



The Development of 2700-3000°F Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites: Challenges and Opportunities

Dongming Zhu

Structures and Materials Division
NASA John H. Glenn Research Center
Cleveland, Ohio 44135



**The 11th International Conference on Ceramic Materials and Components for Energy and Environmental Applications
Vancouver, British Columbia, Canada
June 15-19, 2015**

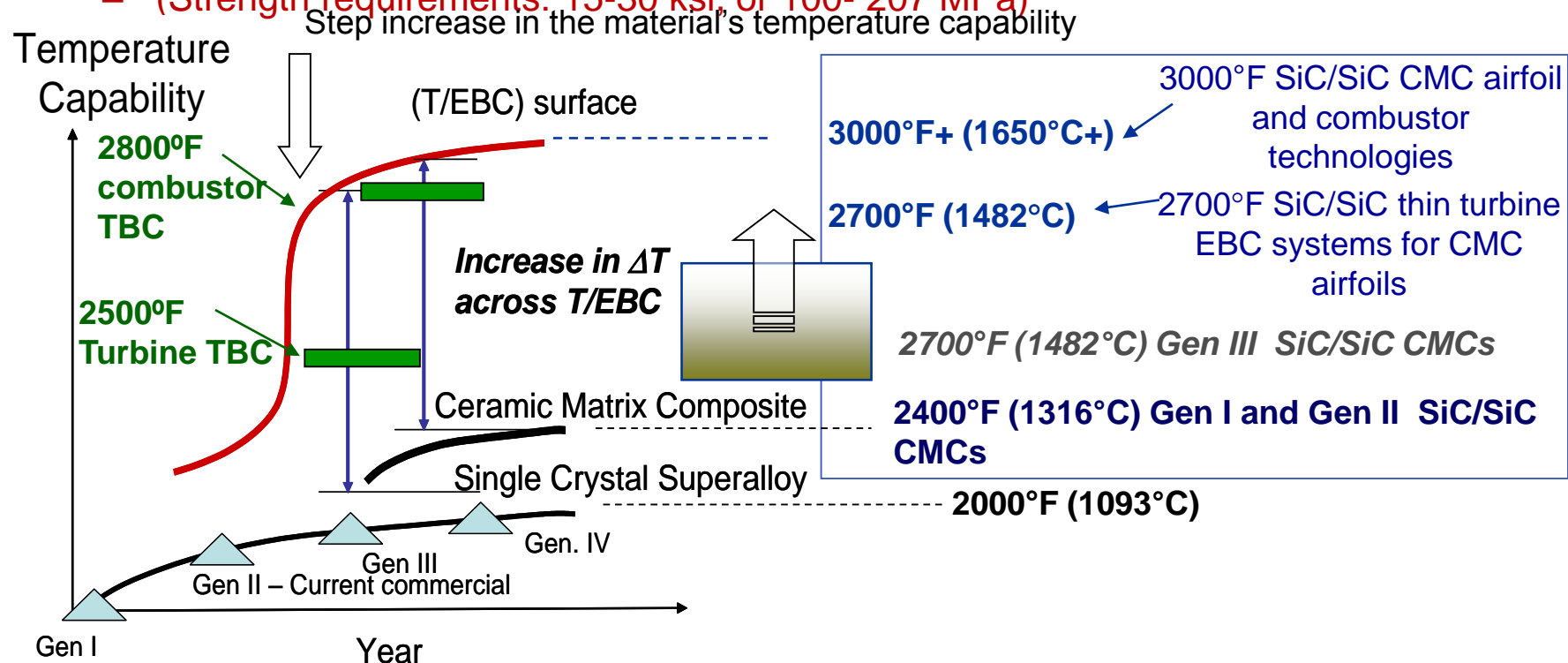


Outline

- **Environmental barrier coating system development: needs and challenges**
- **Advanced environmental barrier coating systems for CMC airfoils and combustors**
 - NASA coating development goals
 - Current turbine and combustor EBC coating development emphases
- **Development of next generation environmental barrier coatings**
 - Advanced processing
 - Subelement and subcomponent demonstrations
- **Summary and Emerging Opportunities**

NASA EBC and CMC System Development

- Emphasize temperature capability, performance and *long-term* 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) **thin** turbine and CMC combustor coatings
 - **Recession: <5 mg/cm² per 1000 h**
- Highly loaded EBC-CMCs capable of thermal and mechanical (static/low cycle and dynamic) loading
 - **(Strength requirements: 15-30 ksi, or 100- 207 MPa)**

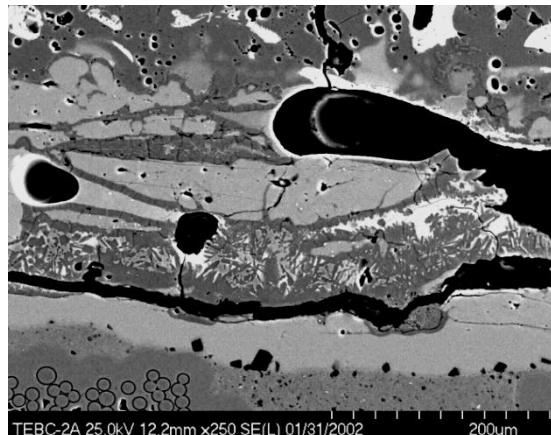
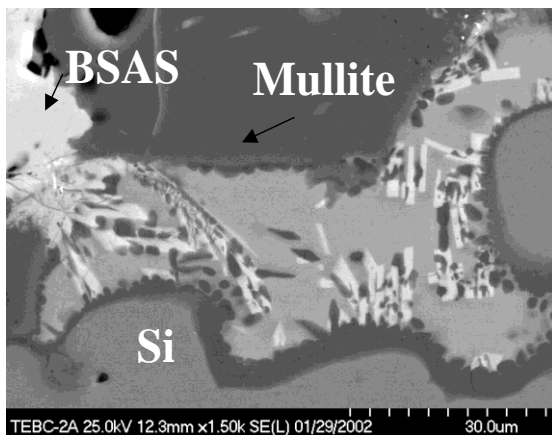




