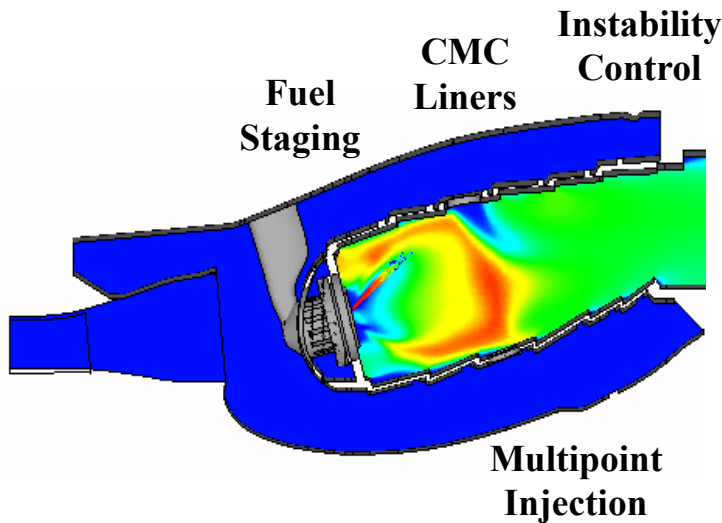
Supersonics Aircraft



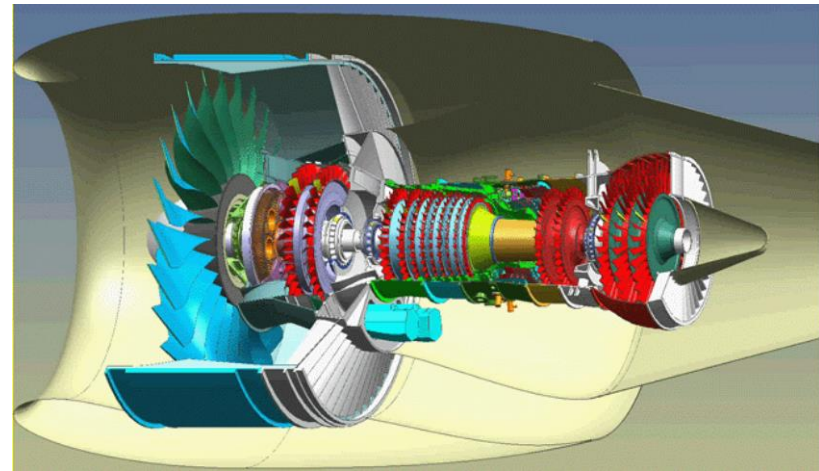
Hybrid Electric Propulsion Aircraft

## NASA Environmental Barrier Coating (EBC) - Ceramic Matrix Composite (CMC) Development Needs

- **NASA Aeronautics Programs:** Next generation high pressure turbine airfoil environmental barrier coatings with advanced CMCs
  - N+3 generation (2020-2025) with advanced 2700°F CMCs/2700-3000°F EBCs (uncooled/cooled)
- **NASA Environmentally Responsible Aviation (ERA) Program:** Advanced environmental barrier coatings for SiC/SiC CMC combustor and turbine vane components, technology demonstrations in engine tests
  - N+2 generation (2020-2025) with 2400°F CMCs/2700°F EBCs (cooled)



Low emission combustor

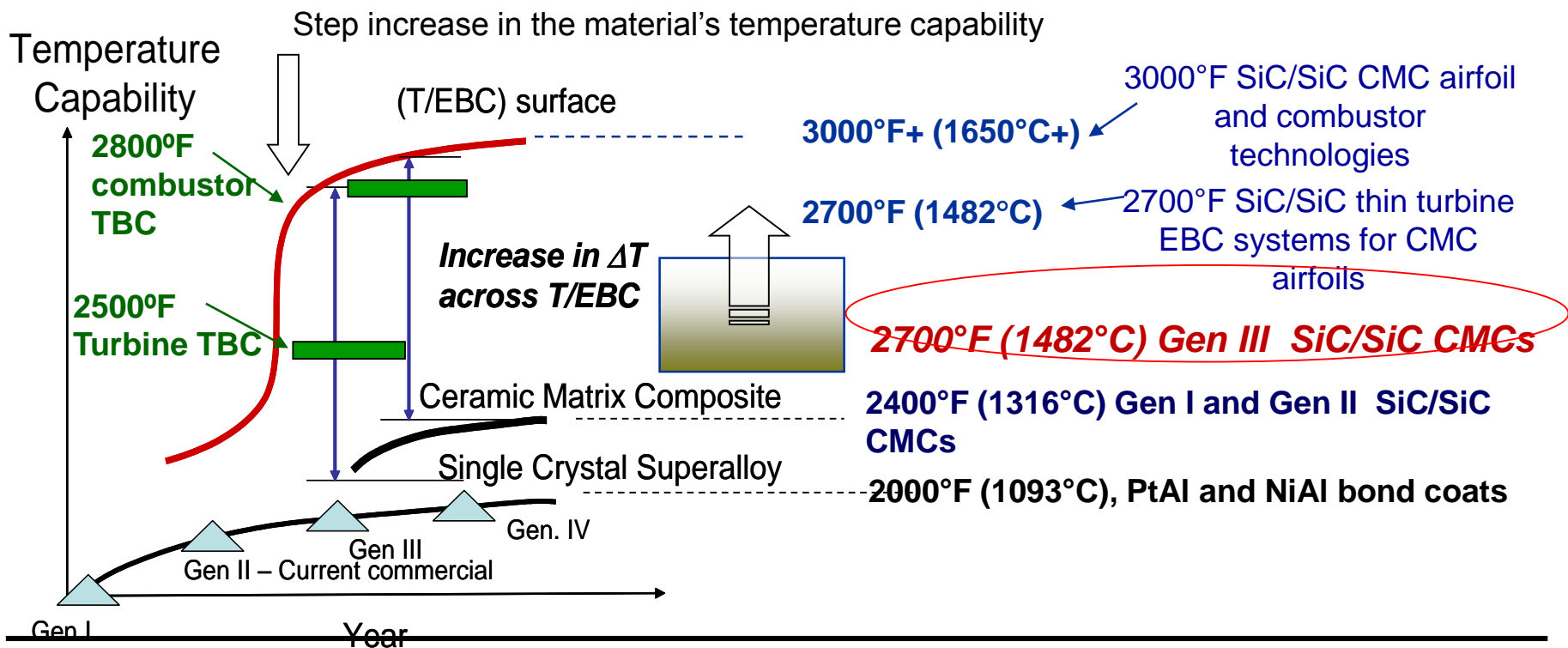


High Pressure Turbine CMC vane and blade



## NASA Environmental Barrier Coatings (EBCs) and Ceramic Matrix Composite (CMC) System Development

- **Emphasize material temperature capability, performance and *long-term* durability-** Highly loaded EBC-CMCs with temperature capability of 2700°F (1482°C)
  - 2700-3000°F (1482-1650°C) turbine and CMC combustor coatings
  - 2700°F (1482°C) EBC bond coat technology for supporting next generation
    - Recession: <5 mg/cm<sup>2</sup> per 1000 h
    - Coating and component strength requirements: 15-30 ksi, or 100- 207 Mpa
    - **Resistance to Calcium Magnesium Alumino-Silicate (CMAS)**





# Outline

- **SiC/SiC ceramic matrix composite environmental barrier coating system development**
  - Environmental barrier coatings combustors – compositions, processing scaleup, and rig tests
    - HfO<sub>2</sub>-Si based 2700°F bond coats
    - Rare Earth and Hafnium-rare earth-silicate EBCs
  - Advanced thermal spray and hybrid vapor - plasma processing coatings for SiC/SiC CMC components – environmental stability assessments
  
- **The EBC system degradations and failure modes in long-term High Pressure Burner Rig liner tests**
  
- **NASA advanced 2700°F CMAS resistance coating developments**
  - CMAS resistance evaluations of plasma sprayed combustor coatings
  
- **Summary and conclusions**



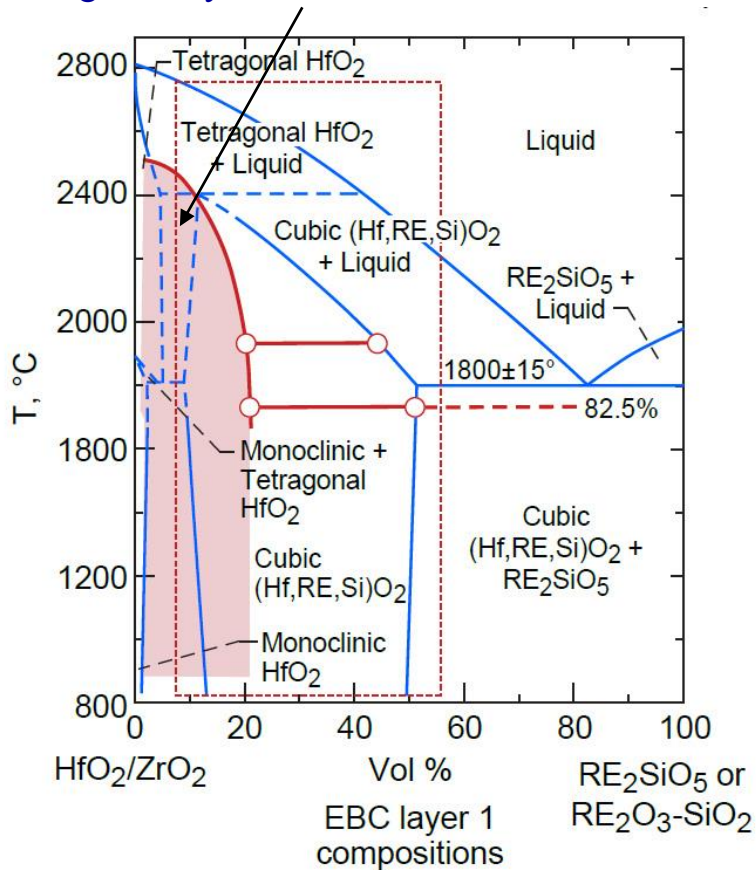
## NASA Combustor EBC Development

- High stability multicomponent  $\text{HfO}_2$  or  $\text{ZrO}_2$ ,  $\text{HfO}_2\text{-RE}_2\text{O}_3\text{-SiO}_2/\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$  / environmental barrier/environmental barrier coating systems
- Advanced 2700°F capable bond coats
  - Hafnium aluminate-silicates and rare earth aluminate silicates developed
  - $\text{HfO}_2\text{-Si}$  first Gen bond coat for component tests
  - Second Gen 2700°F bond coat being developed based on rare earth -Si
  - Calcium Magnesium Alumino-Silicate (CMAS) resistance was addressed
- Develop advanced compositions for combustor EBC applications with Sulzer (Oerlikon) Metco, Praxair and others
- Develop high toughness and CMAS resistant coating systems

