Development of Advanced Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites: Path toward 2700°F Temperature Capability and Beyond

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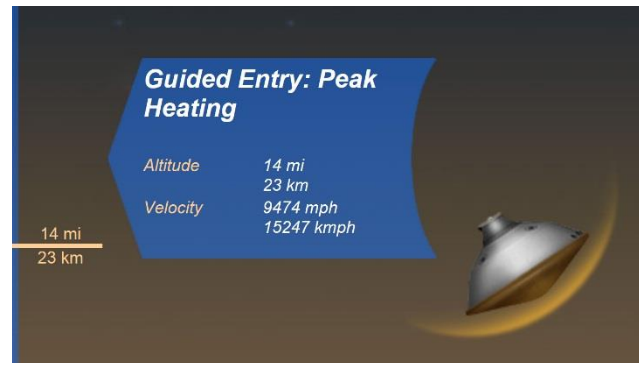
Durable Environmental Barrier Coating Systems for Ceramic Matrix Composites (CMCs):

Enabling Technology for Next Generation Low Emission, High Efficiency and Light-Weight Propulsion, and Extreme Environment Material Systems

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

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

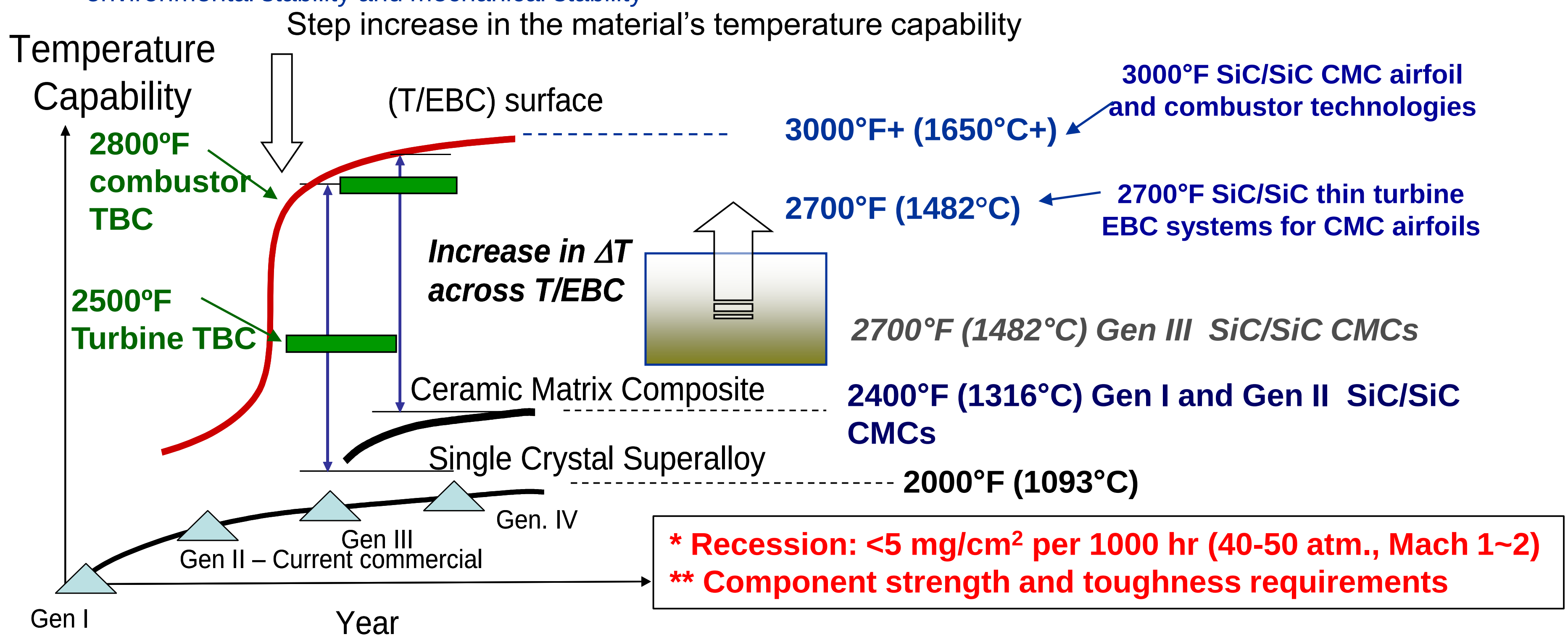
Fixed Wing Subsonic and Supersonics Aircraft:
- Transformational Tools and Technologies Project



Entry, Descending and Landing: Ultra High Ceramics and Coatings (UHTCC) - NASA CIF Project

NASA Environmental Barrier Coating Development Goals

- Emphasize temperature capability, performance and durability
- Develop innovative coating technologies and life prediction approaches
- 2700°F (1482°C) EBC bond coat technology for supporting next generation
- 2700-3000°F (1482-1650°C) turbine and CMC combustor coatings
    - Recession: <5 mg/cm^2 per 1000 h
    - Coating and component strength requirements: 15-30 ksi, or 100- 207 MPa
    - Resistance to Calcium Magnesium Alumino-Silicate (CMAS), impact and erosion
    - Demonstrate feasibility towards *Ultra High* Temperature and Multifunctional Ceramics – Coating Systems: improved environmental stability and mechanical stability



Outline

- **Advanced EBC and Rare Earth – Silicon based 2700°F+ capable bond coat developments**
  - Material systems
  - Oxidation resistance
  - Cyclic and thermomecahnical durability
  - Some bench mark durability tests for 2700°F EBC systems
- **Ultra High Temperature and Multifunctional Ceramic Matrix Composite – Coating Systems for Light-Weight Space and Aero Systems**
  - HfCN based system with Si and RE dopant concepts
  - Develop HfO_2 – Si based coatings for improved oxidation resistance
- **Summary and conclusion**

Environmental Barrier Coating Development: Challenges and Limitations

- EBCs limited in their temperature capability, water vapor stability and long-term durability

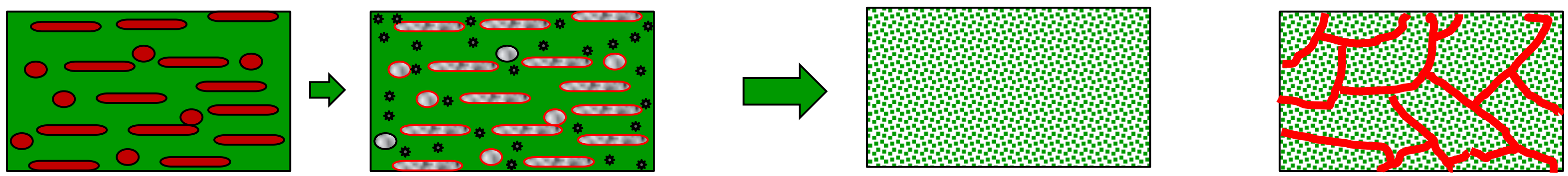
- Advanced EBCs also require higher strength and toughness
  - In particular, resistance to combined high-heat-flux, engine high pressure, combustion environment, creep-fatigue, loading interactions

- EBCs need improved erosion, impact and calcium-magnesium-alumino-silicate (CMAS) resistance

- Also possibly developed to Ultra High Temperature Ceramics applications

Advanced High Temperature and 2700°F+ Bond Coat Development

– NASA advanced EBC Development:
- Advanced compositions ensuring environmental and mechanical stability
- Bond coat systems for prime reliant EBCs; capable of self-healing



High strength, high stability reinforced composites: HfO_2-Si and a series of Oxide-Si systems

HfO_2-Si based and minor alloyed systems for improved strength and stability, e.g., rare earth dopants

Advanced 2700°F bond coat systems: RE-Si based systems

Advanced 2700°F bond coat systems: RE-Si based Systems, grain boundary engineering designs and/or composite systems

HfO_2-Si systems

Advanced 2700°F+ Rare Earth - Si Bond Coat systems

Temperature capability increase

Rare Earth – Si + Hf coating systems

Hf – Rare Earth – Si coating systems

NASA EBC Bond Coats for Airfoil and Combustor EBCs

– Patent Application 13/923,450 PCT/US13/46946, 2012

– Advanced systems developed and processed to improve Technology Readiness Levels (TRL)
– Composition ranges studied mostly from 50 – 80 atomic% silicon
– Silicon and dopant composition being optimized for 2700°F EBC applications