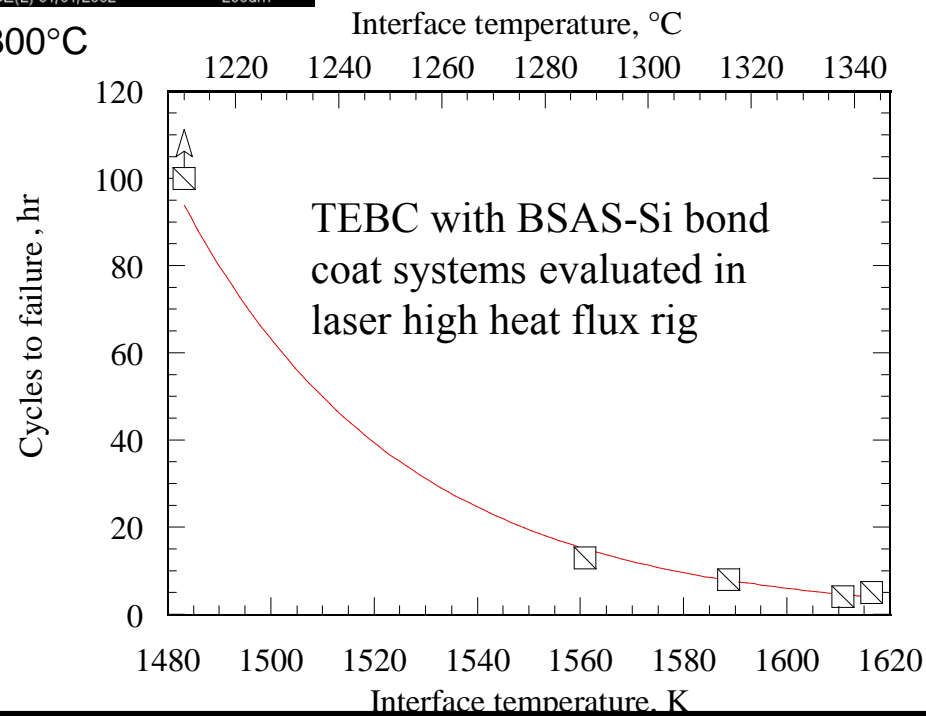
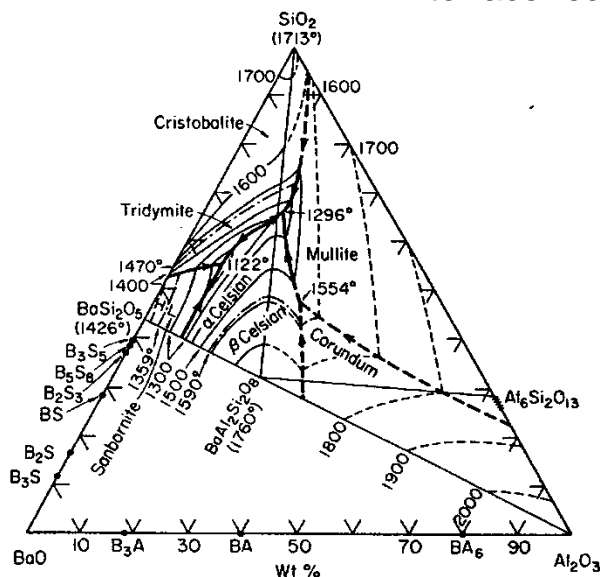
Environmental Barrier Coating Development: Challenges and Limitations

- Current EBCs limited in their temperature capability, water vapor stability and long-term durability, especially for advanced high pressure, high bypass turbine engines
- 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 and interface stability
 - Critical to reduce the EBC Si/SiO₂ reactivity and their concentration tolerance
- EBC-CMC systems need advanced processing for realizing complex coating compositions, architectures and thin turbine configurations for next generation high performance engines
 - Advanced high temperature processing of high stability nano-composites using advanced Plasma Spray, Plasma Spray - Physical Vapor Deposition, EB-PVD and Directed Vapor EB-PVD, and Polymer Derived Coating processing

Environmental Barrier Coating Development: Challenges and Limitations



Interface reactions at 1300°C

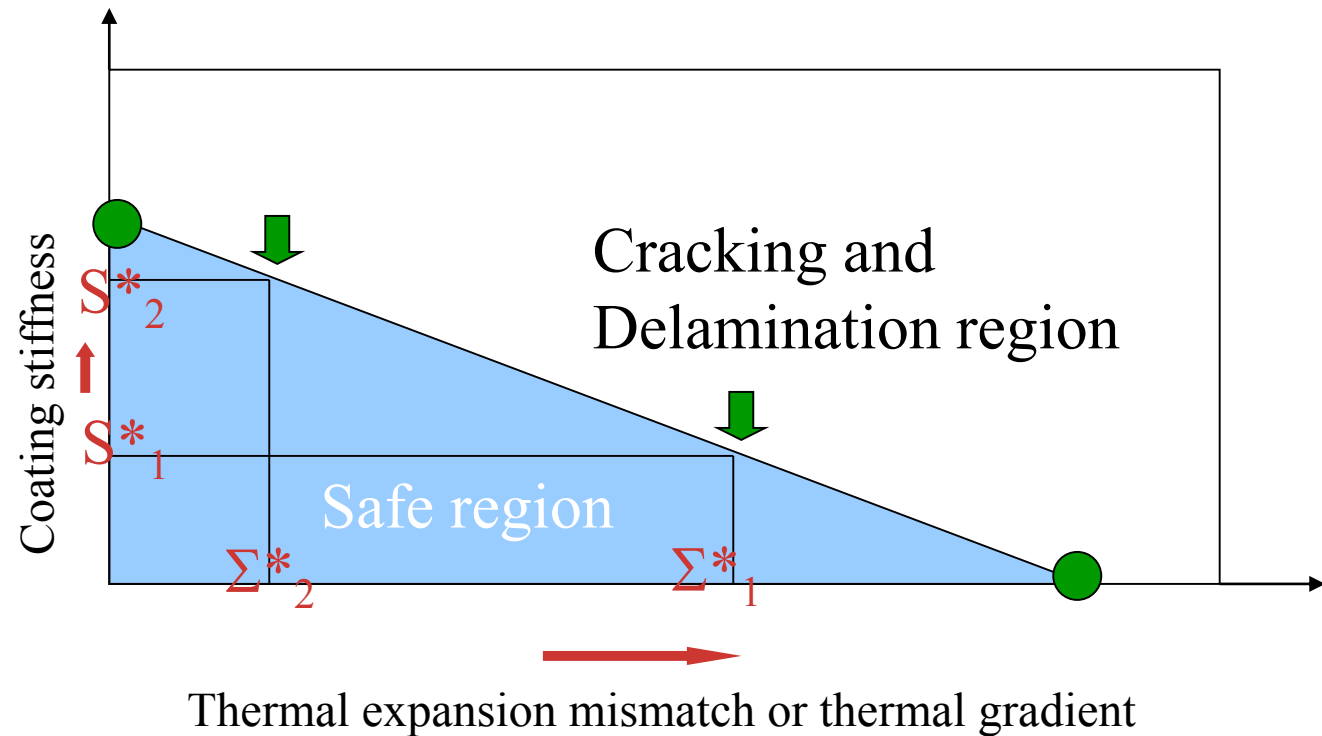




NASA Environmental Barrier Coating Technology Development - Continued

- Fundamental studies of environmental barrier coating materials and coating systems, stability, temperature limits and failure mechanisms
- Focus on high performance and high technology readiness level (TRL), high stability HfO_2 and ZrO_2 - RE_2O_3 - $\text{SiO}_2/\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$ environmental barrier systems
 - Controlled silica content and transition element and rare earth dopants to improve EBC stability and toughness
 - Develop HfO_2 -Si based + X (dopants) and more advanced bond coat systems for 2700°F+ long term applications
 - Develop prime-reliant Rare Earth-Si alloys and composites for integrated EBC-bond coat systems
- Processing optimization for improved coating density and composition control robustness
- Develop advanced NASA high toughness, Alternating Composition Layered Coating (ACLC) compositions and processing for low RE t' low rare earth dopant low k HfO_2 and higher rare earth dopant silicates
 - Achieving high toughness has been one of key emphases for NASA coating technologies
 - Achieving high stability and recession resistance
 - Improve the resistance to CMAS and Volcano ash deposits