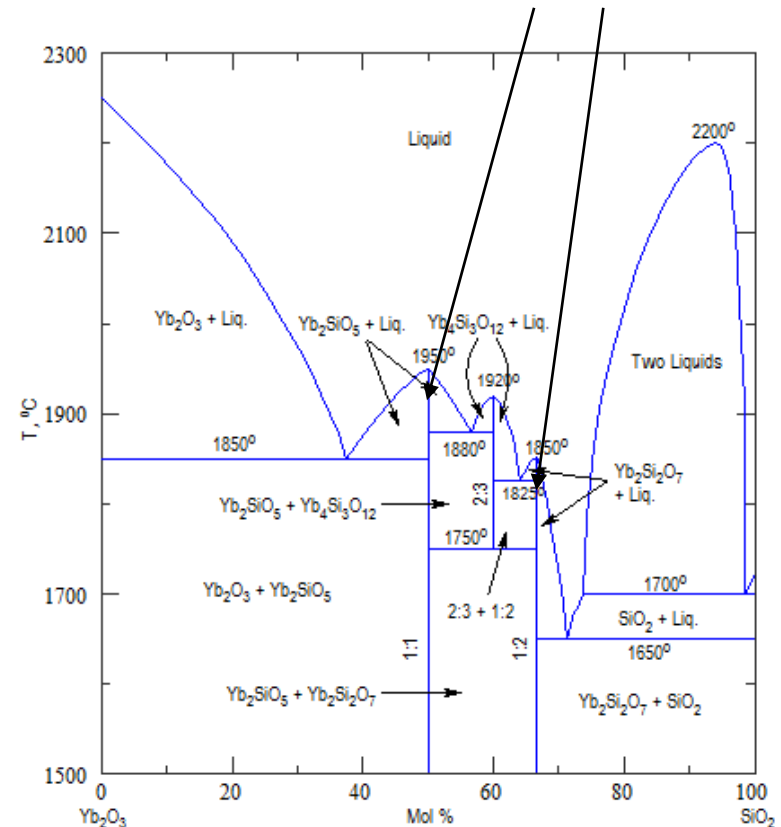
## Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability

- Hafnium Rare Earth Silicate System and Rare Earth Silicate EBC Systems
- Multi-component EBC systems are preferred and being developed

High temperature and  $t'$  phase region high toughness system and CMAS resistance



Rare earth monosilicate and di-silicate systems



# Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability

— Multi-component oxide defect clustering approach for stability and CMAS resistance

e.g.:  $\text{ZrO}_2\text{-Y}_2\text{O}_3\text{-Nd}_2\text{O}_3(\text{Gd}_2\text{O}_3, \text{Sm}_2\text{O}_3)\text{-Yb}_2\text{O}_3(\text{Sc}_2\text{O}_3)$  systems

↳ Primary stabilizer



Oxide cluster dopants with distinctive ionic sizes

e.g.:  $(\text{HfO}_2)\text{-Y}_2\text{O}_3\text{-Nd}_2\text{O}_3(\text{Gd}_2\text{O}_3, \text{Sm}_2\text{O}_3)\text{-Yb}_2\text{O}_3(\text{Sc}_2\text{O}_3) - \text{SiO}_2$  systems

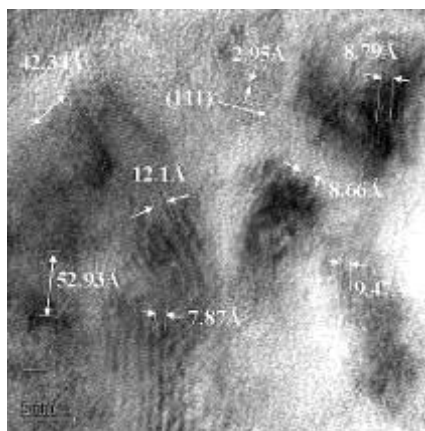
↳ Primary stabilizer



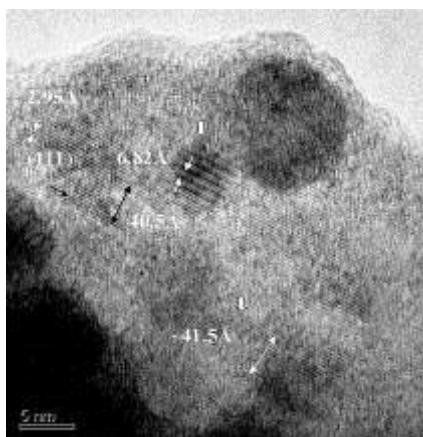
Oxide cluster dopants with distinctive ionic sizes

Nano-reactive high stability  
Yb, Hf silicate based EBCs  
CMAS melt stabilizers Gd or  
Nd dopants

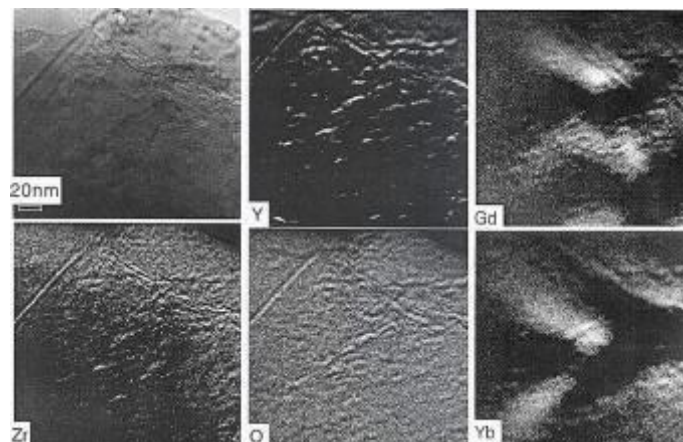
— The nanometer sized clusters for reduced thermal conductivity, improved stability, toughness, CMAS resistance and mechanical properties



Plasma-sprayed  $\text{ZrO}_2\text{-(Y, Nd, Yb)}_2\text{O}_3$



EB-PVD  $\text{ZrO}_2\text{-(Y, Nd, Yb)}_2\text{O}_3$

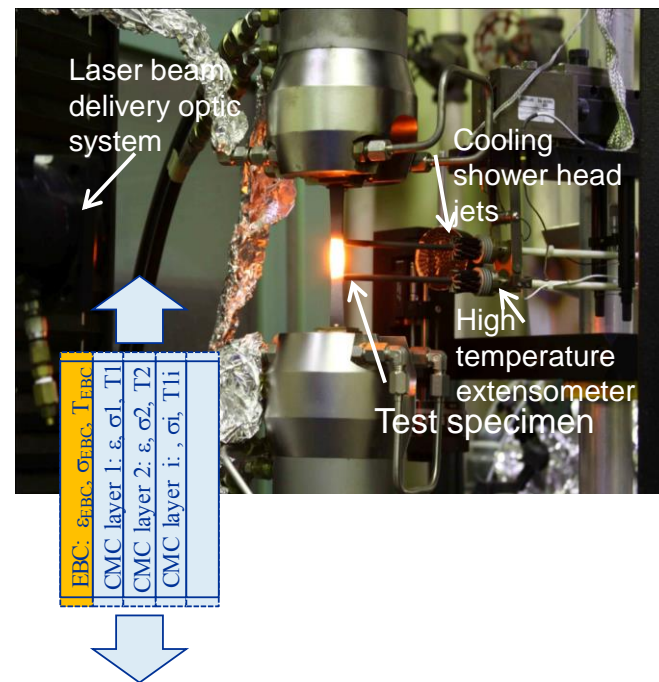
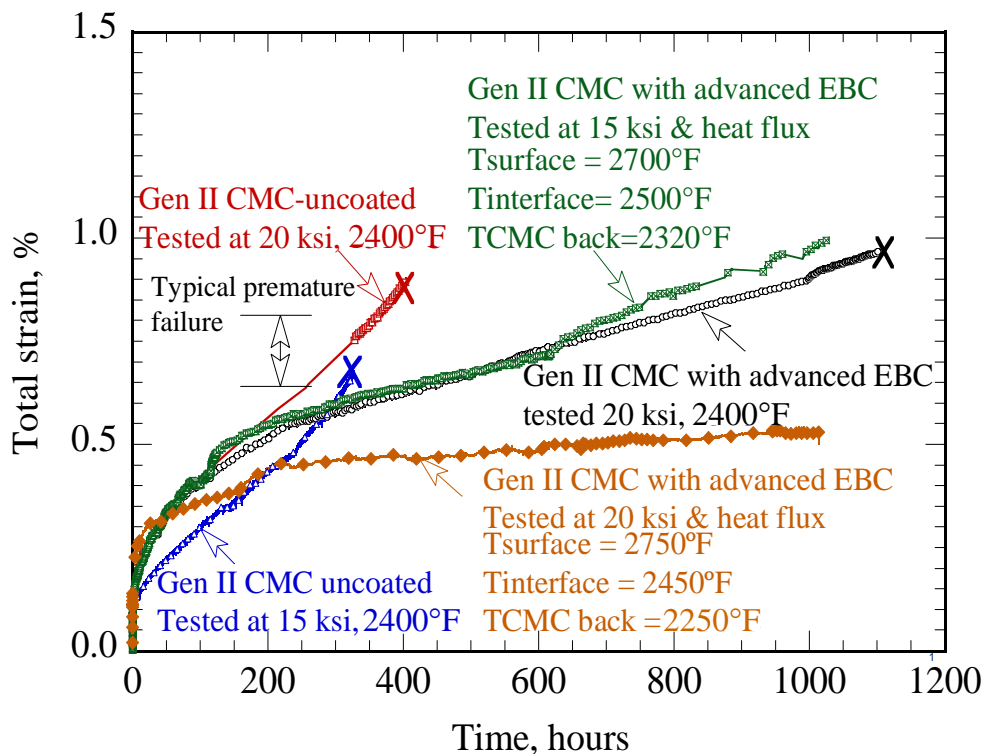


EELS elemental maps of EB-PVD  $\text{ZrO}_2\text{-(Y, Gd, Yb)}_2\text{O}_3$



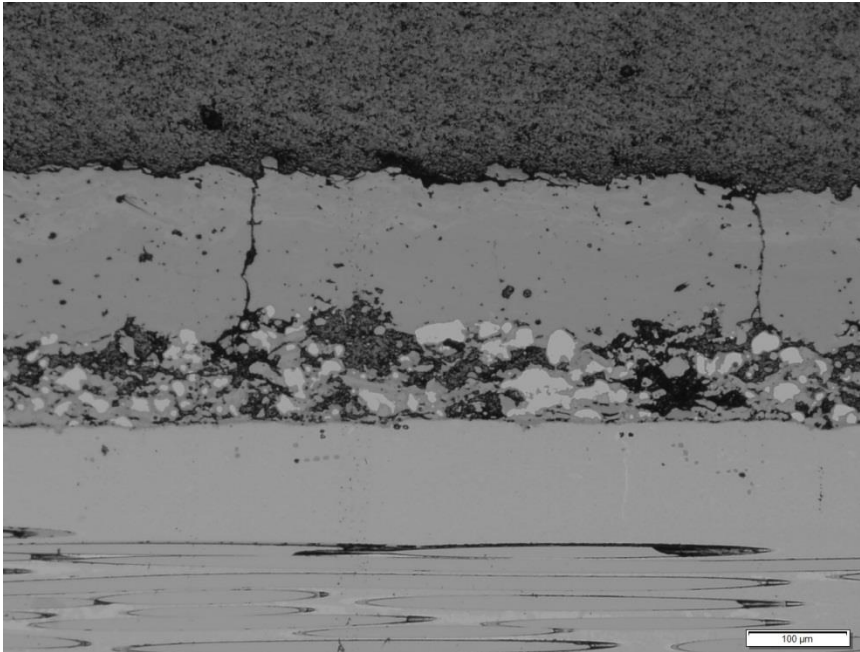
## Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs