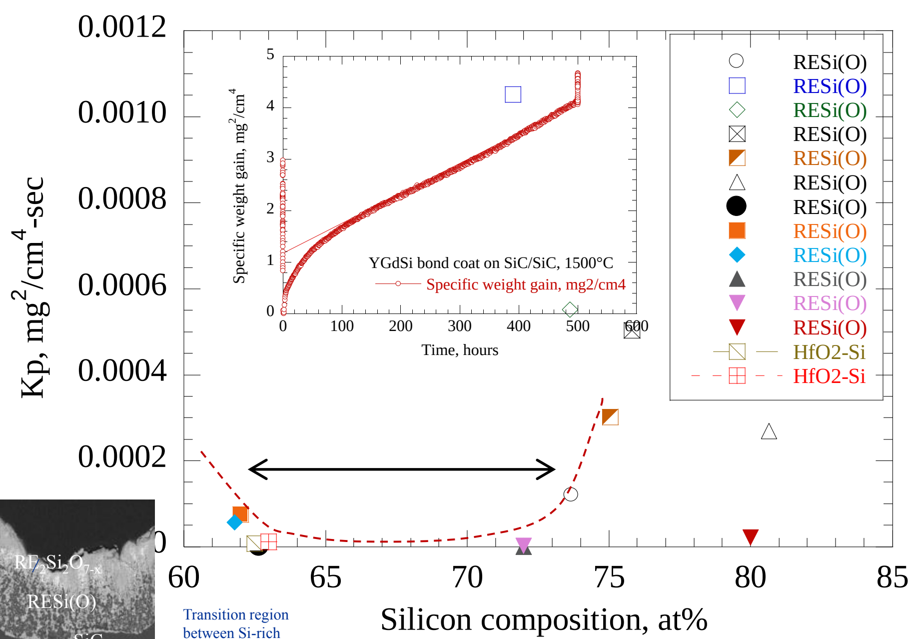
PVD-CVD			EB-PVD	APS*		FurnaceLaser/CVD/PVD
YSi	YbGdYSi	GdYSi	HfO2-Si;	HfO_2-Si		
ZrSi+Y	YbGdYSi	GdYSi	REHfSi	YSi+RESilicate		REHfSi
ZrSi+Y	YbGdYSi	GdYSi	GdYSi	YSi+Hf-RESilicate		
ZrSi+Ta	YbGdYSi	GdYSi	GdYbSi			
ZrSi+Ta	YbGdSi	GdYSi-X	GdYb-LuSi		Used in ERA components as part of bond coat system	
HfSi + Si	YbGdSi	GdYSi-X	NdYbSi	Hf-RESilicate		
HfSi + YSi	YbGdSi					
HfSi+Ysi+Si	YbGdSi					
YbSi	YbGdSi					
	YbSi			Hf-RE-Al-Silicate	Used in ERA components as part of bond coat system	
HfSi + YbSi						
GdYbSi(Hf)						
YYbGdSi(Hf)	YbYSi					
	YbHfSi					
	YbHfSi					
	YbHfSi					
	YbHfSi					
	YbHfSi					
	YbSi					

Process and composition transitions

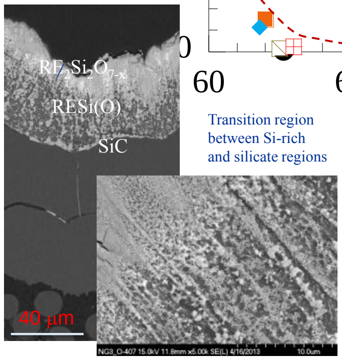
APS*: or plasma spray related processing methods

Oxidation Kinetics and Furnace Cyclic Durability of RESi EBC Bond Coats for 2700°F SiC/SiC CMC Systems

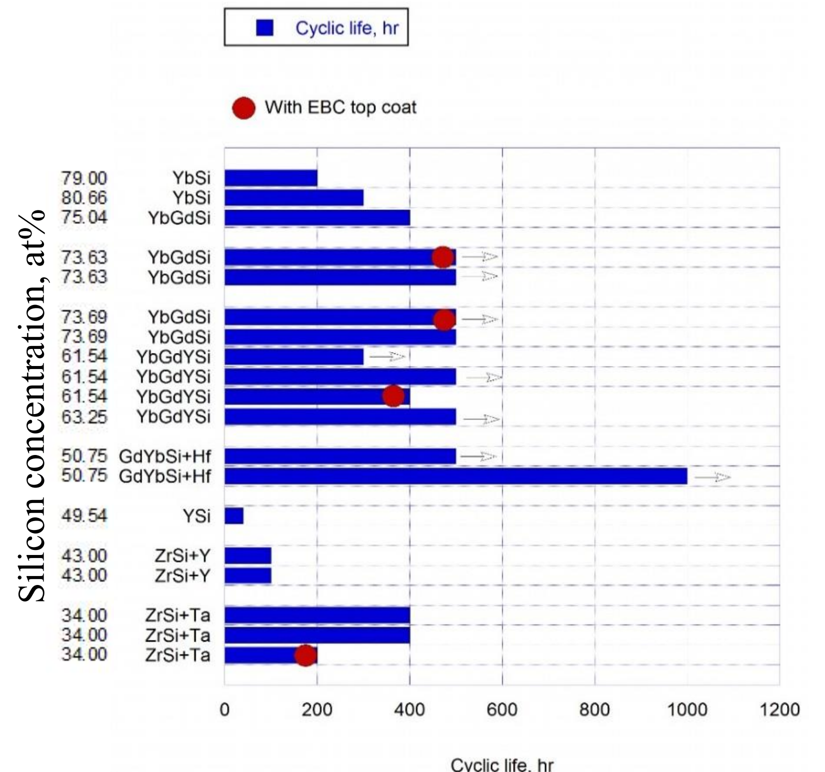
- 1500°C (2700°F+) capable RESiO+X series EBC bond coat compositions developed for turbine engine coatings
- Oxidation kinetics in flowing O_2 showed parabolic or pseudo-parabolic oxidation behavior at high temperatures
- Some early multi-component systems showed significantly improved furnace cyclic durability at 1500°C



Oxidation kinetics



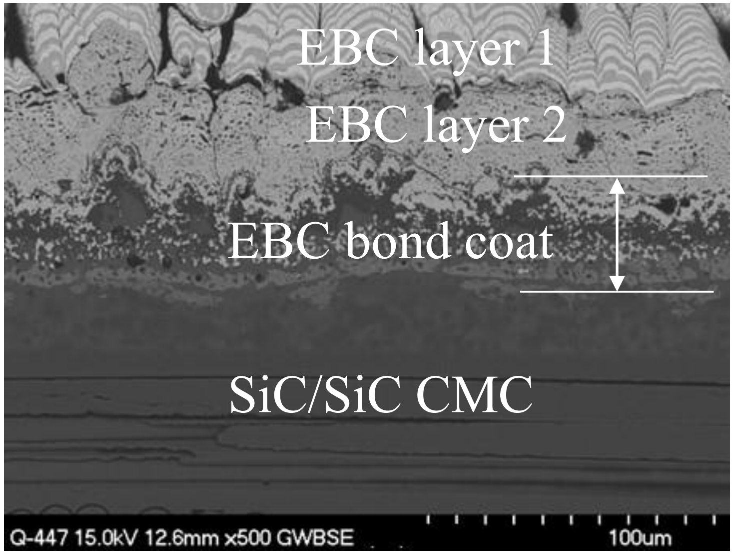
Cross-section TGA tested specimen



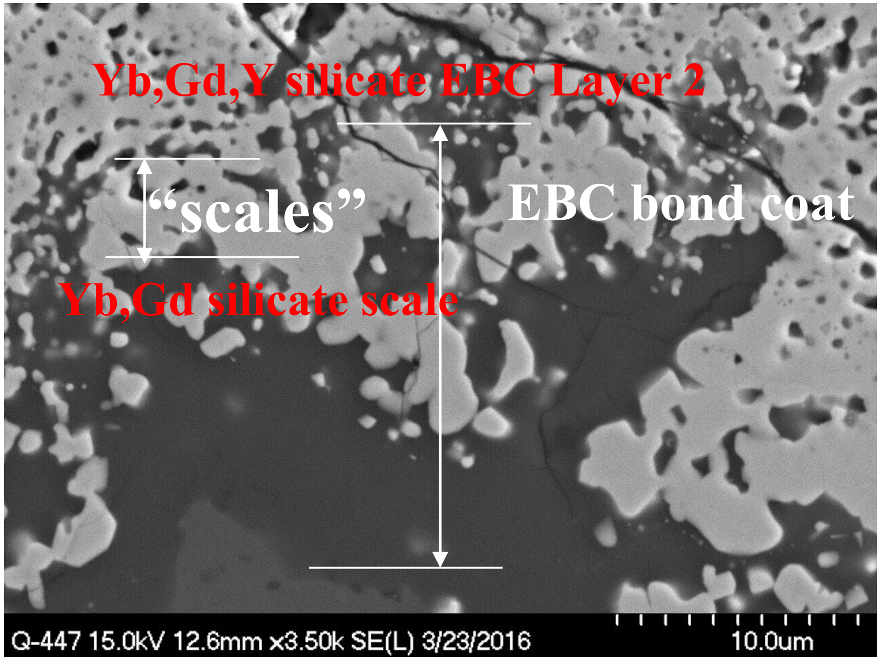
FCT life, Testing in Air at 1500°C, 1 hr cycles

Microstructures of the Advanced EBCs after the Oxidation Tests

- RE-Si system: forming RE silicate “scales”, fully compatible with EBCs
- Reaction and oxidation mechanisms are being further studied, particularly RE containing SiO_2 – rich phase stability
- Further process improvements can help improve the oxidation resistance and durability