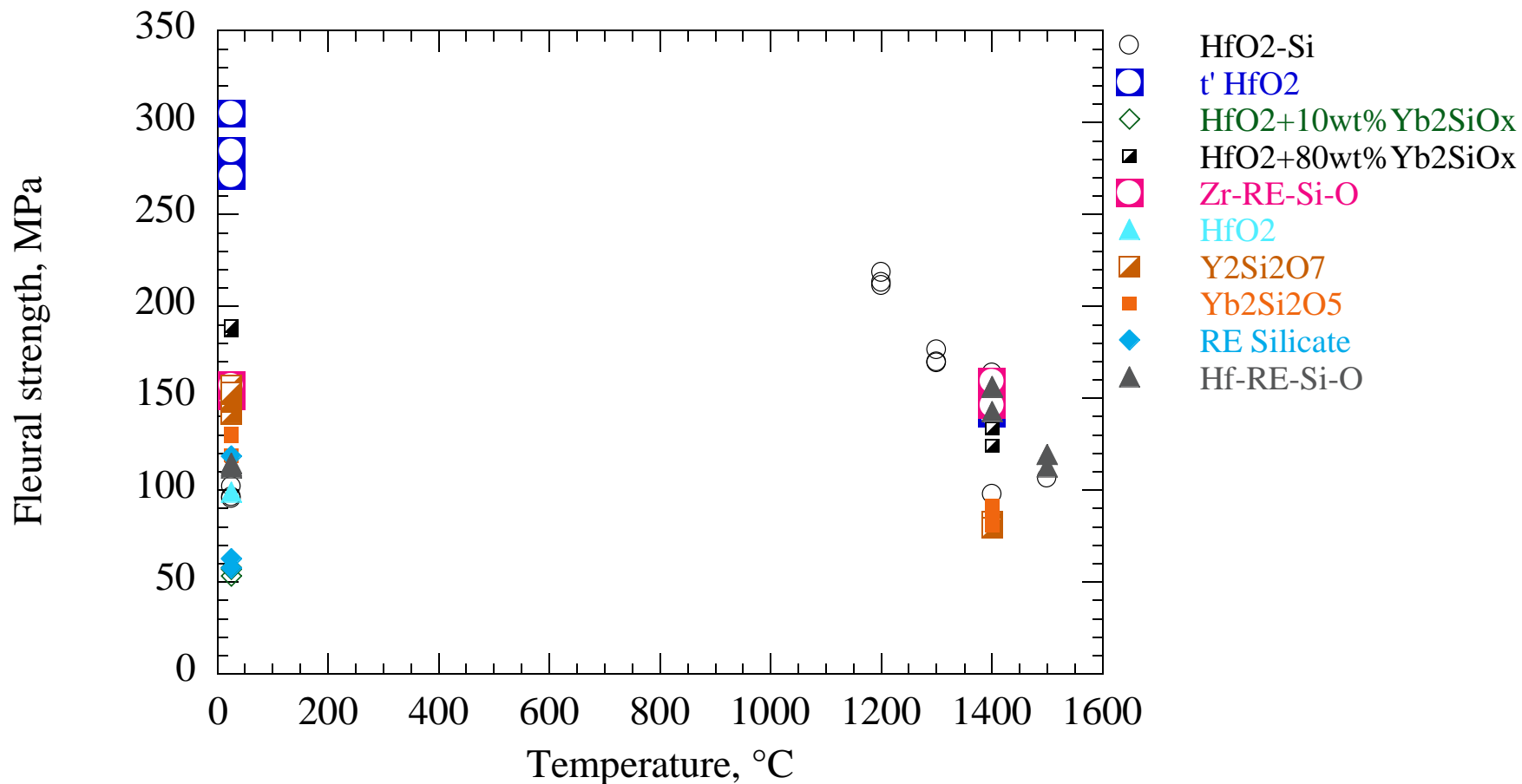
Coating Safe Design Approach



CMC/Bond coat \longrightarrow EBC \longrightarrow TBC (optional)

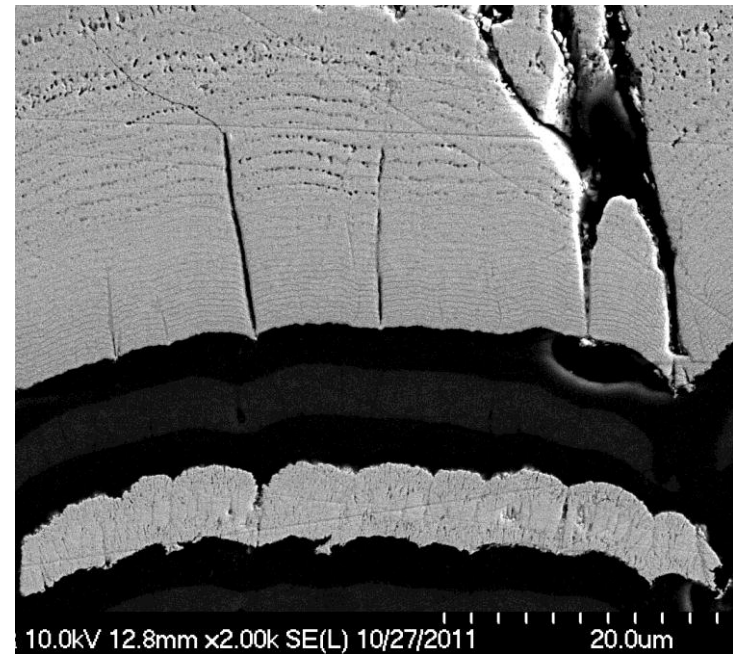
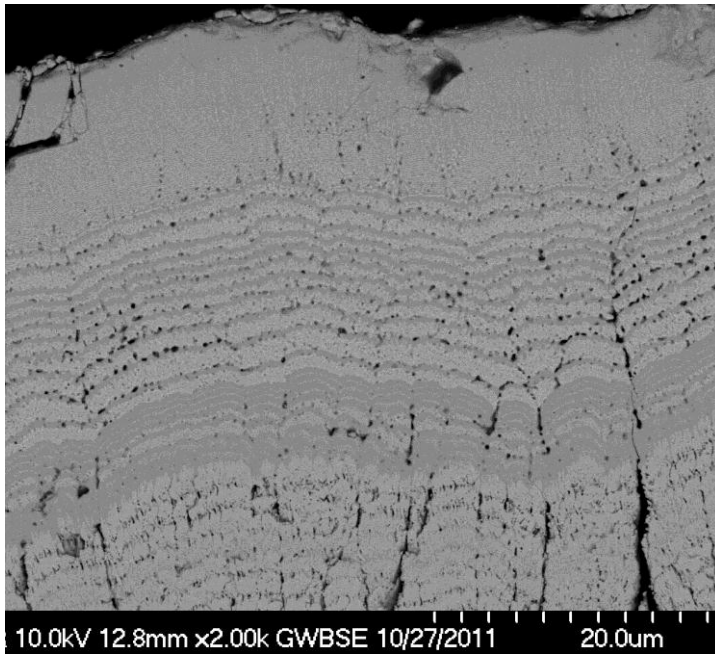
Advanced EBC System Strength Evaluations

- Evaluate and develop high strength and high toughness EBC materials
- Provide property database for design and modeling