- Advanced environmental barrier coatings – prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 ksi at  $T_{\text{EBC}}$  2700°F,  $T_{\text{CMC interface}} \sim 2500^\circ\text{F}$
- EBCs helped extending CMC rupture life in air tests
- The  $\text{HfO}_2$ -Si bond coat showed excellent durability

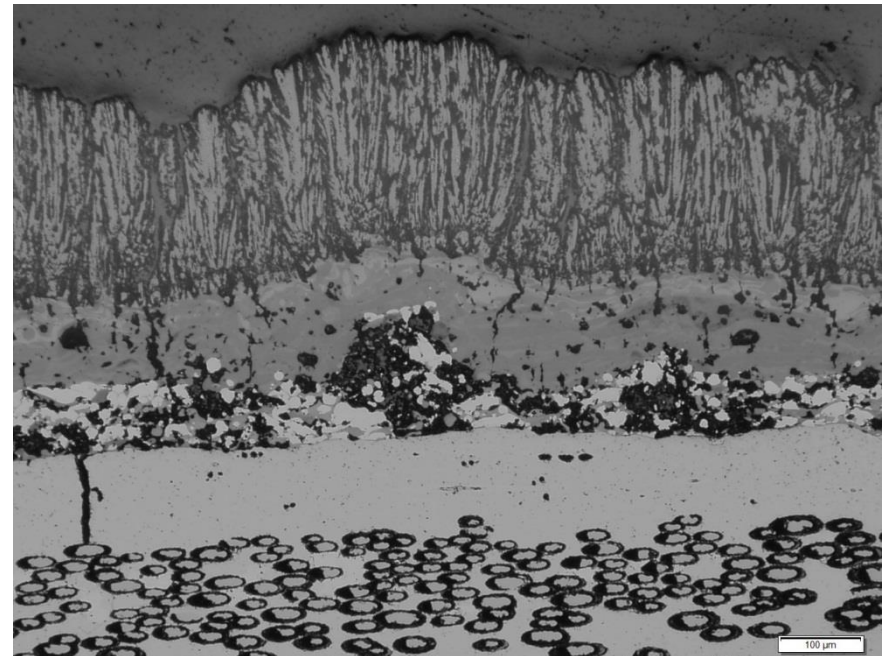


## Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued

- Advanced environmental barrier coatings – Prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 si at  $T_{\text{EBC}} 2700^{\circ}\text{F}$ ,  $T_{\text{CMC interface}} \sim 2500^{\circ}\text{F}$
- The  $\text{HfO}_2$ -Si bond coat showed tensile loading cracking resistance



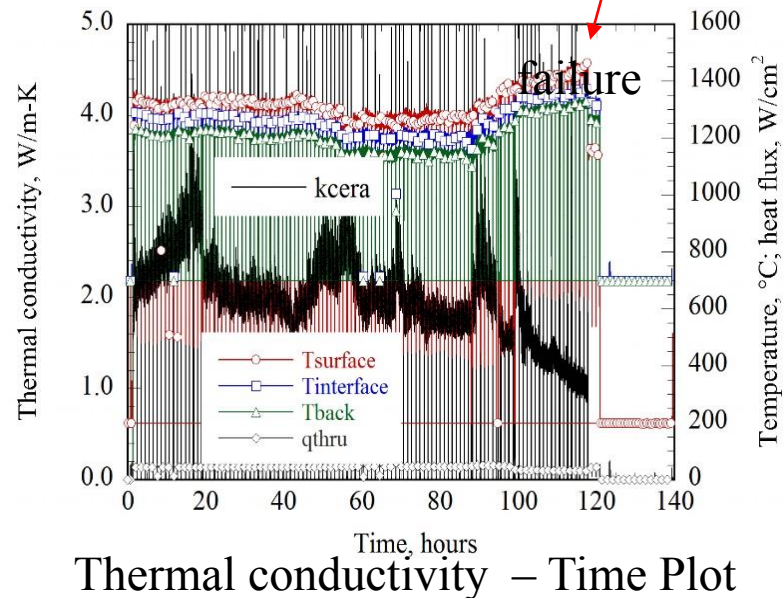
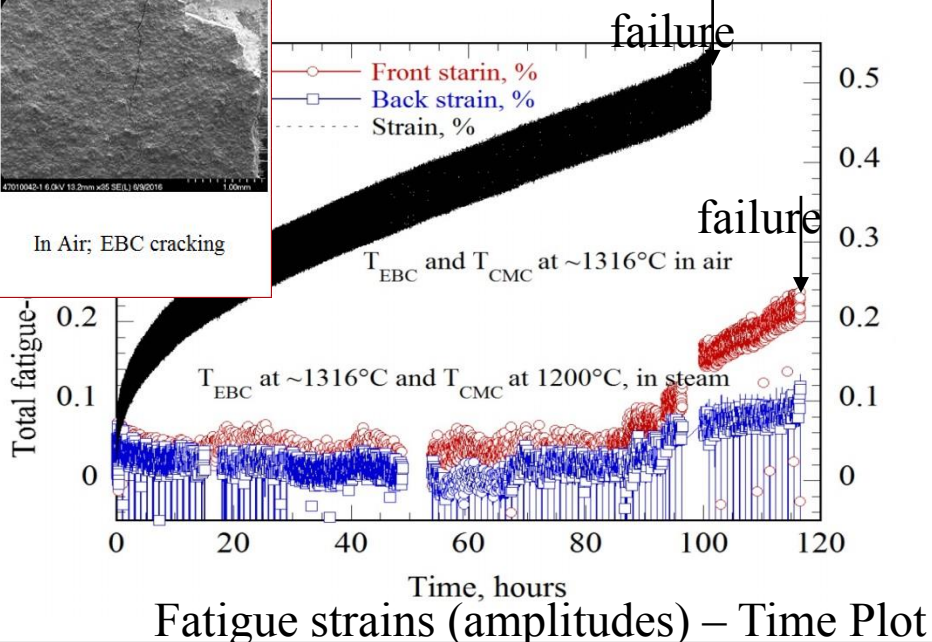
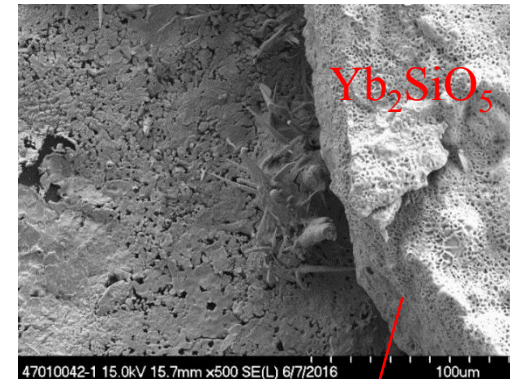
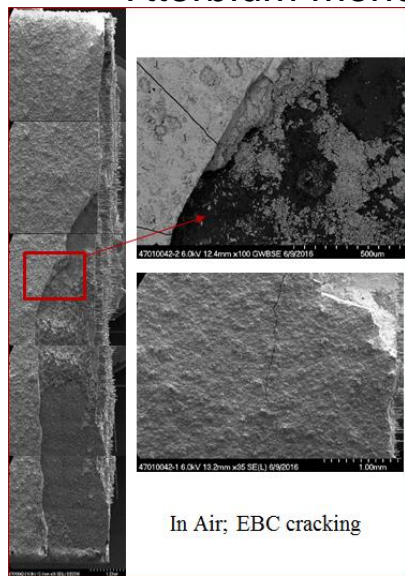
EBCs on Gen II CMC after 1000 hr creep rupture testing



Hybrid EBCs on Gen II CMC after 1000 hr low cycle creep fatigue testing

# Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability

- Environmental Barrier Coatings  $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7/\text{Si}$  on Melt Infiltrated (MI) Prepreg SiC/SiC CMC substrates
- One specimen tested in air, air testing at  $1316^\circ\text{C}$
- One specimen tested in steam, steam testing at  $T_{\text{EBC}} 1316^\circ\text{C}$ ,  $T_{\text{CMC}}$  at  $\sim 1200^\circ\text{C}$
- Lower CMC failure strain observed in steam test environments
- Ytterbium monosilicate recession observed in the test





## Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - Continued

- Plasma sprayed  $\text{HfO}_2\text{-RE}_2\text{O}_3$  (Silicate) top coat EBCs showed good stability from 2" discs specimens
- Demonstrated high pressure environmental stability at 2600-2650°F, no measurable recession weight loss in 160-200 psi (10-16 atm) in the high pressure burner rig test



2" diameter ND3 EBC/SiC/SiC specimen after testing in the high pressure burner rig



EBC ID 23\_3.5.3: Three-layer system  $\text{HfO}_2\text{-Si}$  bond coat/ $\text{Hf-Yb}$  silicate EBC layer /Silica graded multicomponent  $\text{Hf-RE}$  silicate EBC on Prepreg

High pressure burner rig, 16 atm, 31 hr



High pressure burner rig tested new ND series Hybrid EBC systems coated on 2" diameter Gen II Prepreg SiC/SiC CMCs

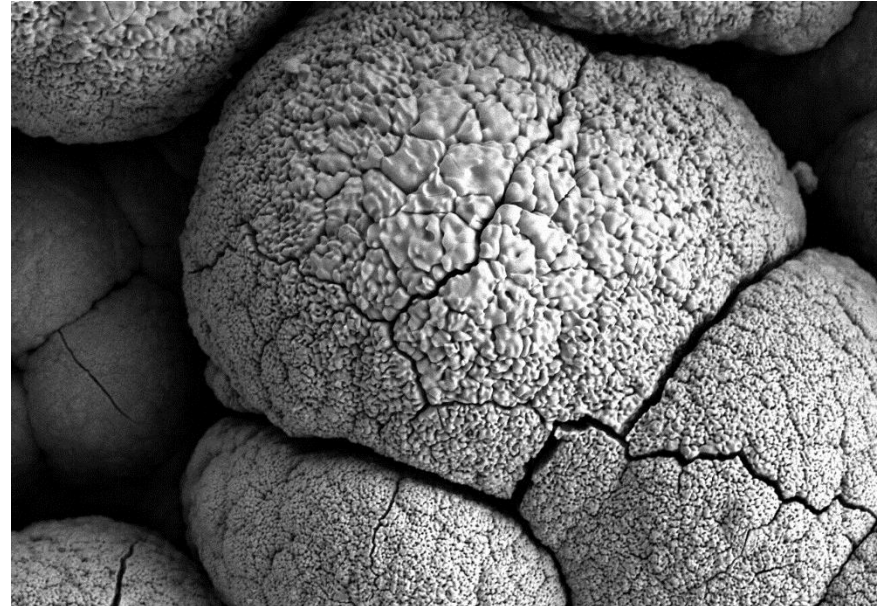
## Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - Continued