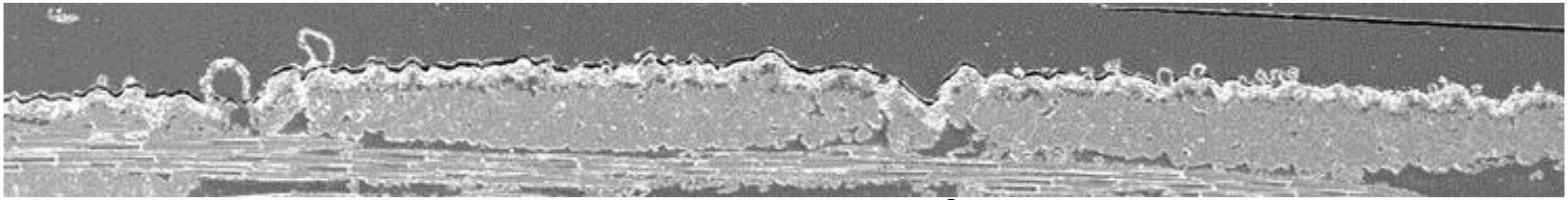


Cross-section micrograph of YbGdSi(O) tested at 1500°C, 500hr

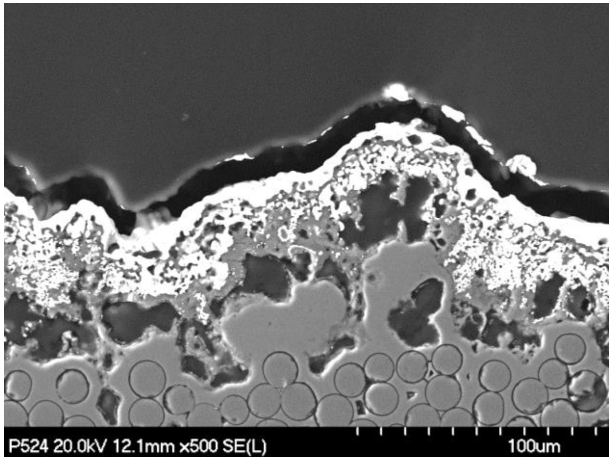


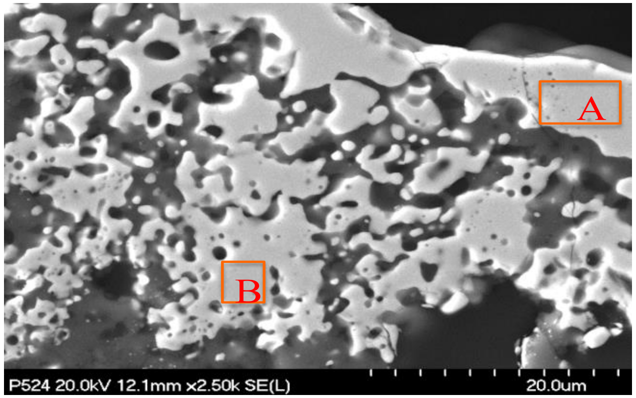
Microstructures of Furnace Cyclic Tested GdYbSi(O) EBC Systems

— Cyclic tested cross-sections of PVD processed YbGdSi(O) bond coat
— Self-grown rare earth silicate EBCs and with some RE-containing SiO_2 rich phase separations
— Relatively good coating adhesion and cyclic durability



1500°C, in air, 500, 1 hr cycles





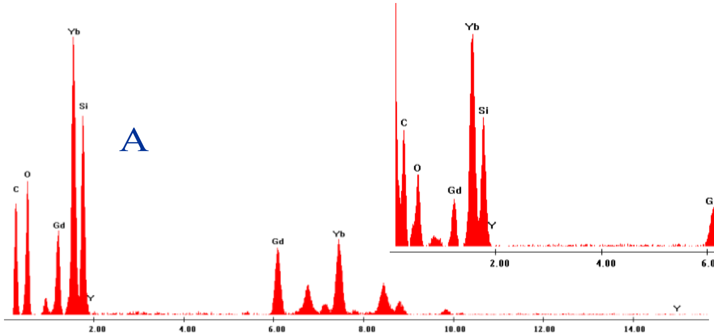
- Complex coating architectures after the testing
- Designed with EBC like compositions – Self-grown EBCs

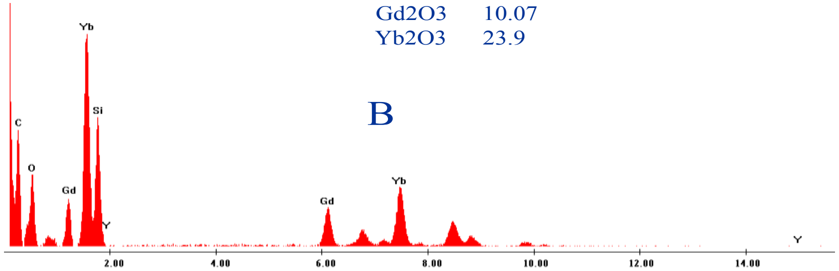
Composition (mol%) spectrum Area #1

SiO2	67.98
Gd2O3	11.95
Yb2O3	20.07

Composition (mol%) spectrum Area #2

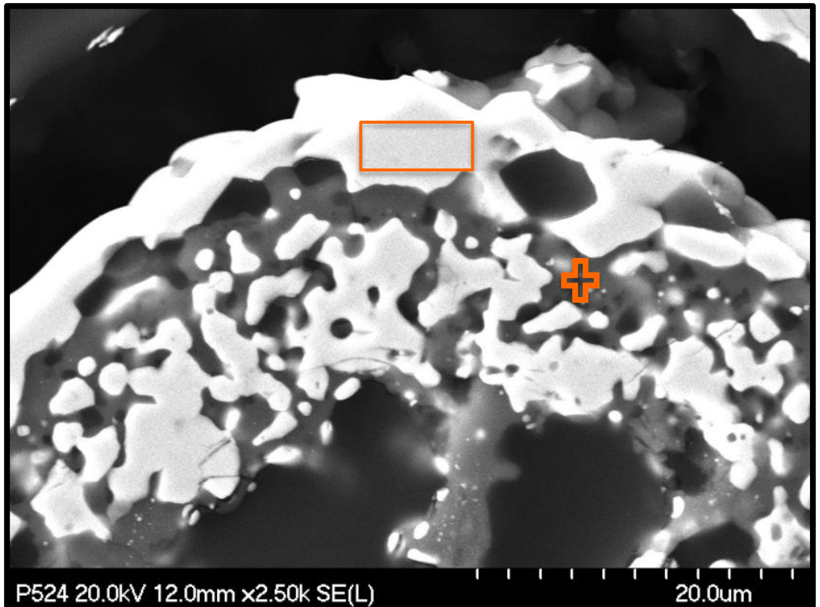
SiO2	66.03
Gd2O3	10.07
Yb2O3	23.9





Microstructures of Cyclic Tested GdYbSi(O) EBC Systems- Continued

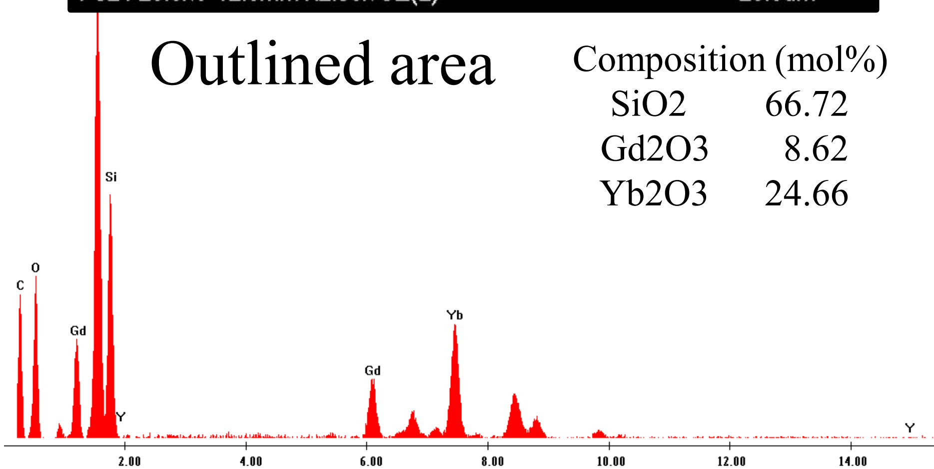
- Cyclic tested cross-sections of PVD processed YbGdSi(O) bond coat
- Self-grown rare earth silicate EBCs and with some RE-containing SiO_2 rich phase separations
- Relatively good coating adhesion and cyclic durability