Advanced EBC System Recession and Stability Evaluations

- Determining optimum compositions of in a high stability system consisting of (e.g., Yb, Gd, Y+Hf/Zr) silicates and oxide systems

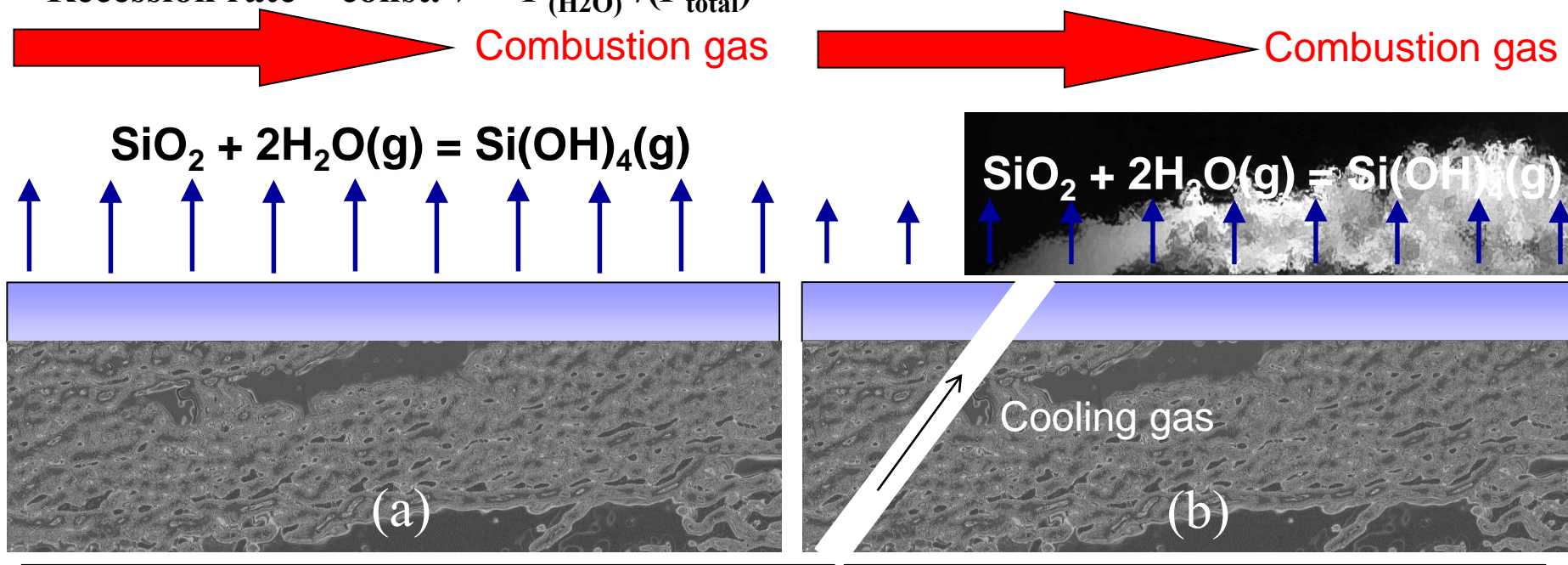


Turbine EBCs: High pressure burner rig, at 10 atm, 2650°F

SiC/SiC and Environmental Barrier Coating Recession in Turbine Environments

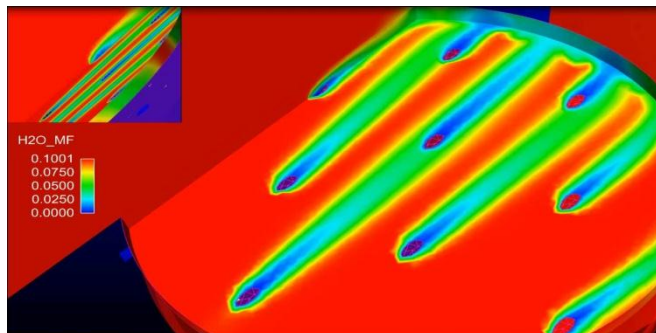
- **Recession of Si-based Ceramics**
 - (a) convective; (b) convective with film-cooling
- **Advanced rig testing and modeling** (coupled with 3-D CFD analysis) to understand the recession behavior in High Pressure Burner Rig
 - Work primarily supported under the ERA Combustor and FAP Supersonics projects

$$\text{Recession rate} = \text{const. } V^{1/2} P_{(\text{H}_2\text{O})}^2 / (P_{\text{total}})^{1/2}$$

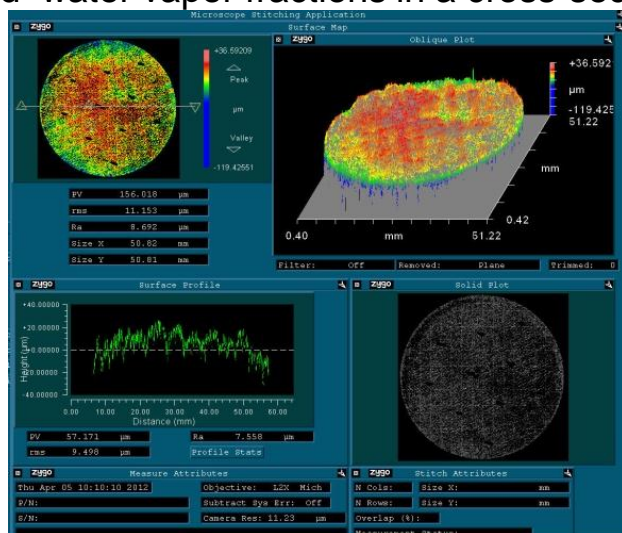


Recession of Film-Cooled SiC/SiC Specimens

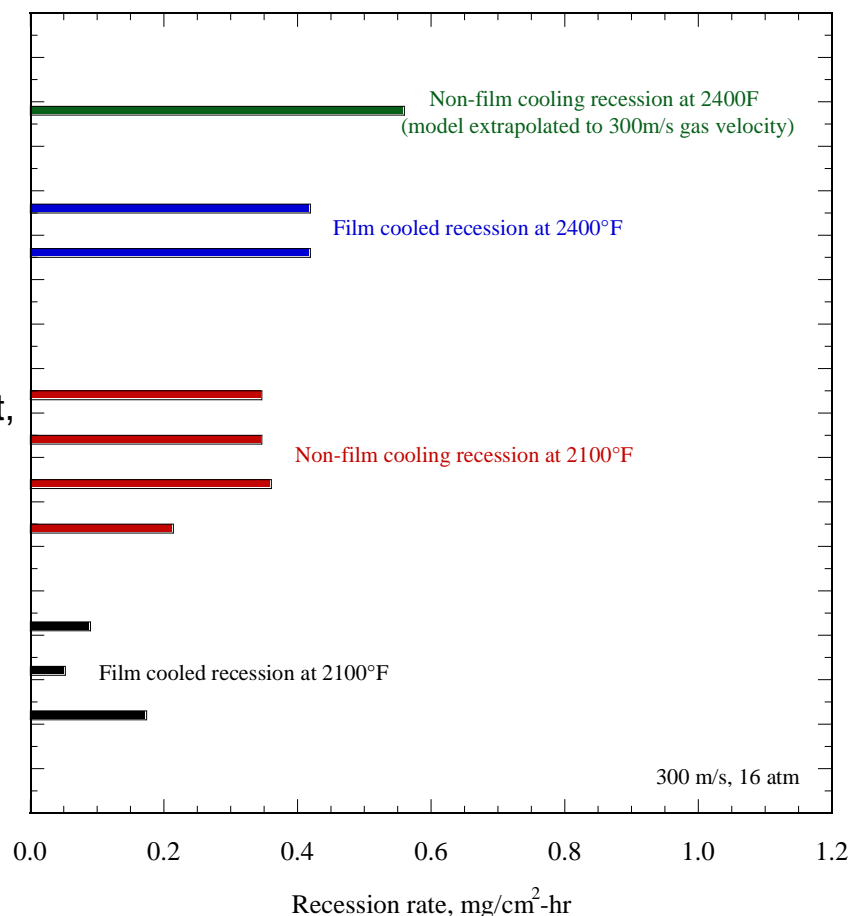
- Potentially improve EBC-CMC stability in combustion environments