- Multicomponent rare earth silicate  $(Yb,Gd,Y)_2Si_{2-x}O_{7-2x}$  EBC Composition showed excellent stability in laser heat flux steam tests at 1500°C
- High strengths and also showed improved CMAS resistance



NBC211-4 2.0kV 5.5mm x500 SE(U,-150) 1/22/2015 100um

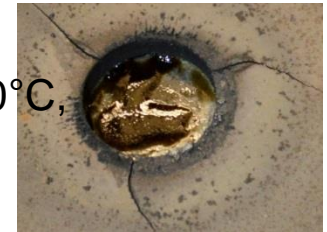
Air, 50h



N211-1#1 15.0kV 12.4mm x500 SE(L) 3/4/2015 100um

Laser Rig Steam, 200h at 1500°C

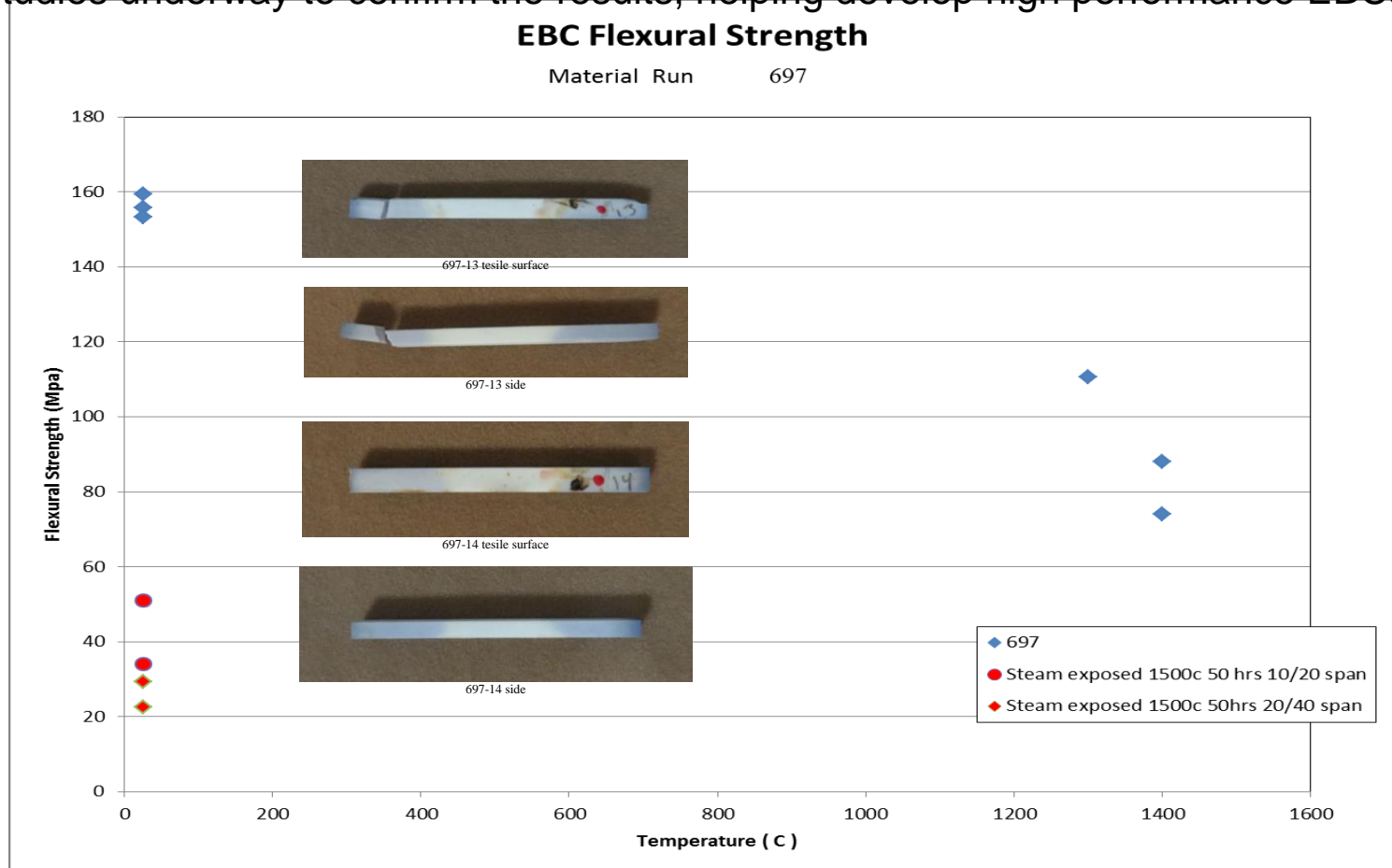
- CMAS resistance tested, 1500°C, 100h cyclic
- More advanced systems in development





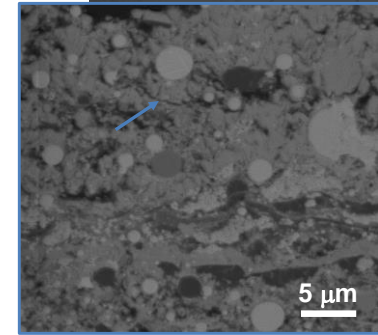
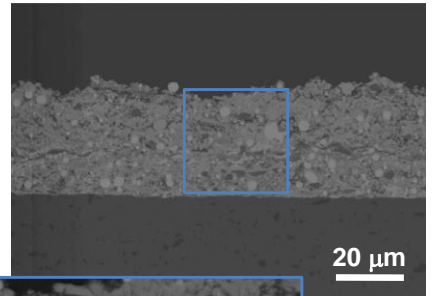
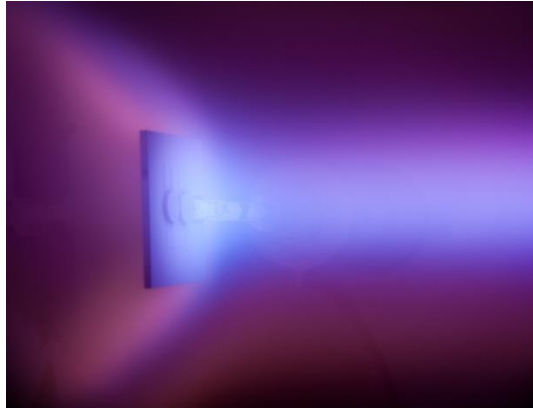
## Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - *Continued*

- NASA Rare Earth Alumino-Silicate EBC composition, a more advanced version of the EBC system, showed high toughness ( $\sim 1.8 \text{ MPa m}^{0.5}$ ) and high strength ( $\sim 160 \text{ MPa}$ ), also with improved creep resistance
- Laser steam 50 h tests showed significant strength reductions
- More studies underway to confirm the results, helping develop high performance EBCs

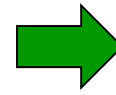


# Advanced EBC Processing- Plasma-Sprayed and EB-PVD Based Approaches

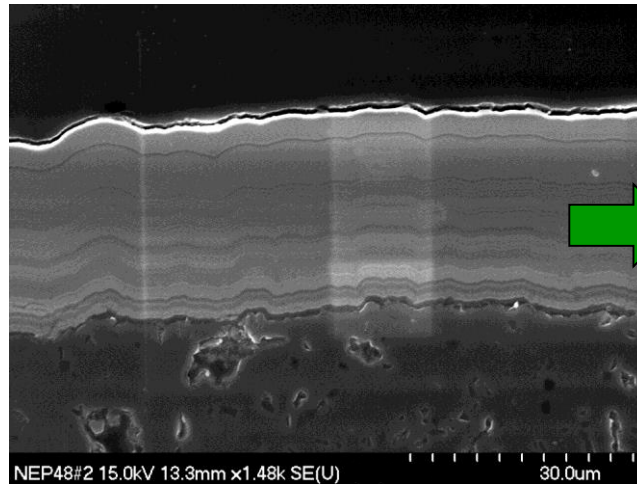
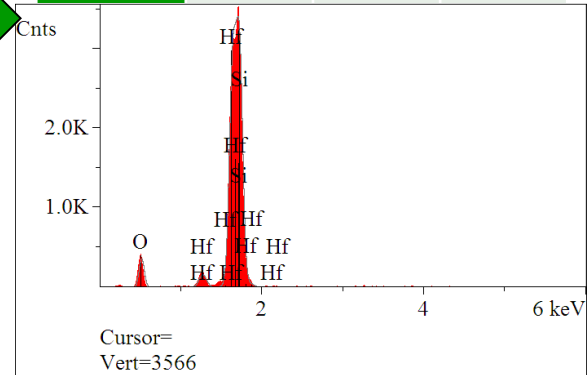
- Processed coatings had  $\text{HfO}_2$ , hafnon, and hafnium silicides nano phases



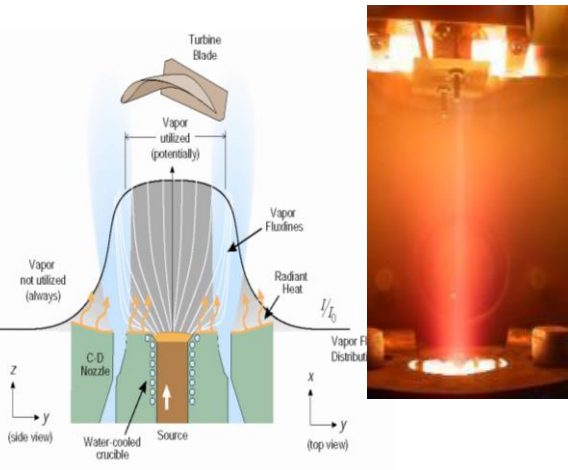
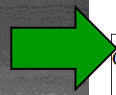
NASA PS-PVD processed  $\text{HfO}_2$ -Si EBC bond coat



$\text{HfO}_2$ -Si	At%	Wt%	Units
O	26.08	6.70	wt.%
Si	49.10	22.15	wt.%
Hf	24.82	71.15	wt.%
<b>Total</b>	<b>100.00</b>	<b>100.00</b>	<b>wt.%</b>