1500°C, in air, 500, 1 hr cycles

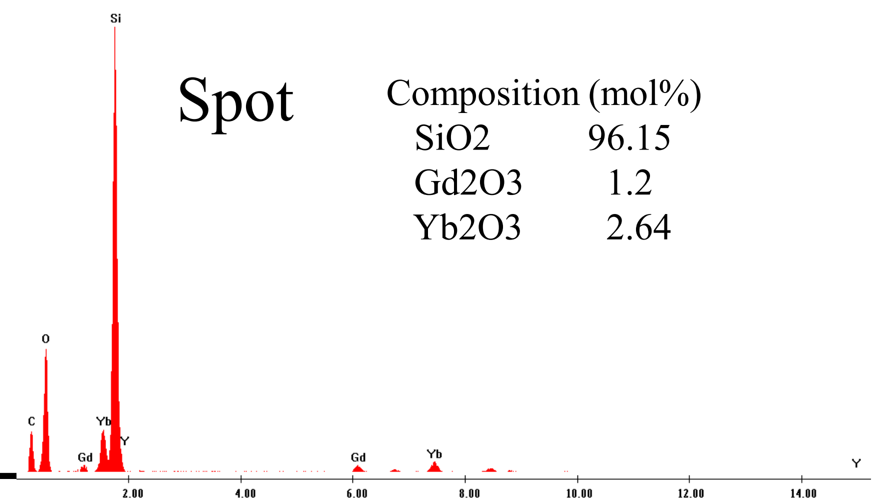
Outlined area

Composition (mol%)	
SiO2	66.72
Gd2O3	8.62
Yb2O3	24.66



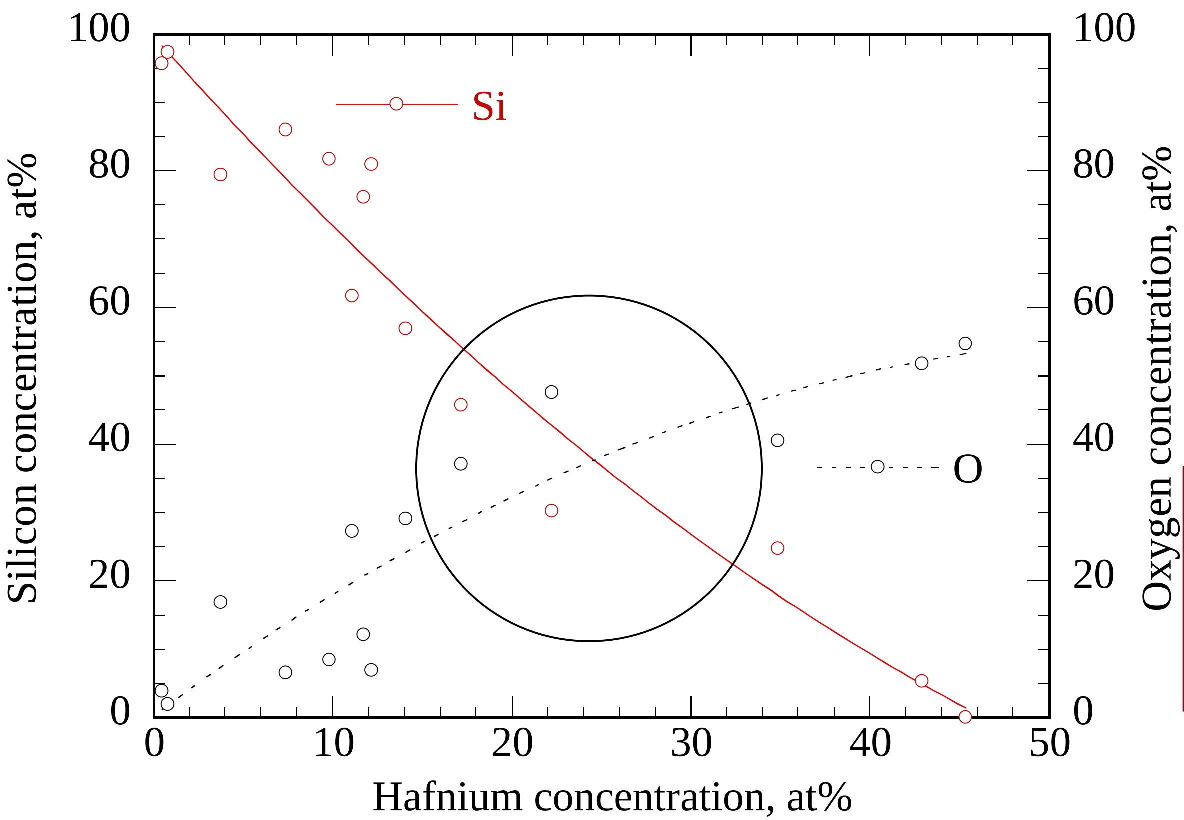
Spot

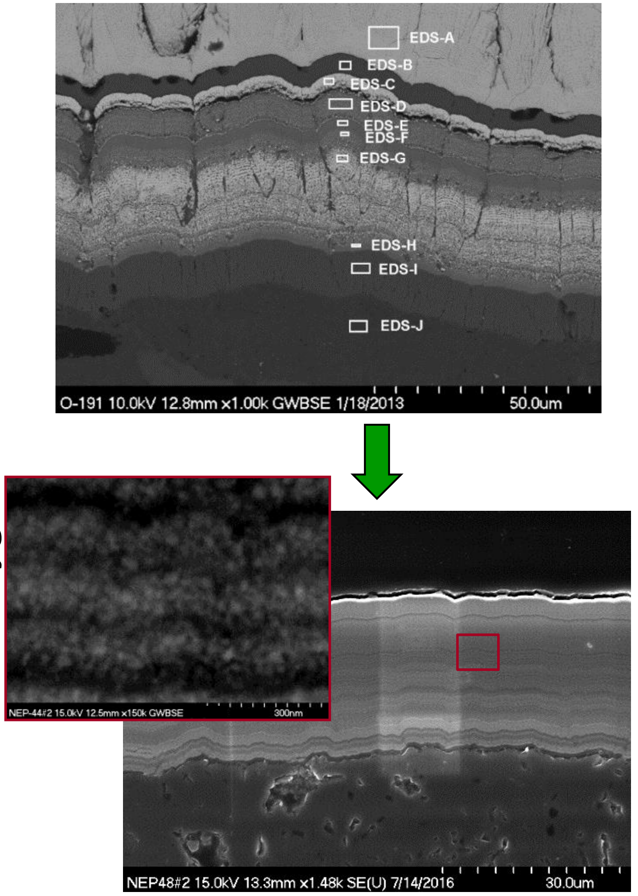
Composition (mol%)	
SiO2	96.15
Gd2O3	1.2
Yb2O3	2.64



HfO_2-Si Bond Coats Processing and Composition Optimizations

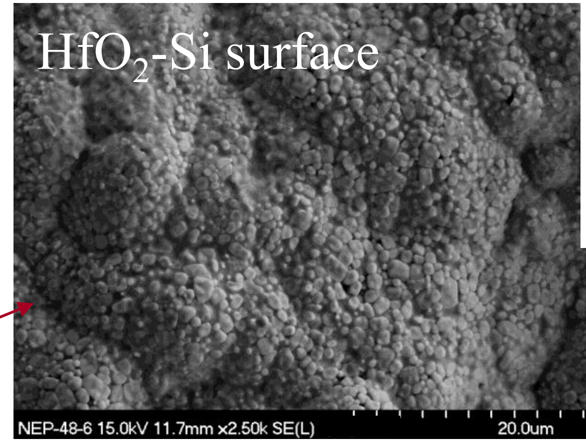
- EB-PVD HfO_2-Si bond coat process and composition extensively studied
- Achieving lower oxygen activity, lower silicon content, with robust processing
- Coating systems demonstrated durability in various lab tests
- Potential systems for using as “scales and coatings” of UHTCC

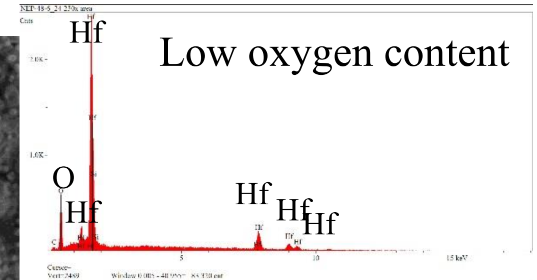




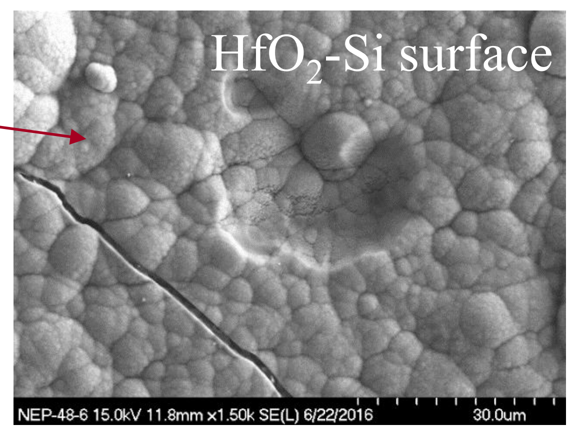
HfO_2-Si EBC Bond Coat Temperature Capability Tested

– HfO_2-Si bond coat tested at up to 1560°C +, 50 h
– Higher temperature region had