The CFD modeling of a film-cooled CMC 10-hole subelement, and water vapor fractions in a cross-section view



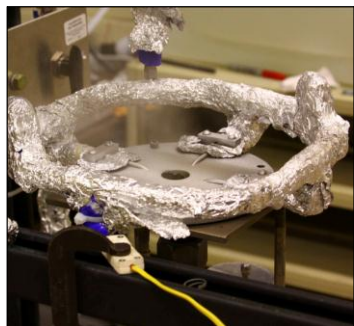
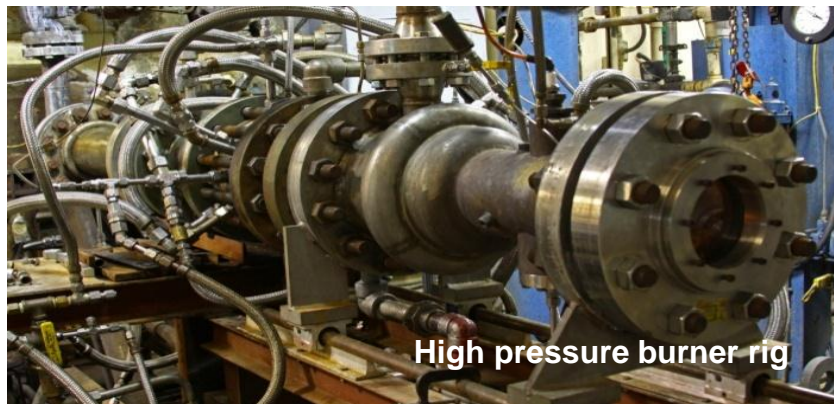
Zygo surface contour



High temperature recession kinetics for film-cooled and non-film cooled SiC/SiC specimens tested at NASA High Pressure Burner rig

Recession of EBCs in Laboratory Rig Tests

- EBC recession kinetics testing for CMCs-EBCs in NASA High Pressure Bruner Rig and Laser Steam High Heat Flux Rig Testing