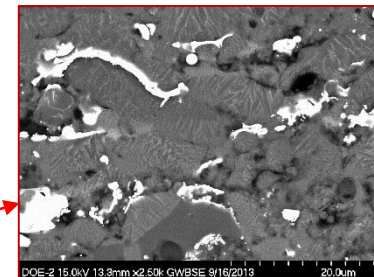
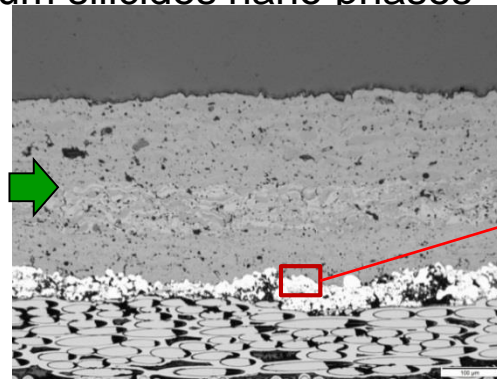
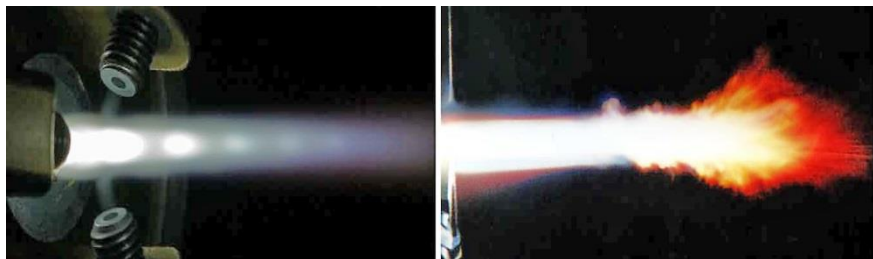
EB-PVD processed  $\text{HfO}_2$ -Si bond coat



Directed Vapor Processing systems

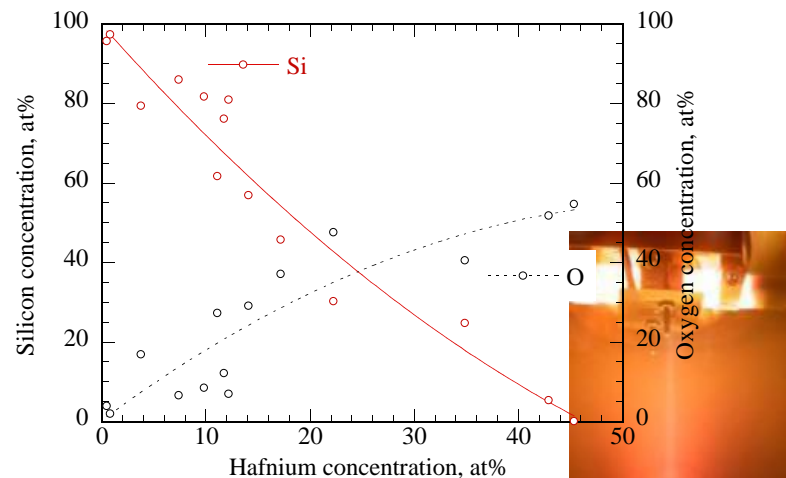
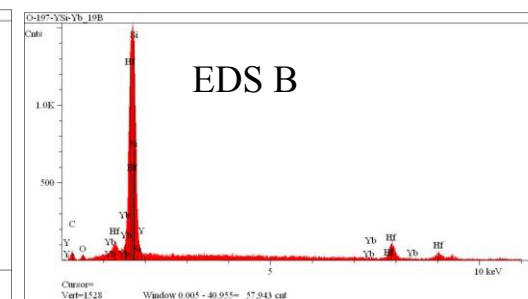
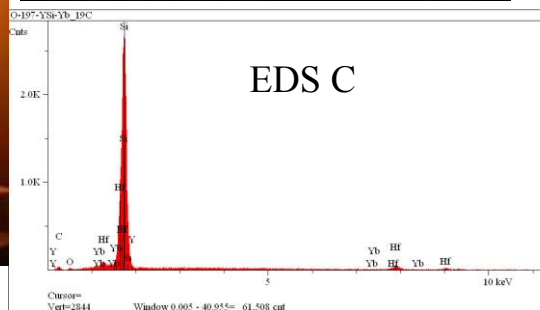
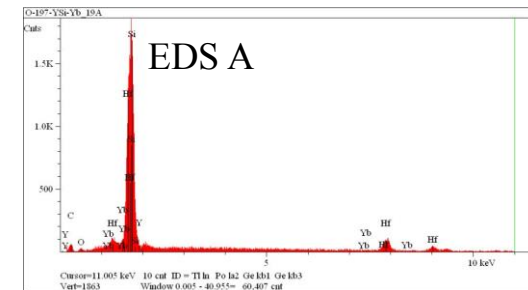
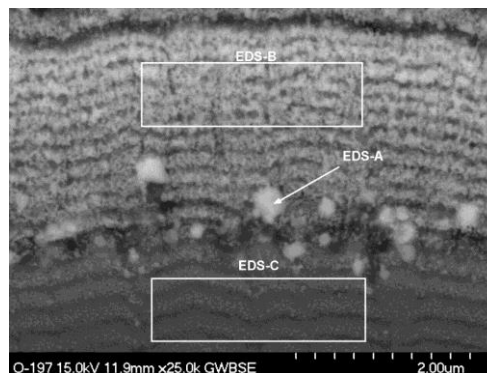
# Advanced EBC Processing- Plasma-Sprayed and EB-PVD Based Approaches - Continued

- Processed coatings had  $\text{HfO}_2$ , hafnon, and hafnium silicides nano phases

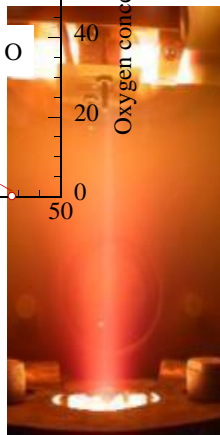


Air plasma processed  $\text{HfSiO}_x$

- Microstructure of a  $\text{HfO}_2$ -doped (Yb,Y)Si(O) bond coat



Hf-Si-O coating systems processed using EB-PVD (US Patent Applications Serial No.: 13/923,450 and 15/582,874)



Developing high toughness and high stability, low diffusion 2700°F (1482°C)  $\text{HfSiO}$  bond coats



## EBC Scaleup, and Designs High Pressure Burner Rig SiC/SiC Liner Test Configurations – SiC/SiC Liner Test Articles Setup

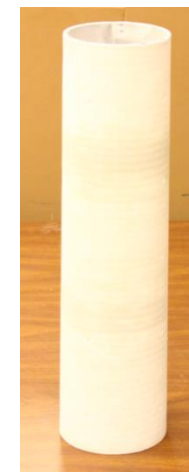
- Focused on advanced composition and processing developments using state-of-the-art techniques
- Long-term durability testing in rig environments



Inner and outer liner articles



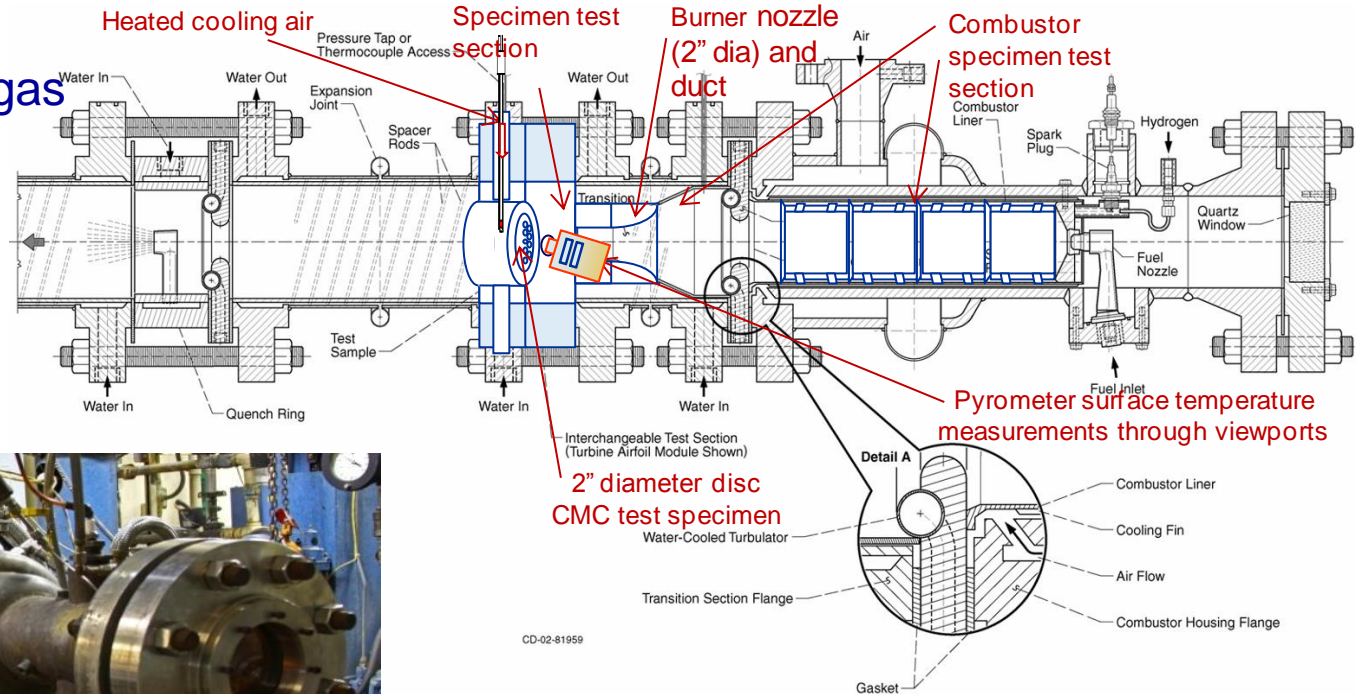
EBC coated SiC/SiC CMC Inner and Outer Liner components



# High Pressure Burner Rig SiC/SiC Liner Test Configurations

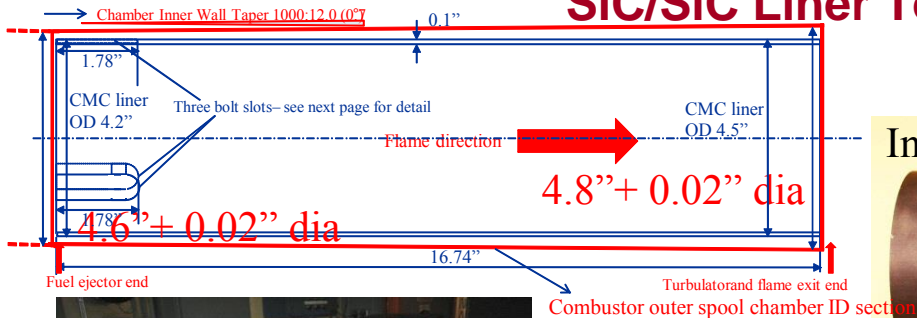
- High Pressure Burner Rig modified for realistic cooled liner subelement and liner component testing
  - Film-cooled durability and recession tests
  - EBC coated SiC/SiC CMC liner tests