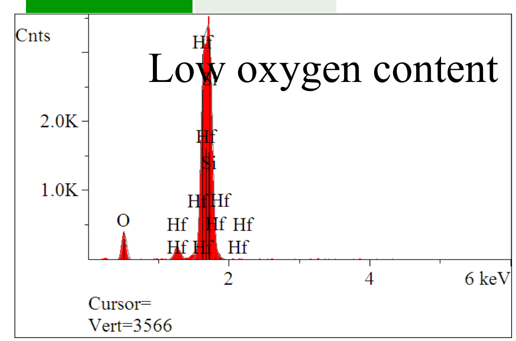




HfO_2-Si	At%
O	50-56
Si	4-10
Hf	30-40
Total	100.00

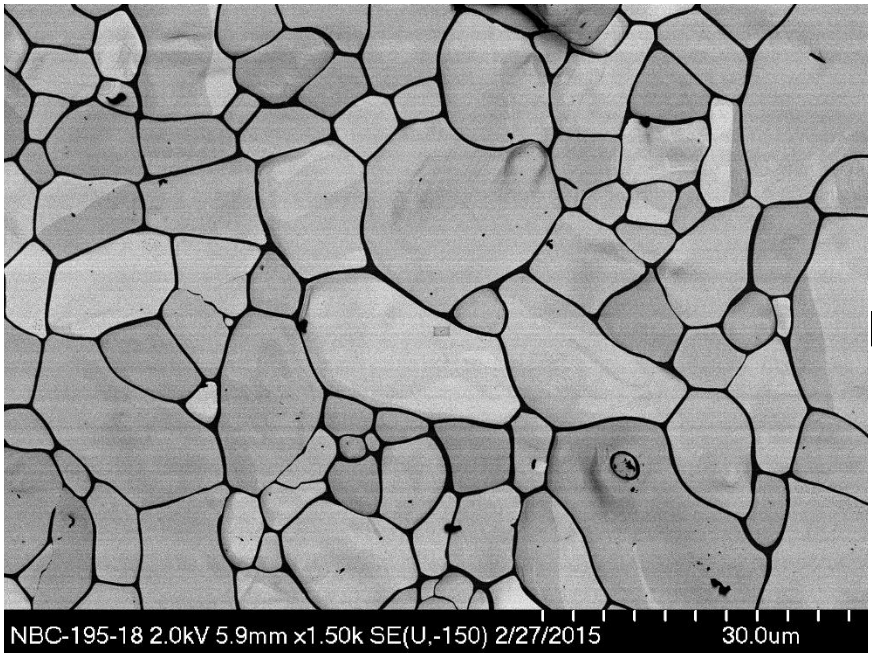


HfO_2-Si	At%
O	26.08
Si	49.10
Hf	24.82
Total	100.00

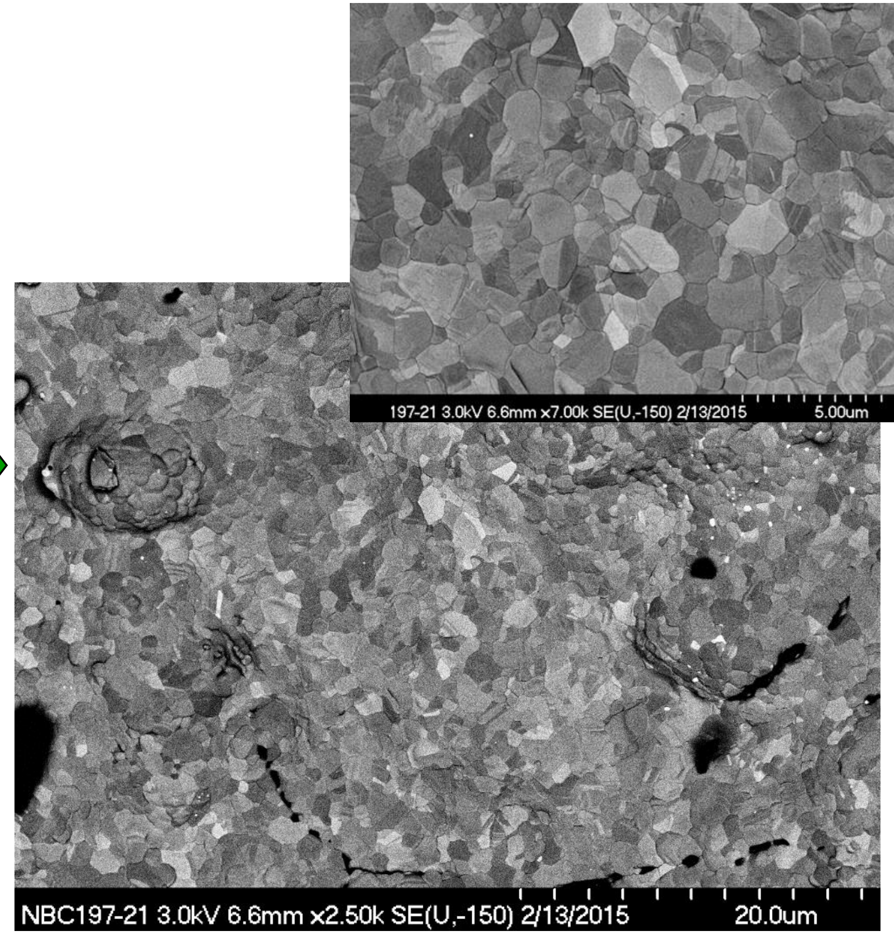


Advanced RE-Si Based EBC Bond Coats: Controlled Oxygen Activities, Dopant Additions

- Advanced compositions improve high temperature stability and environmental resistance
- Refined grain structures observed for hafnium-doped systems after 500 h furnace cyclic tests



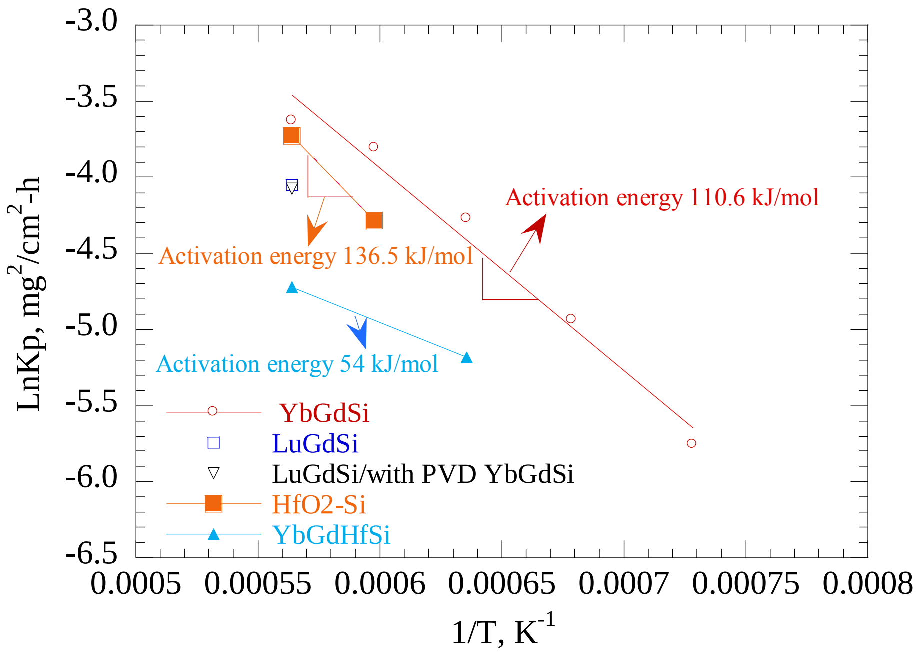
YbSi-YbSi(O) EBC bond coat, 1500°C cyclic tested

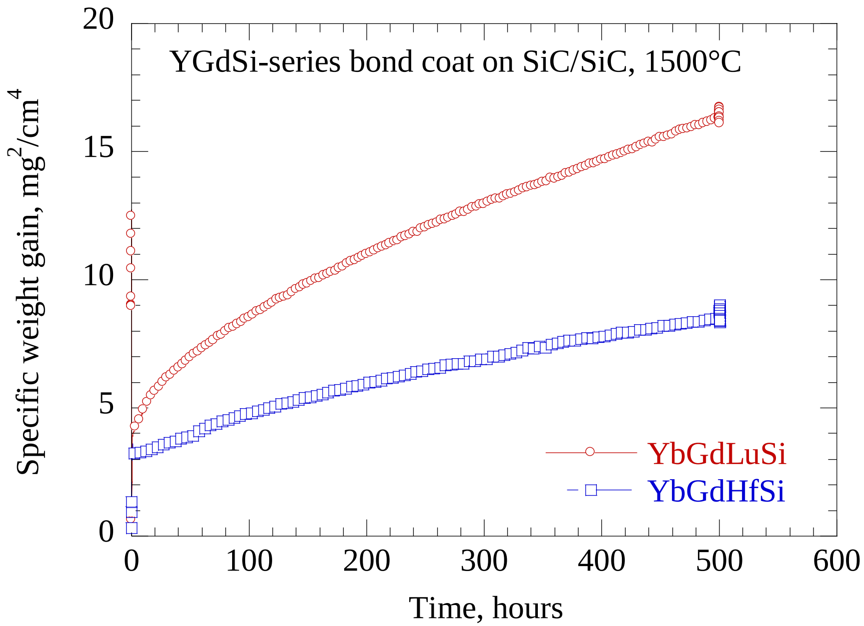


YbSi-YbSi(O)+*Hf* EBC bond coat, 1500°C cyclic tested

Advanced RE-Si Based EBC Bond Coats: Controlled Oxygen Activities, Dopant Additions

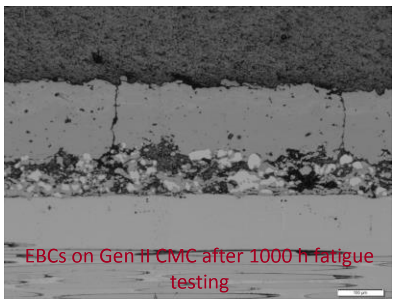
- Oxidation kinetics comparisons for various 2700°F coating systems
- The PVD processed REHfSi shown to have lower oxidation rates