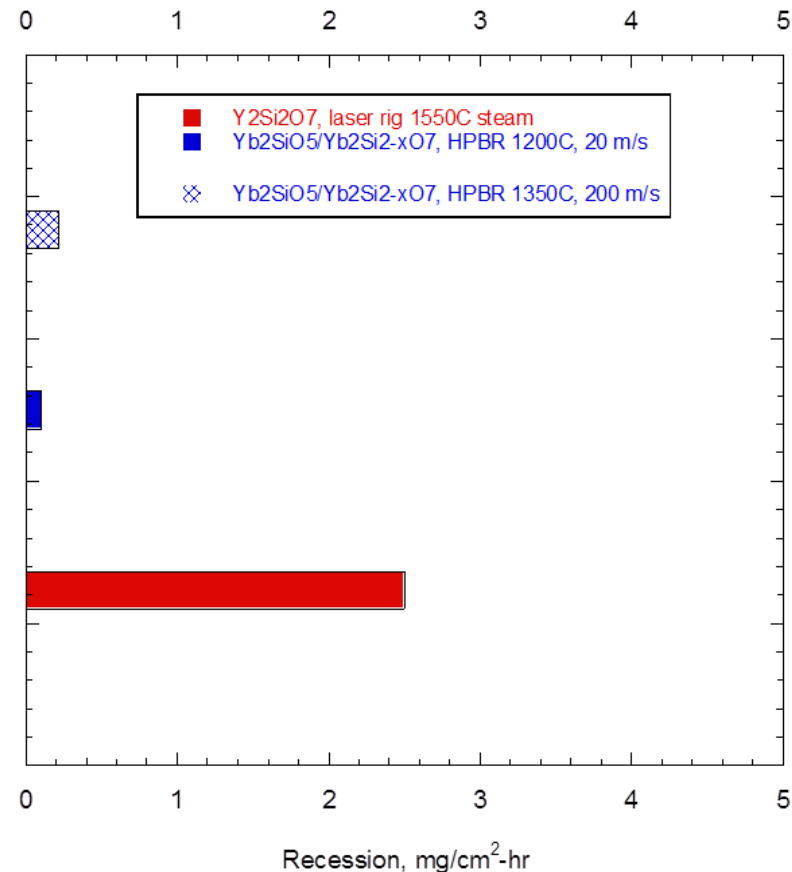


Steam during cooling cycles



High temperature testing with steam flow

(c) High heat flux and high steam rig

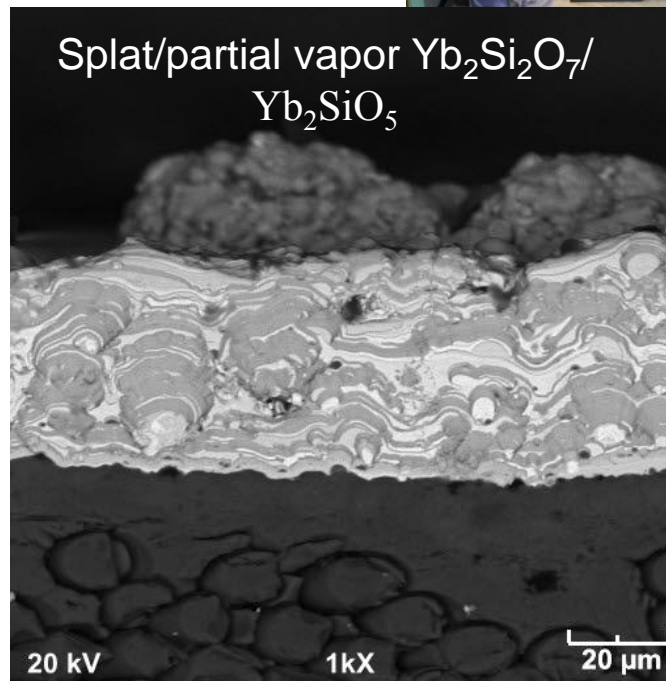
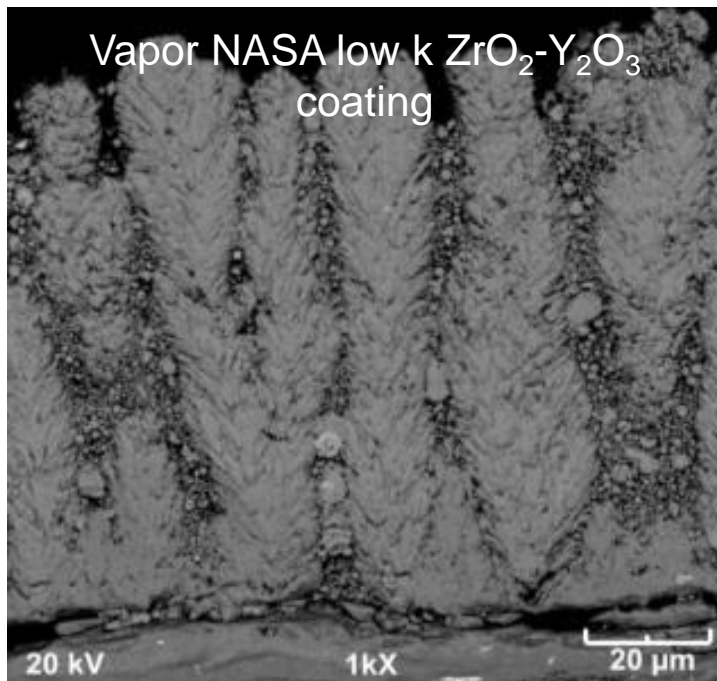
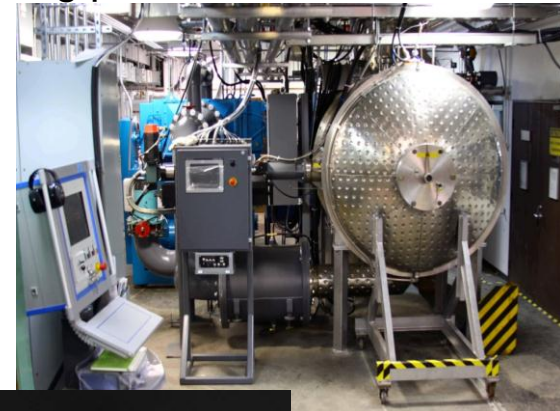


Examples of environmental barrier coating recession in laboratory simulated turbine engine conditions

Plasma Spray - Physical Vapor Deposition (PS-PVD) for SiC-SiC CMC Airfoil Coating Processing

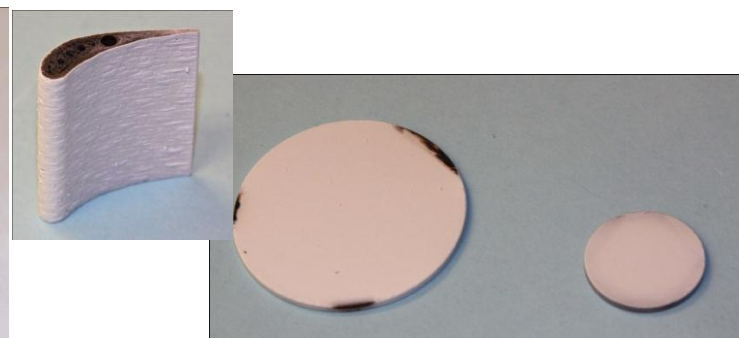
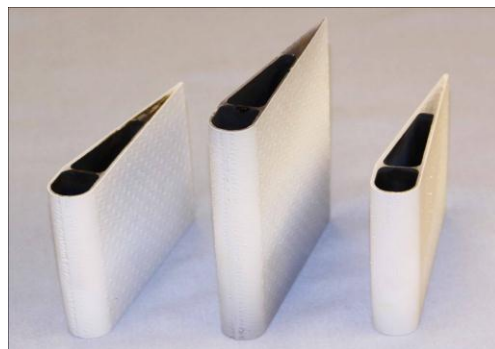
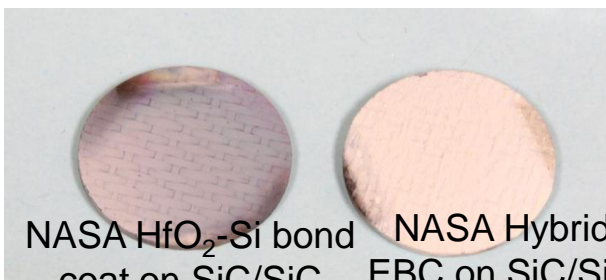
- Emerging processing methods developed by Sulzer Metco showing promise for next-generation SiC/SiC CMC turbine airfoil coating processing

NASA Hybrid PS-PVD coater system

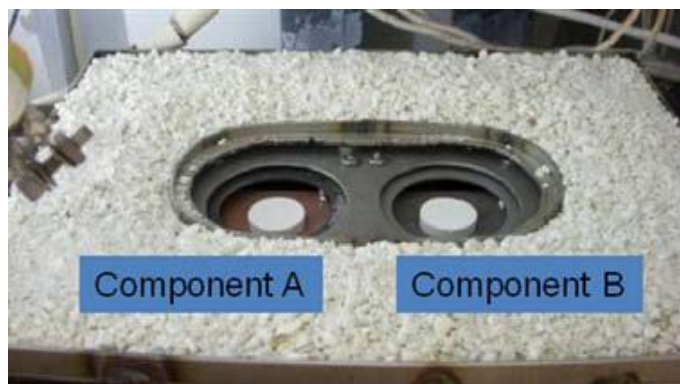
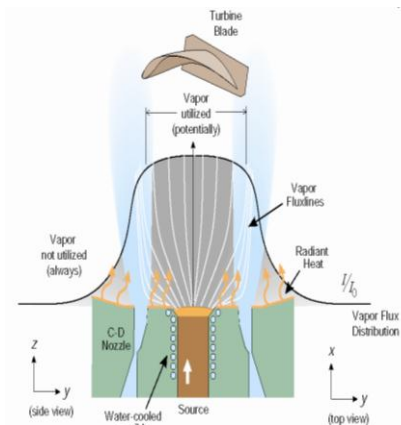


Development of *Directed Vapor* Electron Beam - Physical Vapor Deposition (EB-PVD) Airfoil Environmental Barrier Coating Processing under NASA Programs

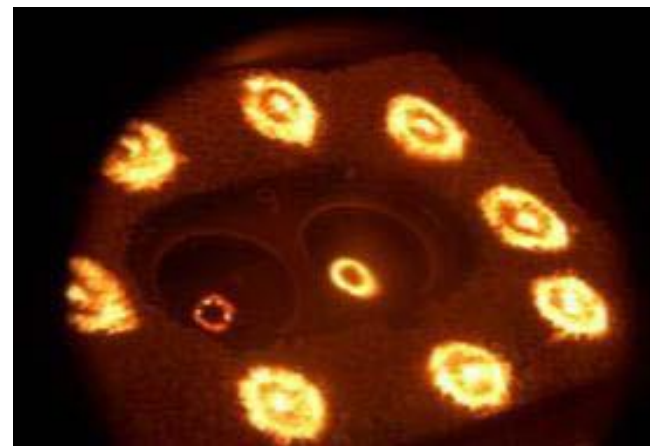
- Advanced coatings processed for higher TRL ERA combustor and turbine component EBCs (TRL 4-5) In collaboration with Directed Vapor Technologies