- 3000°F combustor gas temperature
- 16 atm pressure
- Heated cooling air
- Up to 300 m/s gas velocity used

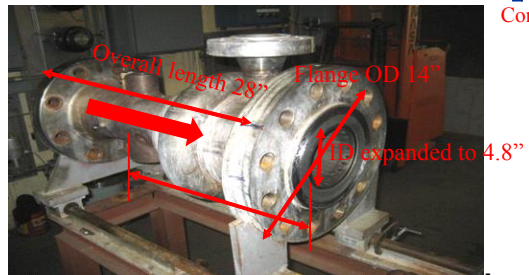


High pressure burner rig

# High Pressure Burner Rig SiC/SiC Liner Test Configurations – SiC/SiC Liner Test Articles Setup



Inner liner with instrumentation TC holes

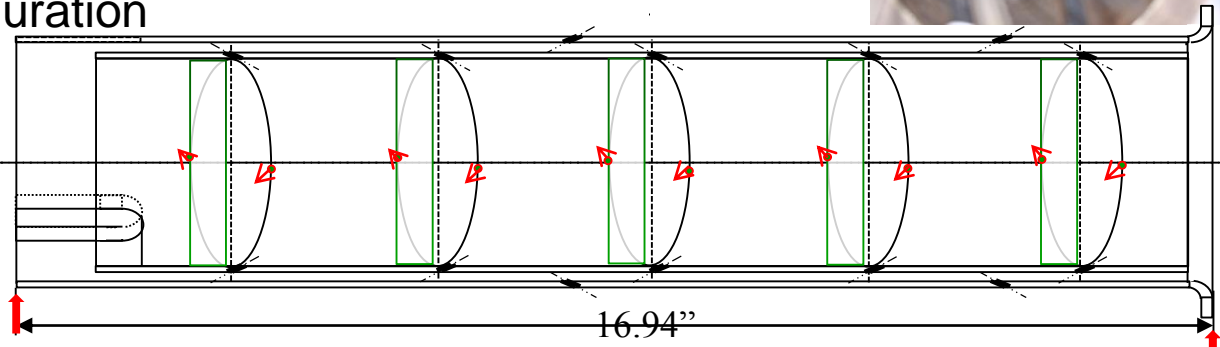


Combustion outer chamber modified to accommodate increased dimension and tapered configuration

Injector and turbulator modified for CMC liner integration and testing



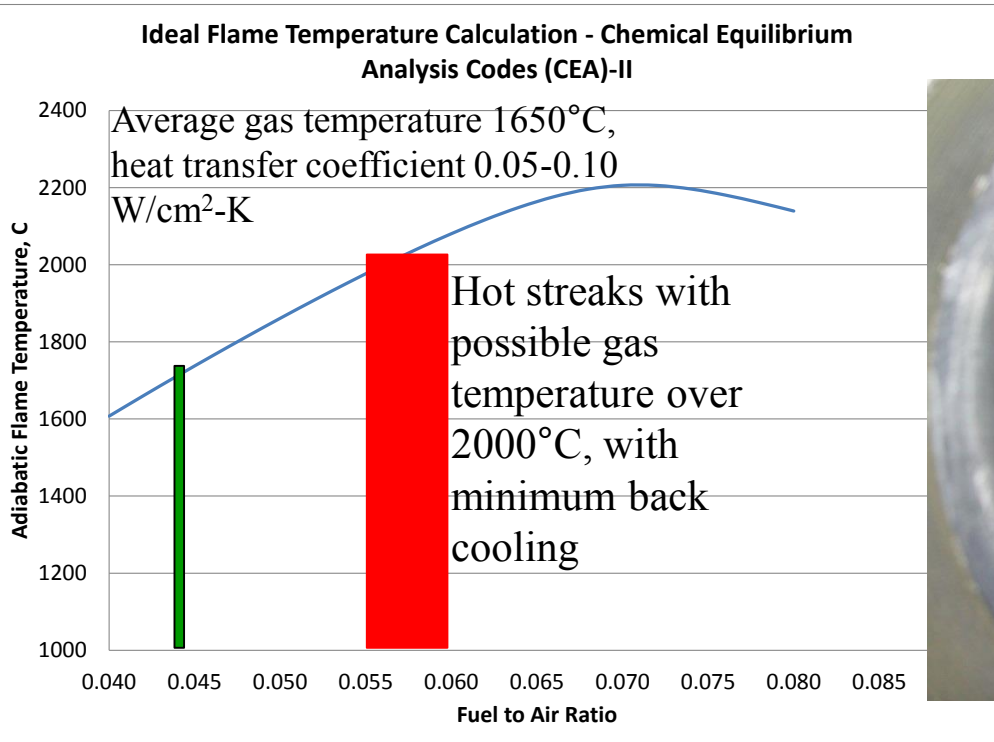
TC bundle picture



First set SiC/SiC liner Thermocouple (TC) arrangement configurations (total 24 TCs, 1/16" size), film cooled liner planned for second and third set testing

## The Prepreg SiC-SiC CMC Combustor Liners Successfully Tested for 50h Durability in NASA High Pressure Burner Rig up to 3600°F

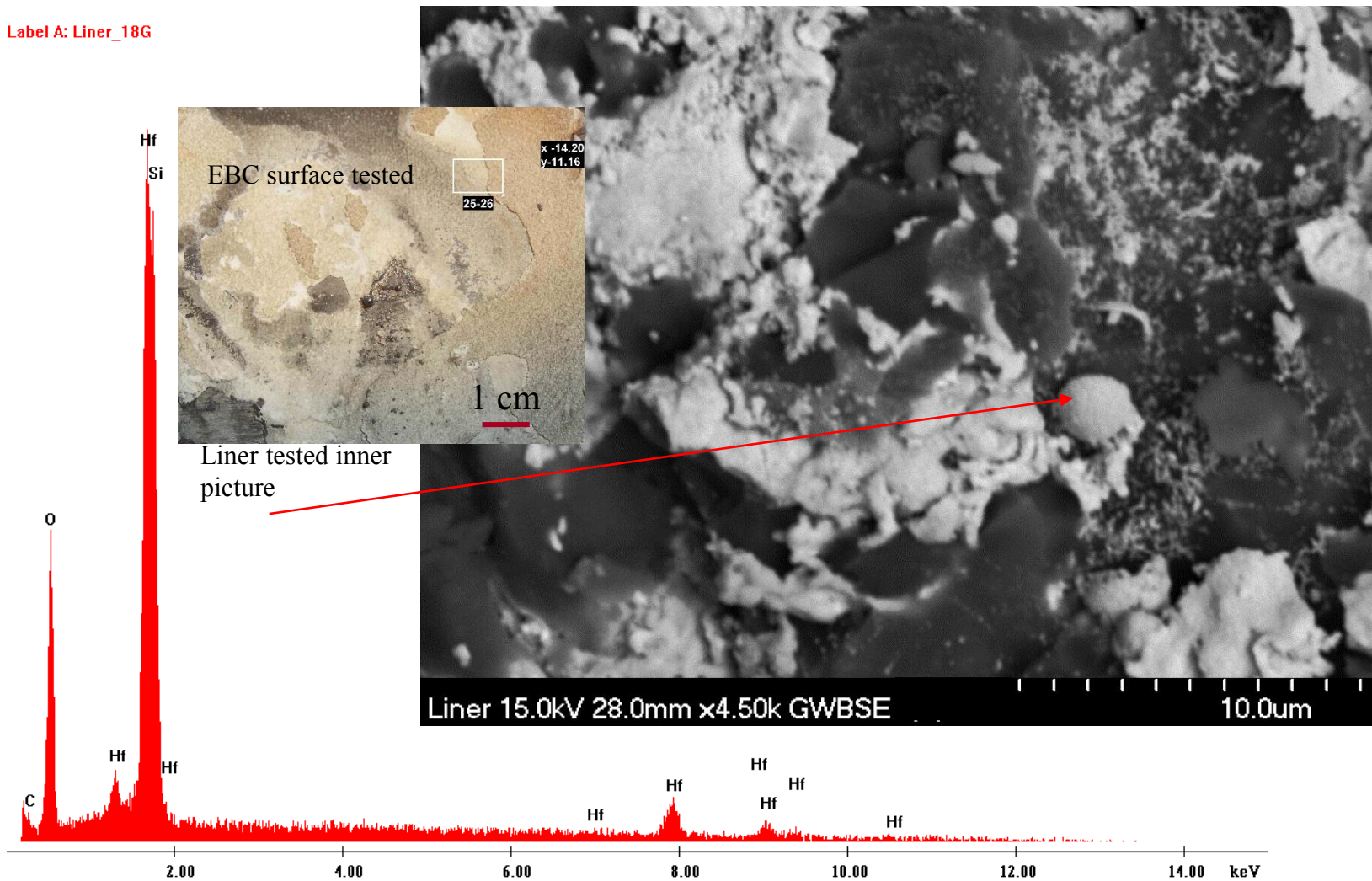
- Tested pressures at 500 psi external for outliner, and 220 psi inner liners in the combustion chamber (16 atm)
- Average gas temperatures at 3000°F (1650°C), the liner EBCs tested at 2500-2600°F with heat fluxes 20-35 W/cm<sup>2</sup>, and the CMC liner component at 1800-2100°F
- Hot gas streaks may have had temperatures over 3632°F (2000°C), with higher transfer coefficients
- SiC/SiC CMC liners and EBCs survived 255 h



Some minor coating spalling at hot streak impingement during testing

# The Prepreg SiC-SiC CMC Combustor Liners Successfully Tested for 50h Durability in NASA High Pressure Burner Rig up to 3600°F - Continued