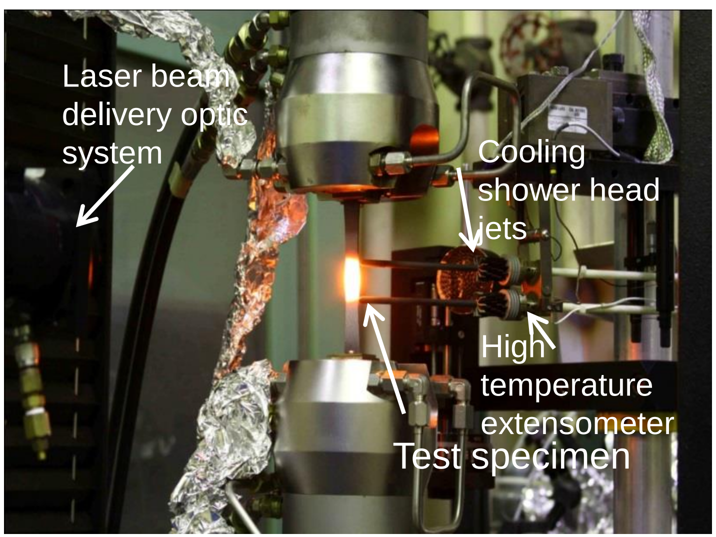


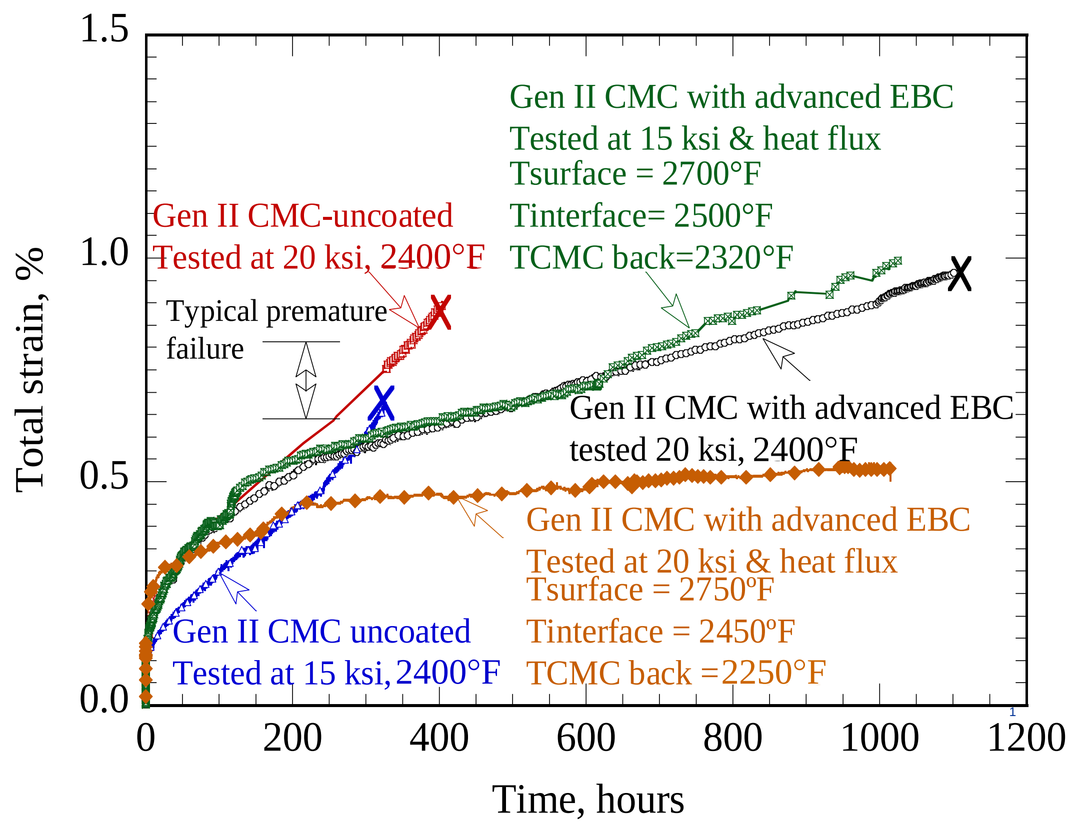


Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Some Benchmark Tests

– Advanced multi-component hafnia-rare earth silicate based turbine environmental barrier coatings being tested for up to 1150 h creep rupture
– Helped understand EBC-CMC creep, fatigue and environmental interactions, and modeling



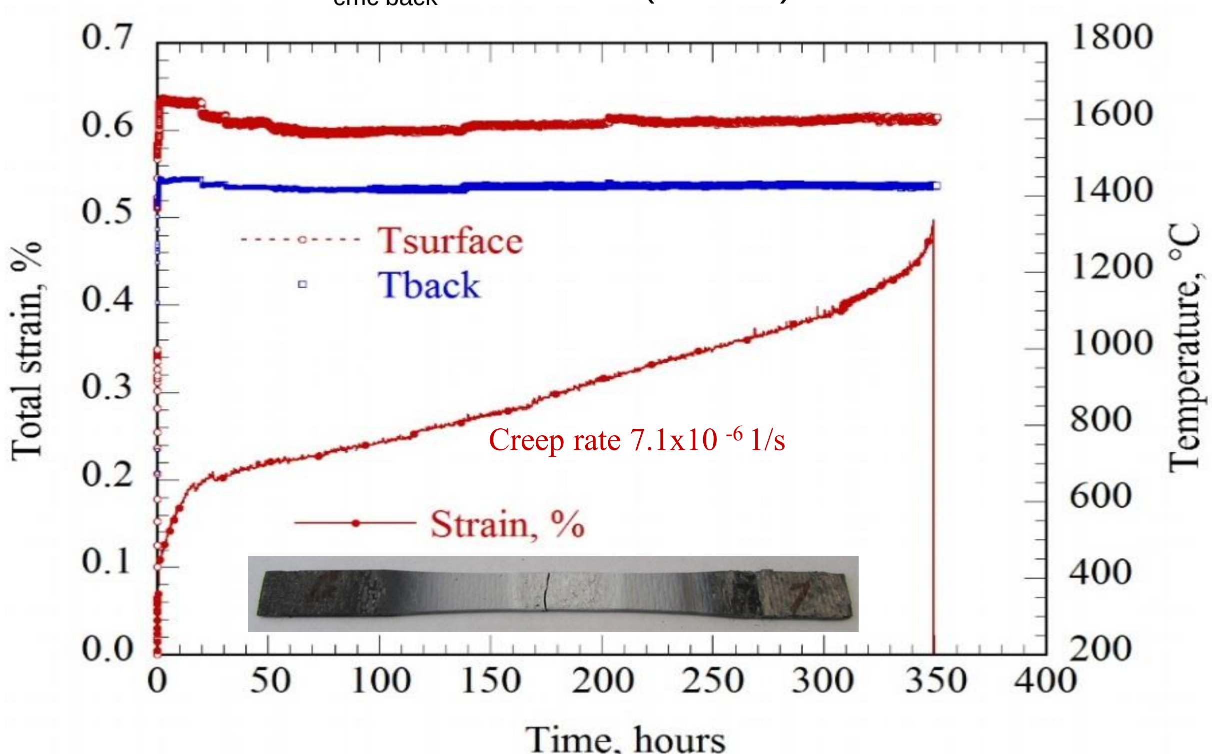




Laser Thermomechanical Creep - Fatigue Tests of Advanced 2700°F+ EBC Systems

- APS, PVD and EB-PVD processed 2700°F bond coats and EBCs on SiC/SiC CMC: focus on creep, fatigue high heat flux testing at temperatures of 1316-1482°C+ (2400-2700°F+) – selected Examples

EB-PVD Rare Earth Silicate EBC/YbGdYSi bond coat on Hyper Therm CVI-MI
$T_{EBC\ surface}$ 2850-3000°F (1600-1650°C)
$T_{cmc\ back}$ at ~2600°F (1426°C)

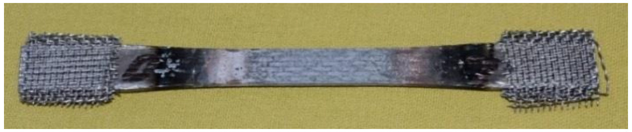


D. Zhu, “Advanced Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composite Turbine Components”, Engine Ceramics – Current Status and Future Prospects, pp 187-202, 2016

Laser Thermomechanical Creep - Fatigue Tests of Advanced 2700°F+ EBC Systems - Continued

- APS, PVD and EB-PVD processed 2700°F bond coats and EBCs on SiC/SiC CMC: focus on creep, fatigue high heat flux testing at temperatures of 1316-1482°C+ (2400-2700°F+) – Selected Examples

Fatigue Tested (furnace)



PVD GdYSi(O) coated on CVI-MI SiC/SiC
1316°C, 10ksi, 1000 h fatigue (3 Hz, R=0.05)

Fatigue Tested

EB-PVD ($RE_2Si_{2-x}O_{7-x}$ EBC/HfO_2-Si bond coat on 3D CVI+PIP SiC/SiC (1482°C, 10ksi, 300 h SPLCF fatigue at 3 Hz, R=0.5; furnace tested)

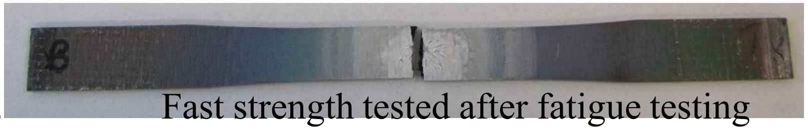
Creep and Fatigue Tests with CMAS

Air Plasma Sprayed APS YSi+Hf-RESilicate
EBC Bond Coat series on CVI-MI SiC/SiC
1400°C,at 10 ksi, 400 hr