Advanced multi-component and multilayer turbine EBC systems



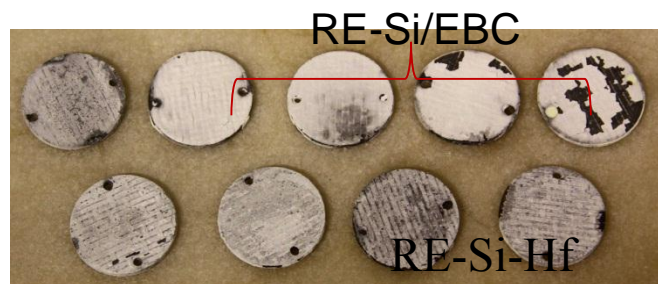
Directed Vapor Processing Systems



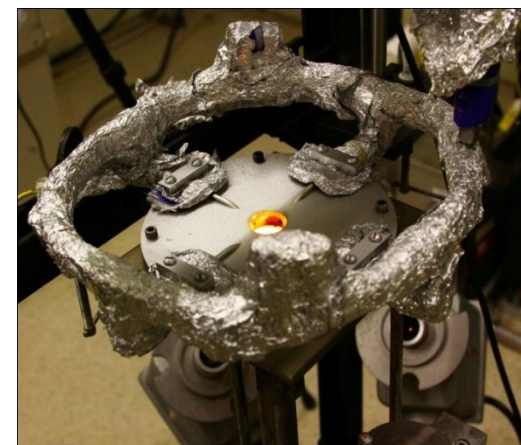
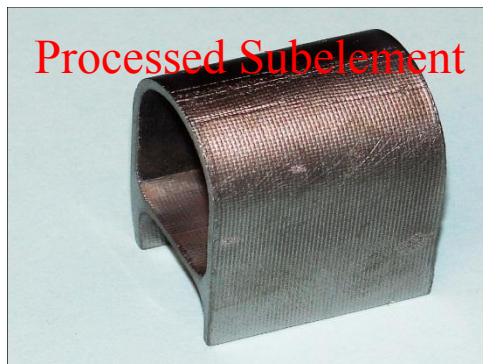
Advanced EBC Bond Coats for Turbine Airfoil and Combustor

EBCs Developed

- 1500°C (2700°F) capable NASA RESi+X(Ta, Al, Hf, Zr ...) EBC bond coat compositions and related composite coatings developed for combustor and turbine airfoil applications
- The bond coat systems demonstrated durability in the laser high heat flux rig in air and steam thermal gradient cyclic testing
- The bond coatings also tested in thermal gradient mechanical fatigue and creep rupture conditions



Selected
Composition Design
of Experiment
Furnace Cyclic Test
Series 1500°C, in air,
Demonstrated 500hr
durability



Steam heat flux test rig of
the bond coat



100% steam
High heat flux cyclic rig tested Zr/Hf-RE-Si series
EBC bond coats on the bond coated woven
SiC/SiC CMCs at up to 1500°C in air and full
steam environments

Effect of CMAS Reactions on Grain Boundary Phases

- CMAS Related EBC Degradations
- Grain boundary low melting phases
 - Eutectic region with a high Al_2O_3 content $\sim 1200^\circ\text{C}$ melting point
 - Loss of SiO_2 due to volatility

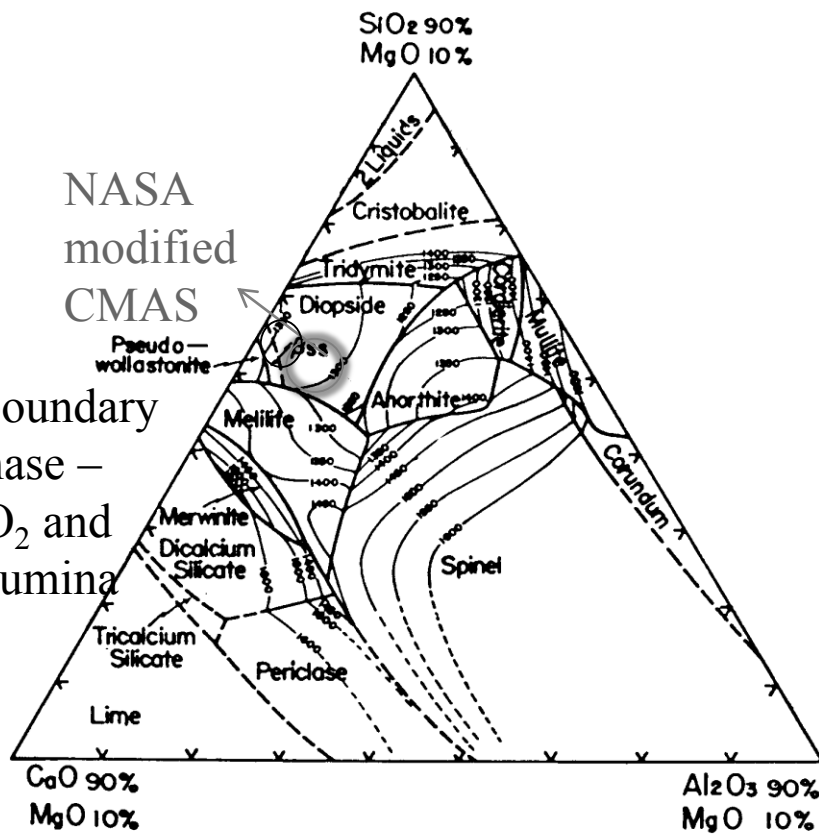
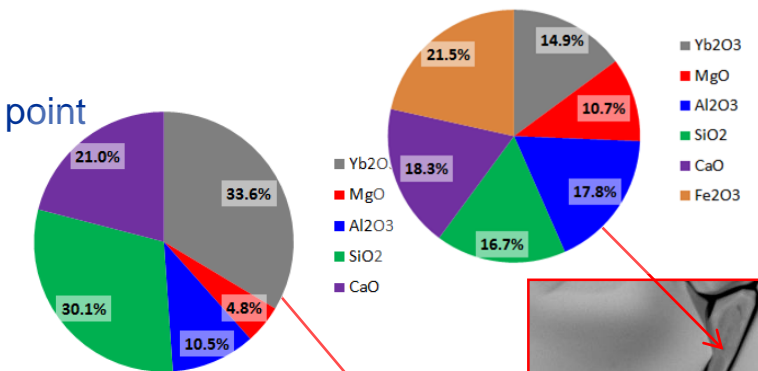
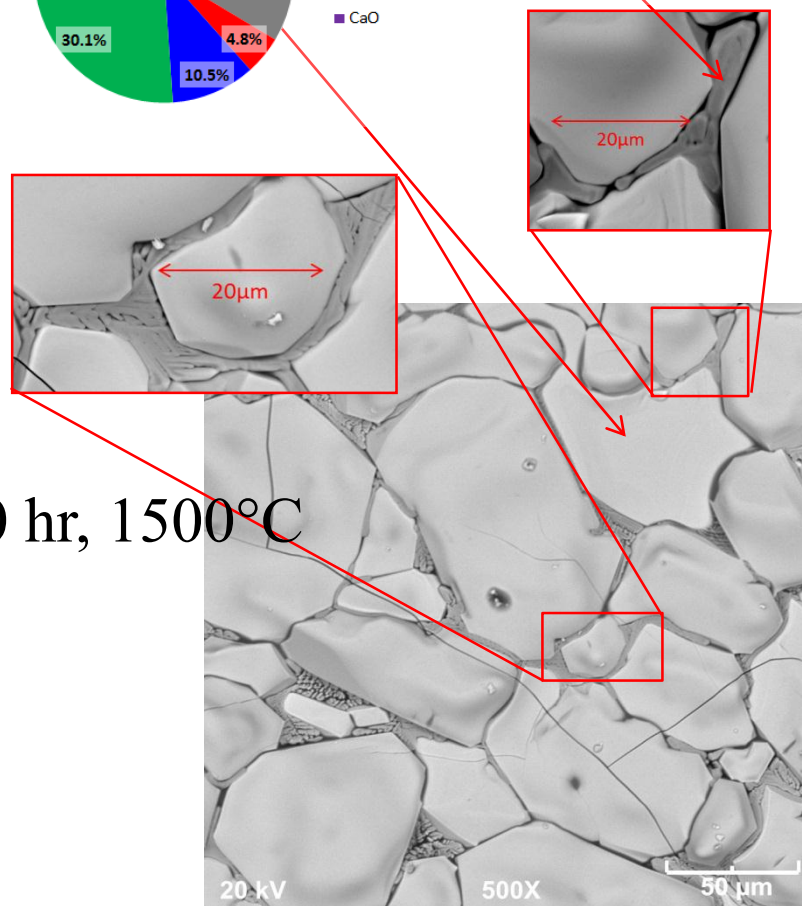