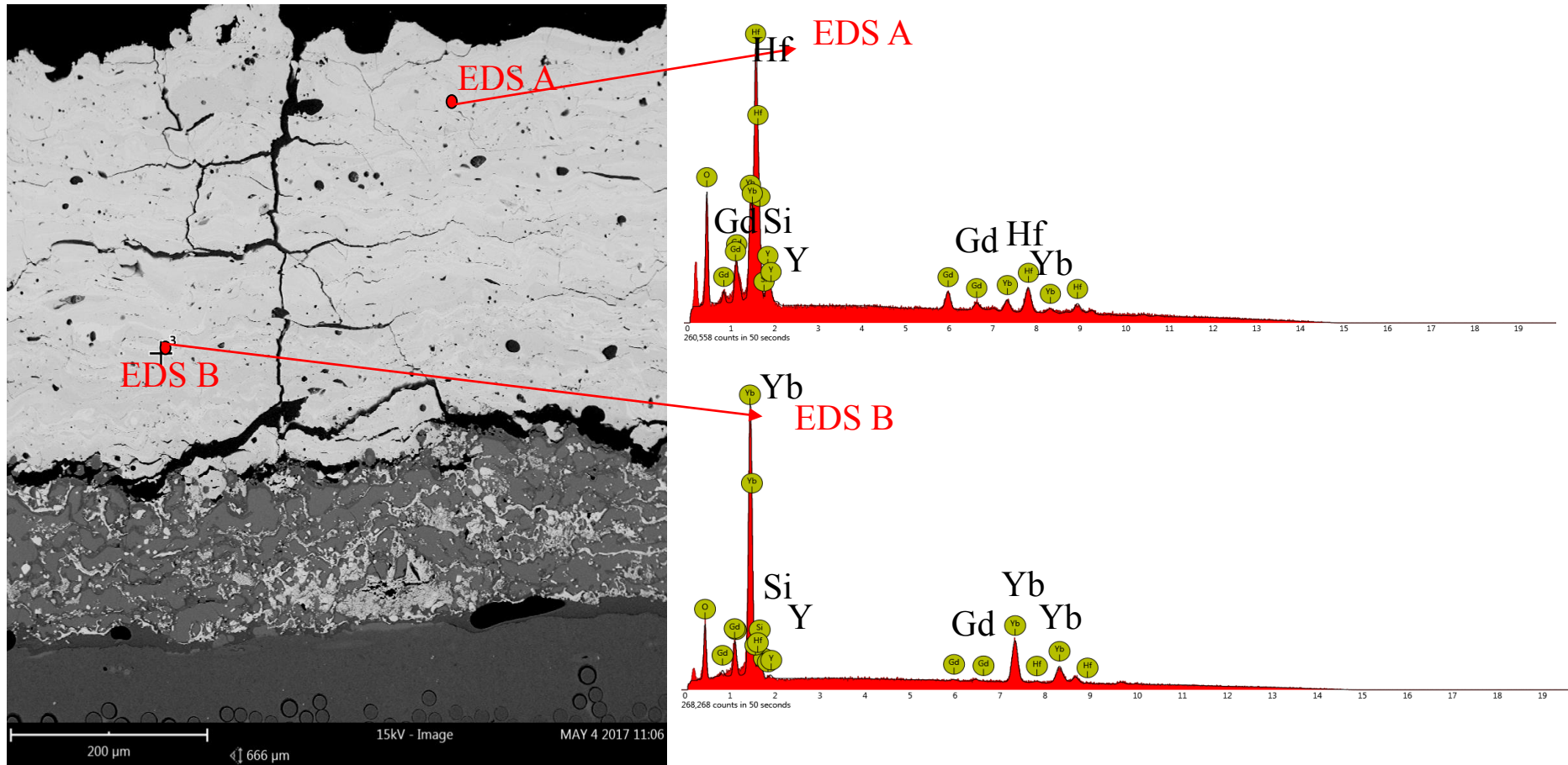
Label A: Liner\_18G



Hafnium-rich phase melting in the hottest liner section confirms the locally very high temperatures

## Some Observed Degradations after 250 h Tests

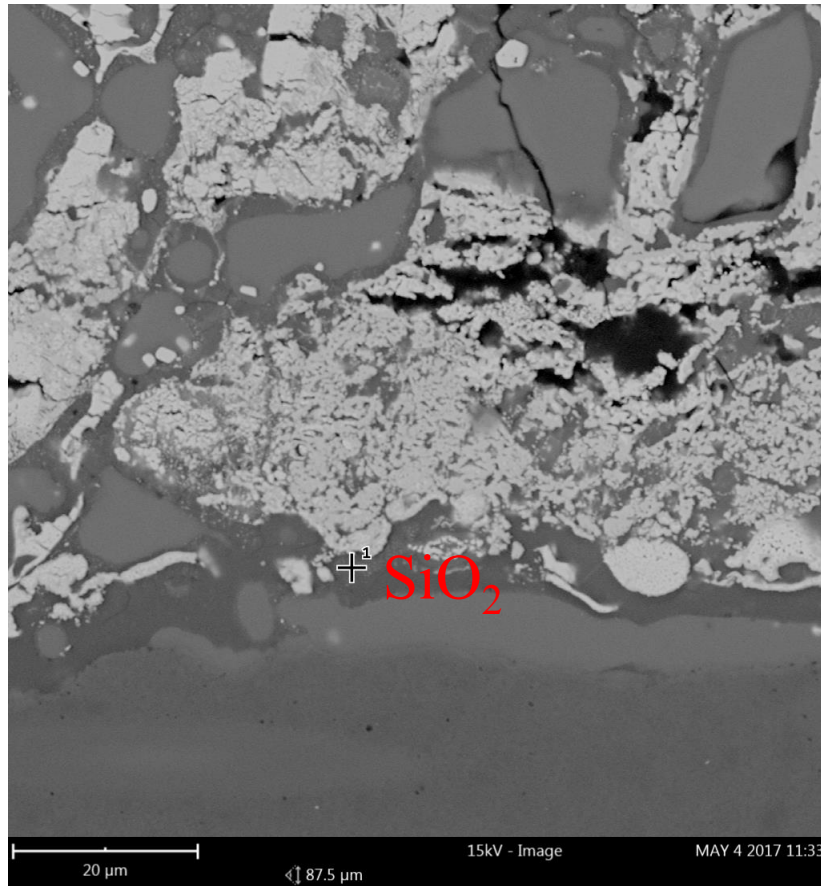
- Observed EBC delamination, possibly under combined thermal and mechanical loading in one of the most severe condition tested sections
- Plasma sprayed  $\text{HfO}_2$ -Si bond coat showed better adhesion and durability



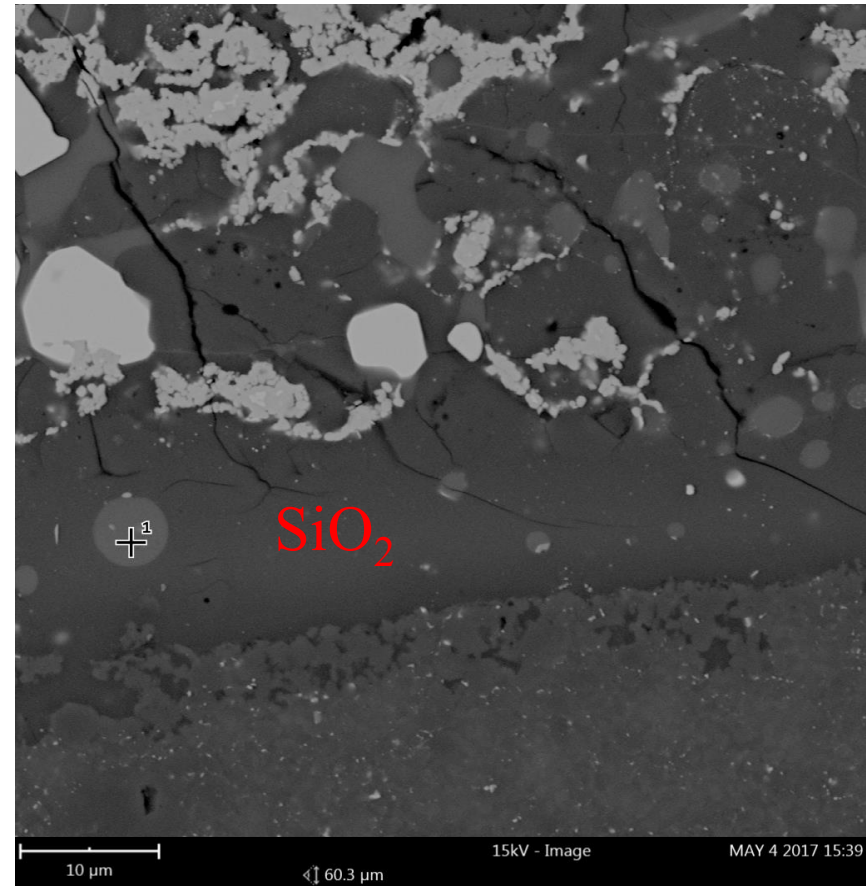
RE silicates and Cubic phase EBC may not have sufficient strength and toughness

## Some Observed Degradations after 250 hr Tests - Continued

- Plasma sprayed  $\text{HfO}_2$ -Si bond coat showed good adhesion and durability
- Some silica formation at the bond coat/CMC interface after 250hr tests