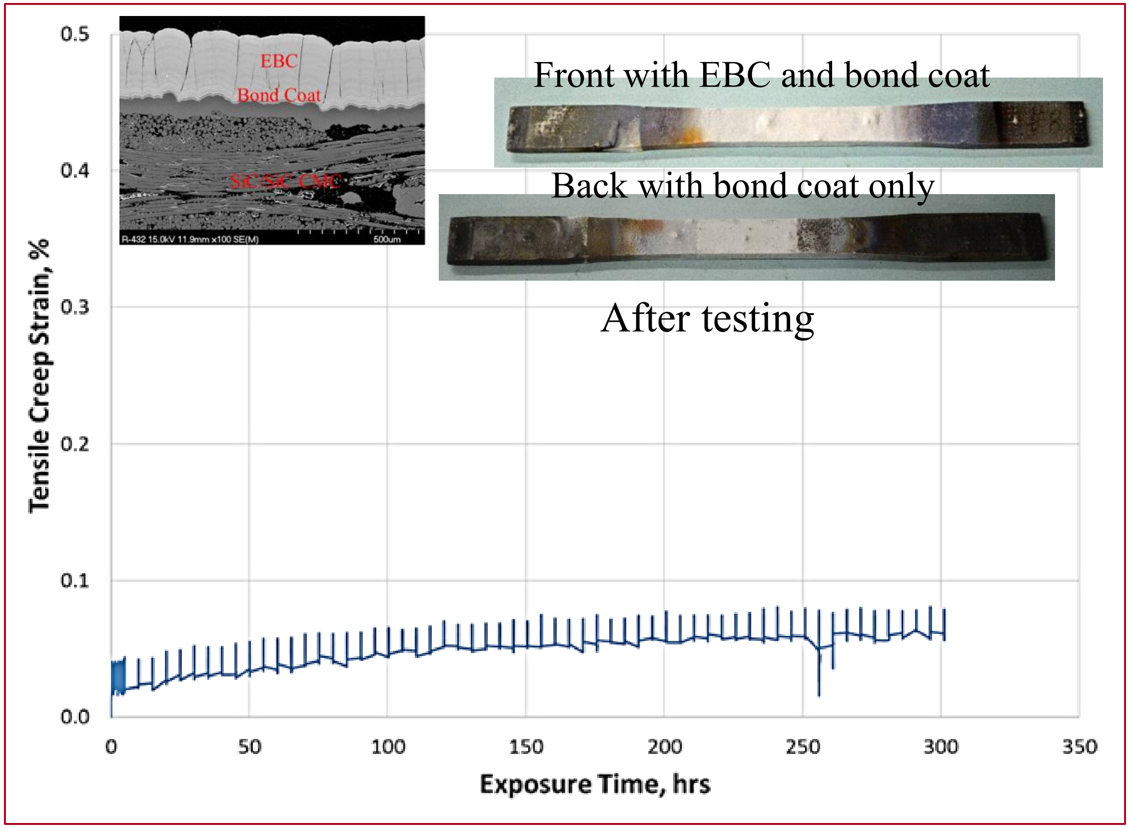
EB-PVD ($HfRE_2Si_{2-x}O_{7-x}$ EBC/GdYbSi(O) bond coat on CVI-MI SiC/SiC (with CMAS)
1537°C, 10ksi, 300 h fatigue (3 Hz, R=0.05)

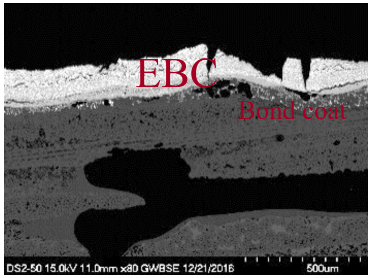
Laser Thermomechanical Creep - Fatigue Tests of Advanced 2700°F+ EBC Systems - Continued

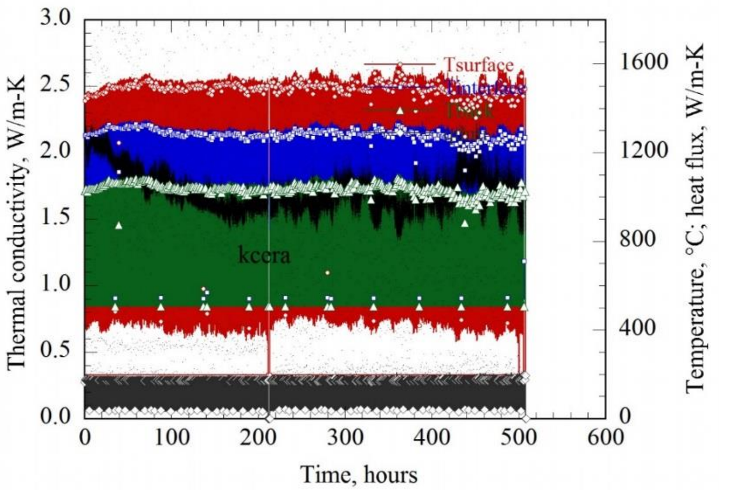
- Benchmark fatigue testing at 2700°F of coating system
- Also demonstrating laser steam rig 500 hr at laser rig tests at 2700°F+ EBC temperatures
- Development towards 3000°F+ thin coatings



3hz fatigue testing at 10 ksi loading
Completed 500 hr testing in heat flux rig with steam

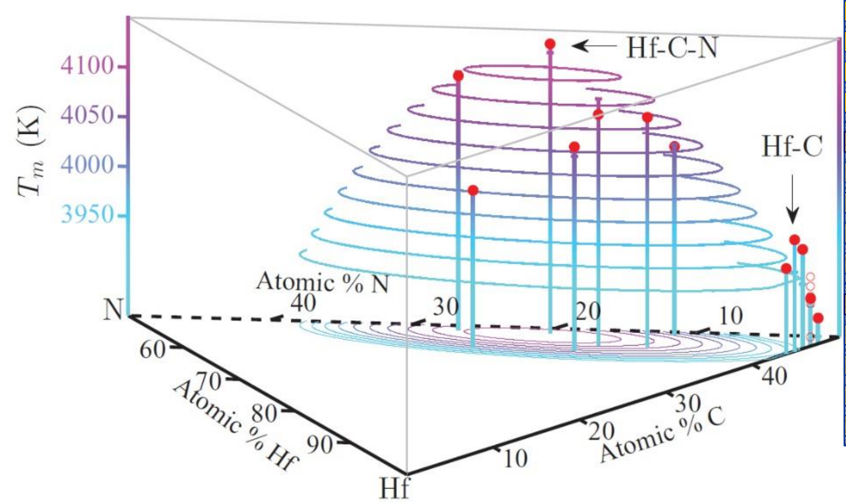


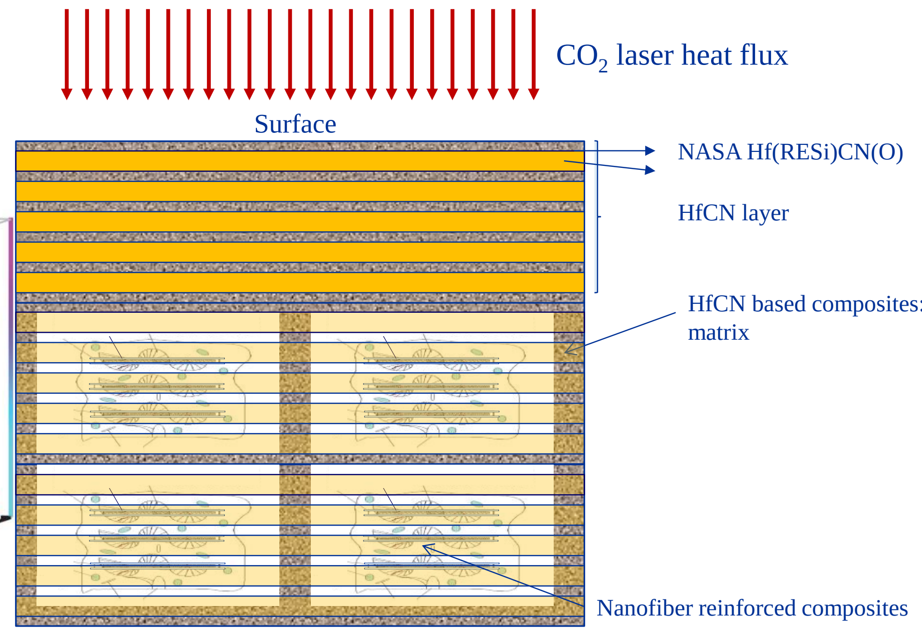




Ultra High Temperature and Multifunctional Ceramic Matrix Composite – Coating Systems for Light-Weight Space and Aero Systems

– Develop Ultra High Temperature Ceramics and Coatings (UHTCC) based on HfCN and HfTaCN
– Focused on Hf-RE-Si-O-(CN) protective scales or coatings to improve oxidation resistance
– Evaluate and improve mechanical properties
– Incorporated atomistic modeling, and thermodynamic measurements and modeling
– Initiated the system testing and coating developments

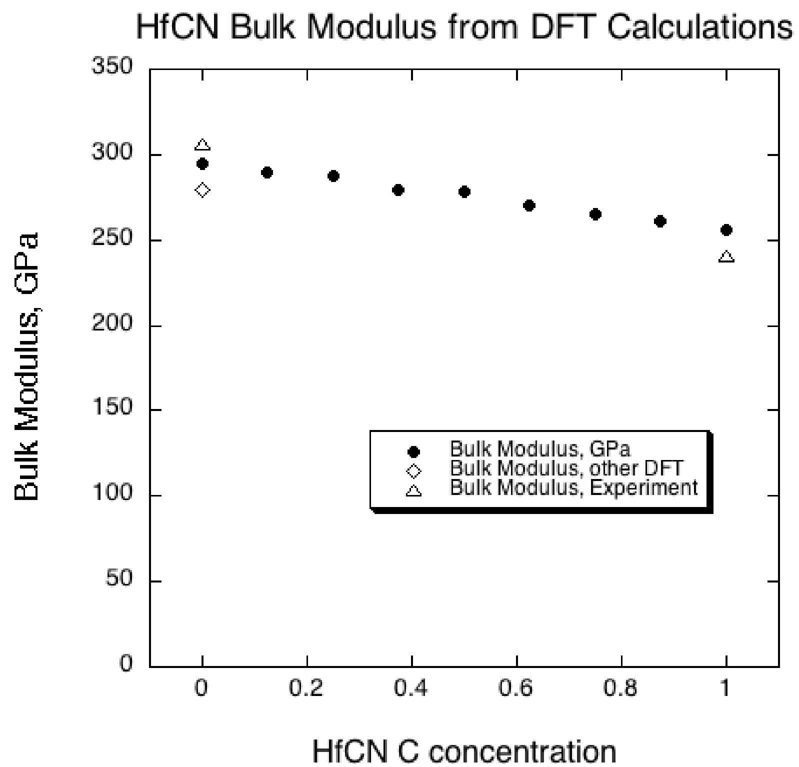




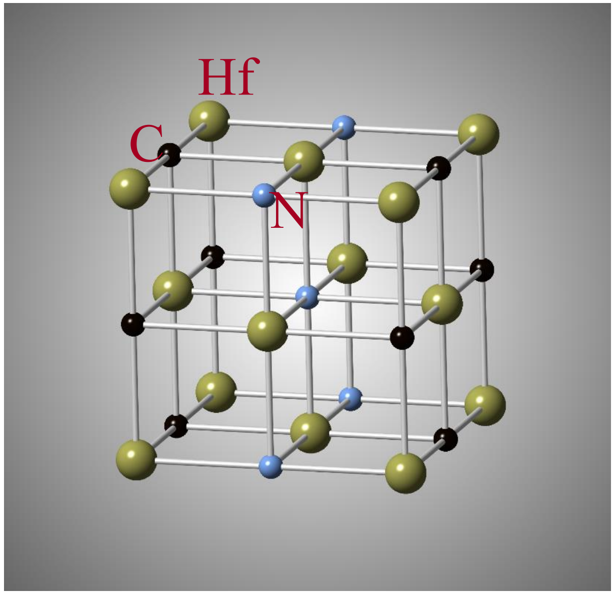
– Qi-June Hong and Axel van de Walle, "Prediction of the material with highest known melting point from ab initio molecular dynamics calculations", Physical Review B92, 020104-1 - 020104-6 (R) (2015).

Ultra High Temperature and Multifunctional Ceramic Matrix Composite – Coating Systems for Light-Weight Space and Aero Systems - continued

- Downselected initial $HfC_{0.54}N_{0.40}$ and $HfC_{0.5}N_{0.5}$ for evaluations
- Demonstration of calculation of HfCN bulk modulus using Density Function Theory (DFT), understanding the atomic bonding with dopants

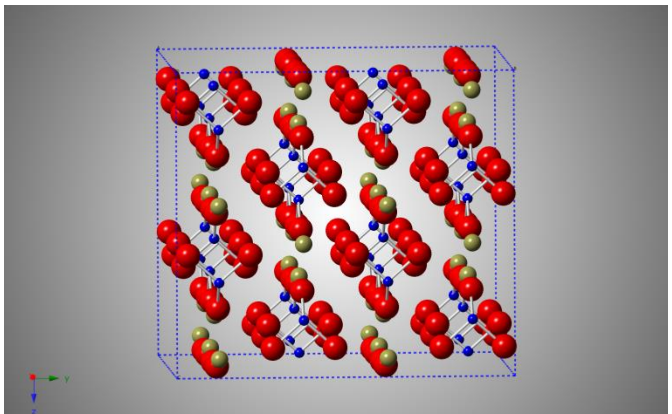


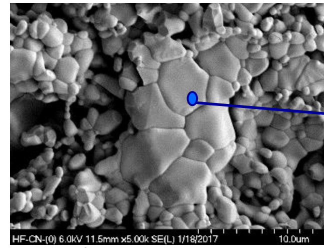
Bulk Moduli For HfC, HfN and various compositions of HfCN were computed using the Vienna Ab initio Simulation Package (VASP).

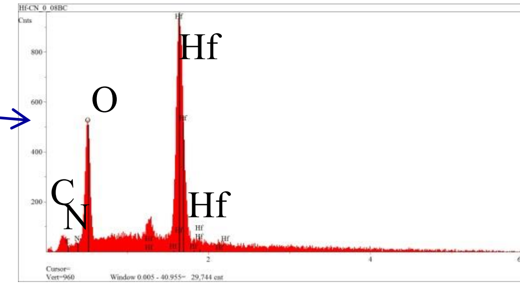


Develop HfO_2 and Hf-RE Based Coatings or Protective Scales

- Based on HfO_2-$HfSiO_x$ and HfYb-SiO_x systems for improved temperature capabilities and durability
- Evaluating HfOCN stability

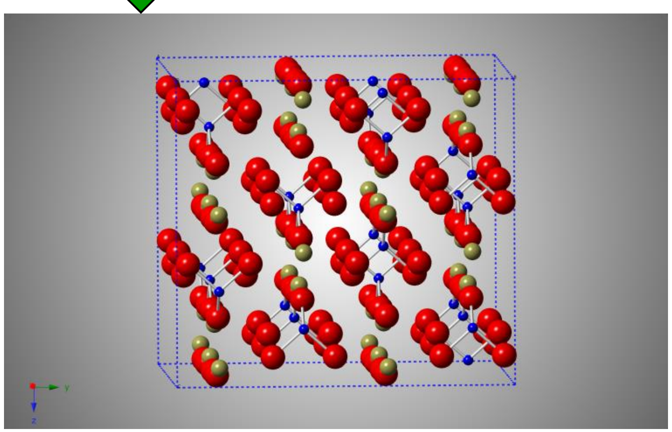


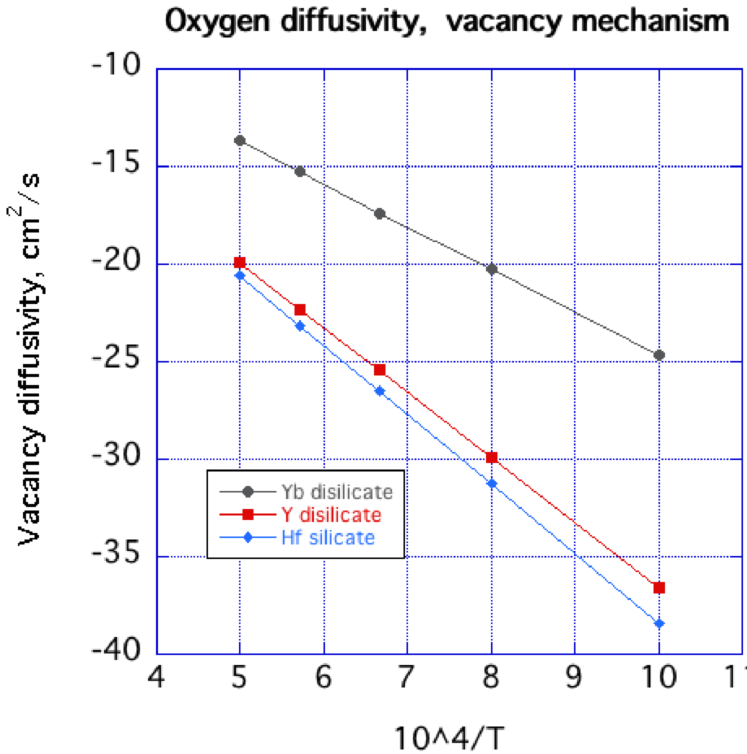




1300°C oxidized, 75hr

Reduced SiO_2





DFT Modeling results (by Brian Good)

Summary

- Durable EBCs are critical to emerging SiC/SiC CMC component technologies
- Multicomponent EBC oxide/silicates being developed with higher stability and improved durability
- HfO_2-Si and RE-Si bond coats being developed for realizing 1482°C+ (2700°F+) temperature capabilities
  - Further temperature capability improvement can be improved using RE-Si+Hf bond coats
  - Multicomponent RE-Hf-silicate top coat also developed to improve combustion steam and CMAS resistance
  - Hf-Y/Yb-RE silicates system also being explored for higher temperature capabilities
- EBC-CMC system rig durability testing and demonstrations
- Ultra high temperature materials also benefit from the coating and material system technologies

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