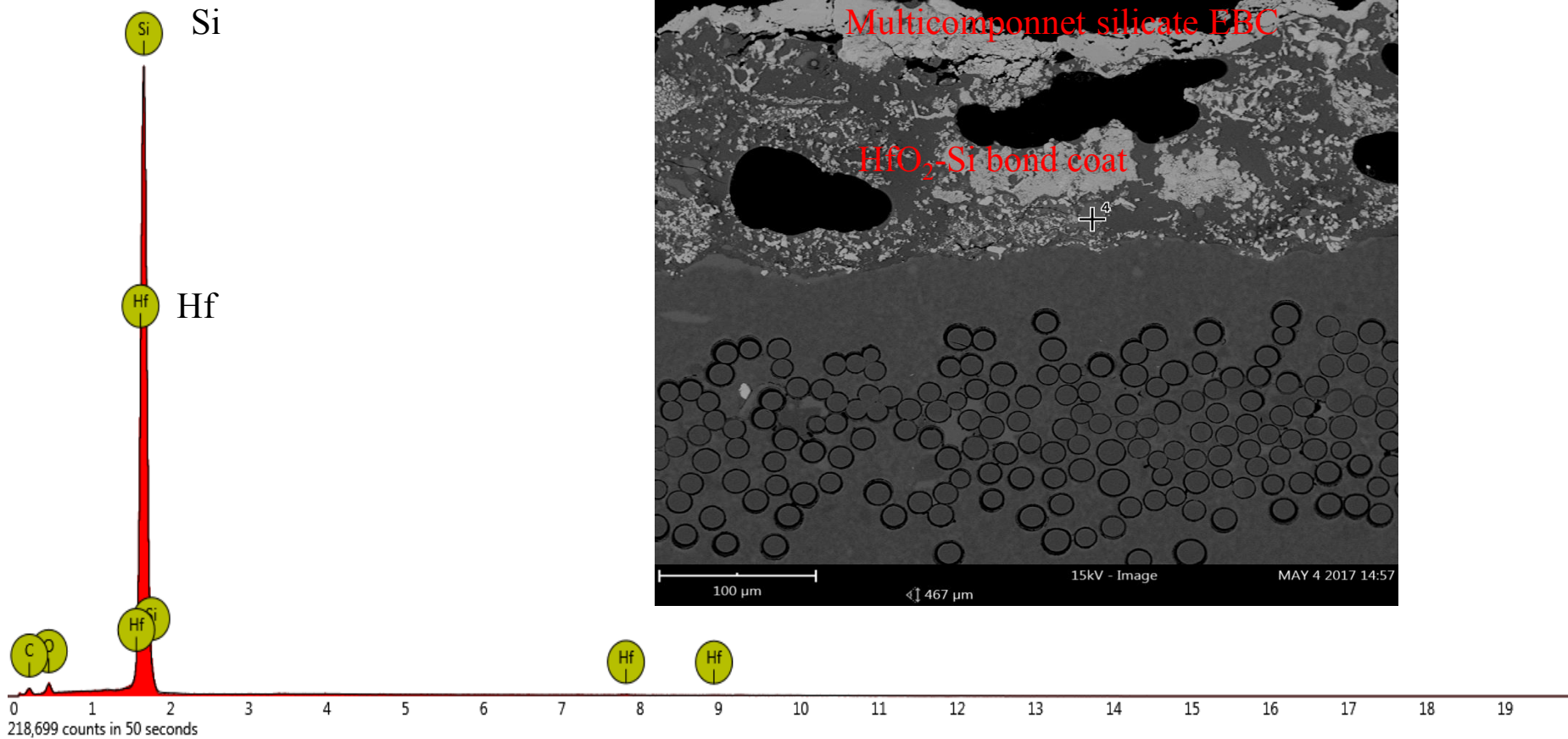
Lower temperature region



Higher temperature region

## Some Observed Degradations after 250 hr Tests - Continued

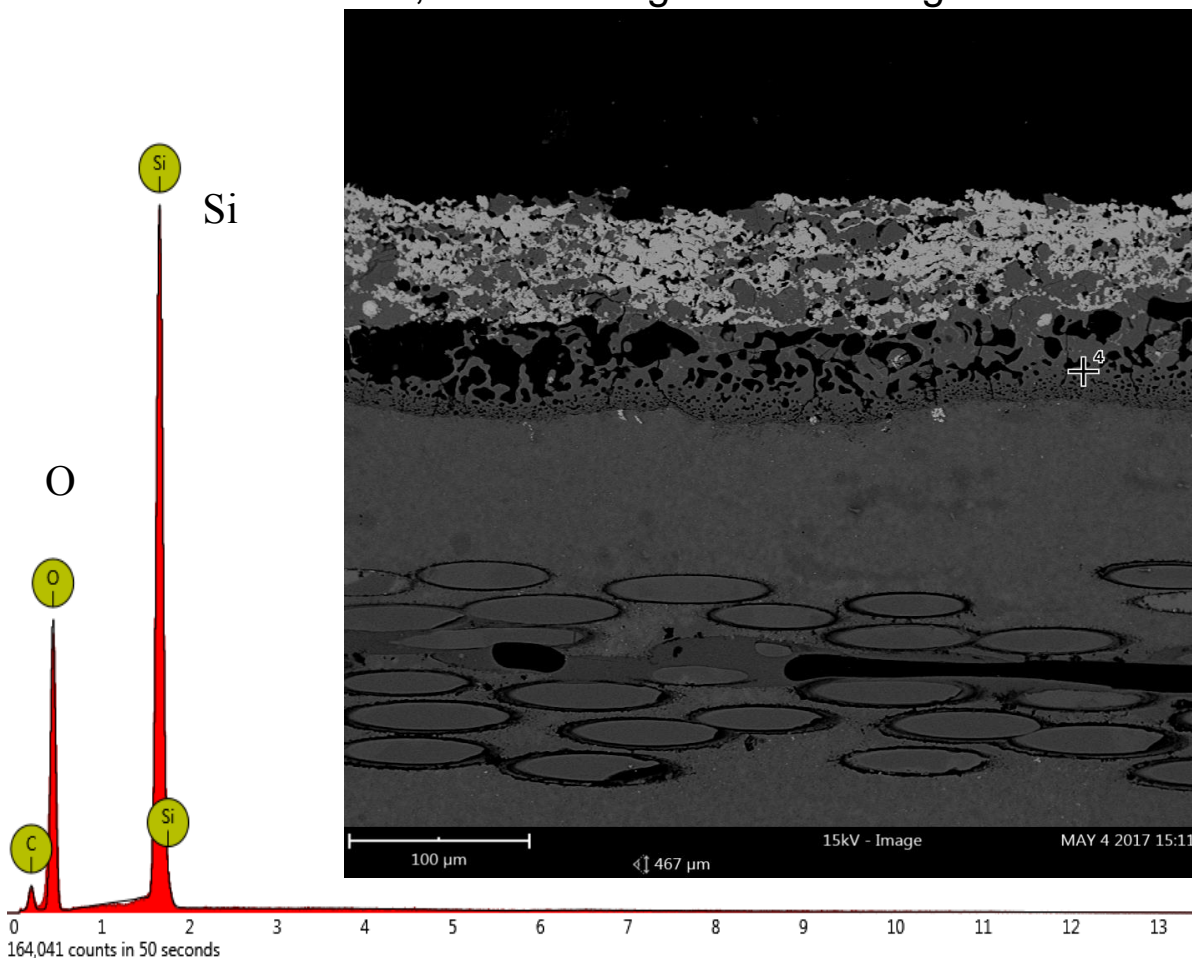
- Plasma sprayed  $\text{HfO}_2$ -Si bond coat showed good adhesion and durability
- Maintained low oxygen content at the bond coat – CMC interface even at very high temperature regions
- Some fiber degradations observed





## Some Observed Degradations after 250 hr Tests - Continued

- Observed degraded bond coat region and with more extensive C containing SiO<sub>2</sub> scale formation
- More severe fiber, fiber coating and CMC degradations



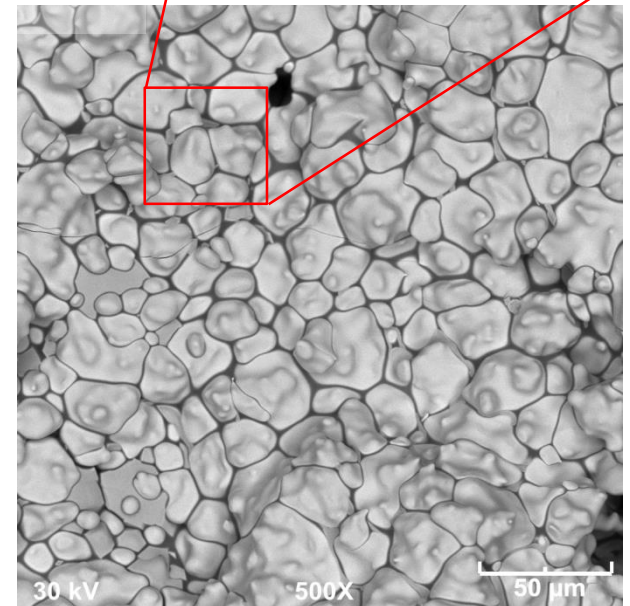
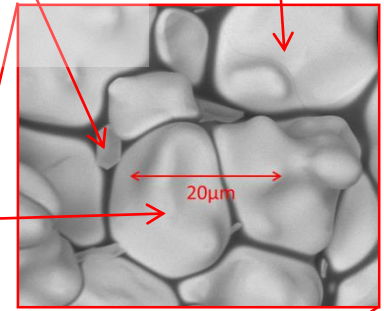
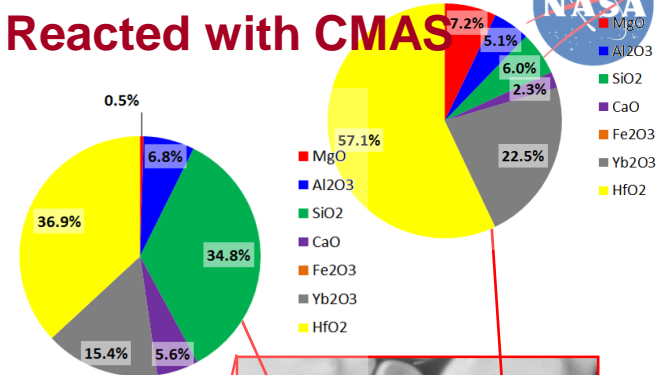
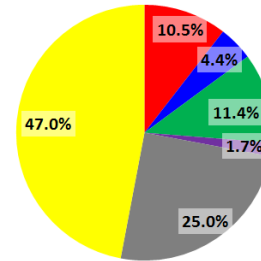
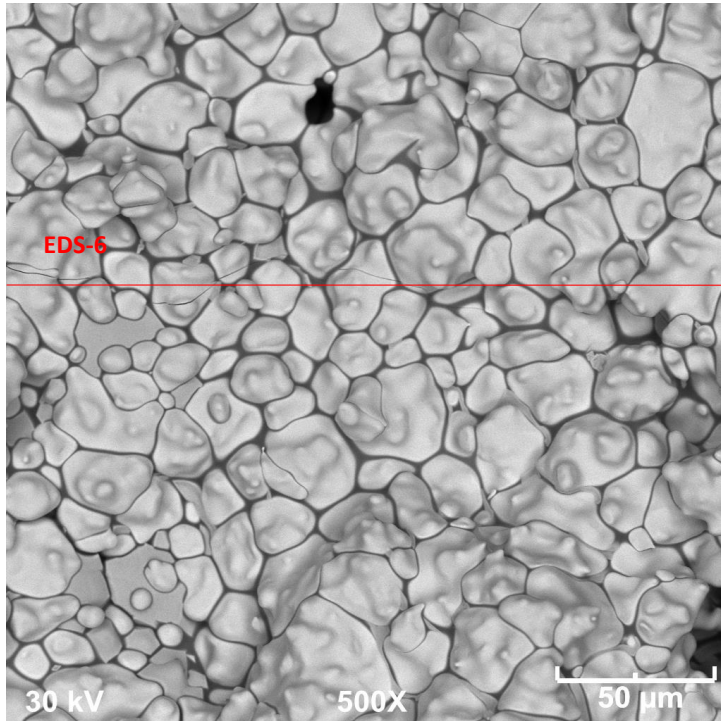
Element Symbol	Atomic Conc.	Weight Conc.
O	55.25	50.90
Si	19.62	31.73
C	25.13	17.38

# CMAS Resistance of the Advanced EBCs: Reacted with CMAS 1500°C, 100h

- The Advanced EBC generally developed showed low Rare Earth (RE) dissolution and thus better CMAS resistance

EDS-6 Line scan

Component	Type	Mole Conc.	Conc.	wt.%
MgO	Calc	7.041	1.426	wt.%
Al <sub>2</sub> O <sub>3</sub>	Calc	4.614	2.364	wt.%
SiO <sub>2</sub>	Calc	17.999	5.435	wt.%
CaO	Calc	3.808	1.073	wt.%
Yb <sub>2</sub> O <sub>3</sub>	Calc	20.925	41.445	wt.%
HfO <sub>2</sub>	Calc	45.613	48.256	wt.%
		100.000	100.000	wt.%





## Summary

- Advanced EBCs developed, evaluated for 2700-3000°F CMC combustor liner applications in NASA high pressure burner rig
  - Valuable test data obtained on EBCs and CMCs
  - Several new compositions evaluated and developed
- Multicomponent EBC showed promise for high temperature capability, steam and combustion environment stability, and initial CMAS resistance
  - Bond coat composition optimization being optimized and also for commercial applications
  - Rare earth –Si bond coats also developed
- The EBC – SiC/SiC liner component demonstrated initial durability in very harsh test conditions, improved the Technical Readiness Levels under the NASA programs
- Property data and EBC Failure modes also studied



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