Fig. 4. The 10% MgO plane of the system $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ showing the isotherms and fields of primary crystallization. A.T.Prince, J.Amer.Ceram.Soc., 37(9)1954 p402-408



200 hr, 1500°C





Advanced 2700°F Based Bond Coats – System Processing for Various Process and Component Applications

- Advanced systems developed and to improve Technology Readiness Levels (TRL)
- Composition ranges studied mostly from 50 – 80 atomic% silicon
 - PVD-CVD processing, for composition downselects - also helping potentially develop a low cost CVD or laser CVD approach
 - Compositions initially downselected for selected EB-PVD and APS coating composition processing
 - Viable EB-PVD and APS systems downselected and tested; development new PVD-CVD approaches

PVD-CVD

YSi	YbGdYSi	GdYSi
ZrSi+Y	YbGdYSi	GdYSi
ZrSi+Y	YbGdYSi	GdYSi
ZrSi+Ta	YbGdYSi	GdYSi
ZrSi+Ta	YbGdSi	GdYSi-X
HfSi + Si	YbGdSi	GdYSi-X
HfSi + YSi	YbGdSi	
HfSi+Ysi+Si YbSi	YbGdSi YbGdSi YbSi	
HfSi + YbSi		
GdYbSi(Hf)		
YYbGdSi(Hf)	YbYSi YbHfSi YbHfSi YbHfSi YbHfSi YbHfSi YbSi	

EB-PVD

HfO₂-Si;
REHfSi
GdYSi
GdYbSi
NdYSi

APS*

HfO₂-Si
YSi+RESilicate
YSi+Hf-RESilicate

Hf-RESilicate

Hf-RE-Al-Silicate

APS*: or plasma spray related processing methods

FurnaceLaser/CVD/PVD

REHfSi

Used in ERA components as part of bond coat system

Used also in ERA components
Used in ERA components as part of bond coat system

Process and composition transitions

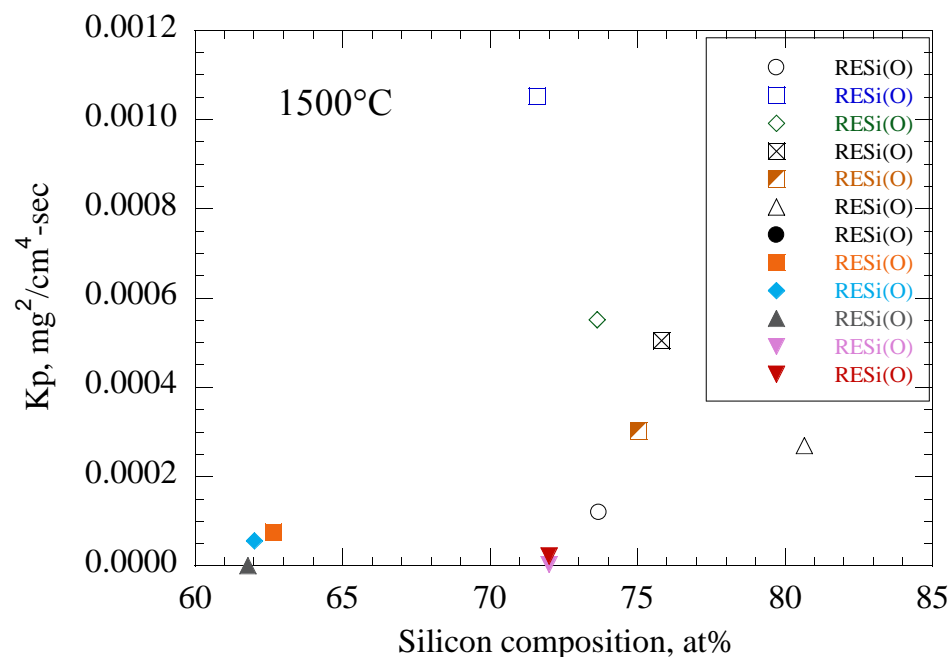


Oxidation Resistance of Doped Rare Earth Silicide - Effect of Stoichiometry

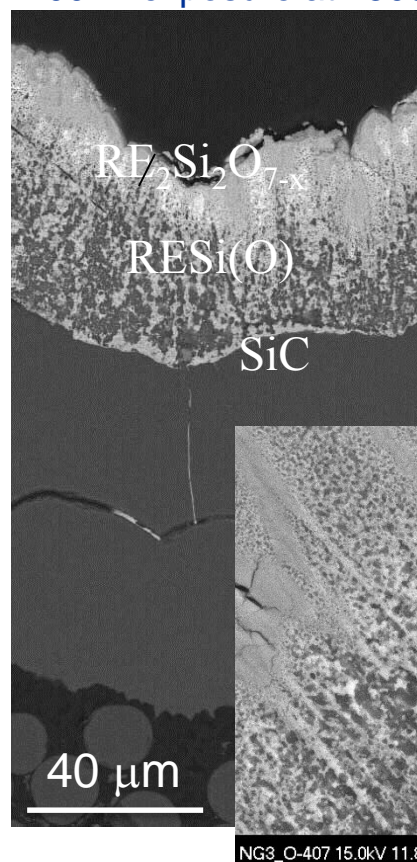
(oxidation vs. atomic percent Si)

- Thermogravimetric analysis (TGA) in dry O₂ at 1500°C, using SiC/SiC CVI CMC substrate
- Excellent oxidation resistance with composition optimizations
- “Protective” scale of rare earth di-silicate formed (10-15 micrometers)

Multicomponent doped RE Silicide system after 100 hr exposure at 1500°C in O₂



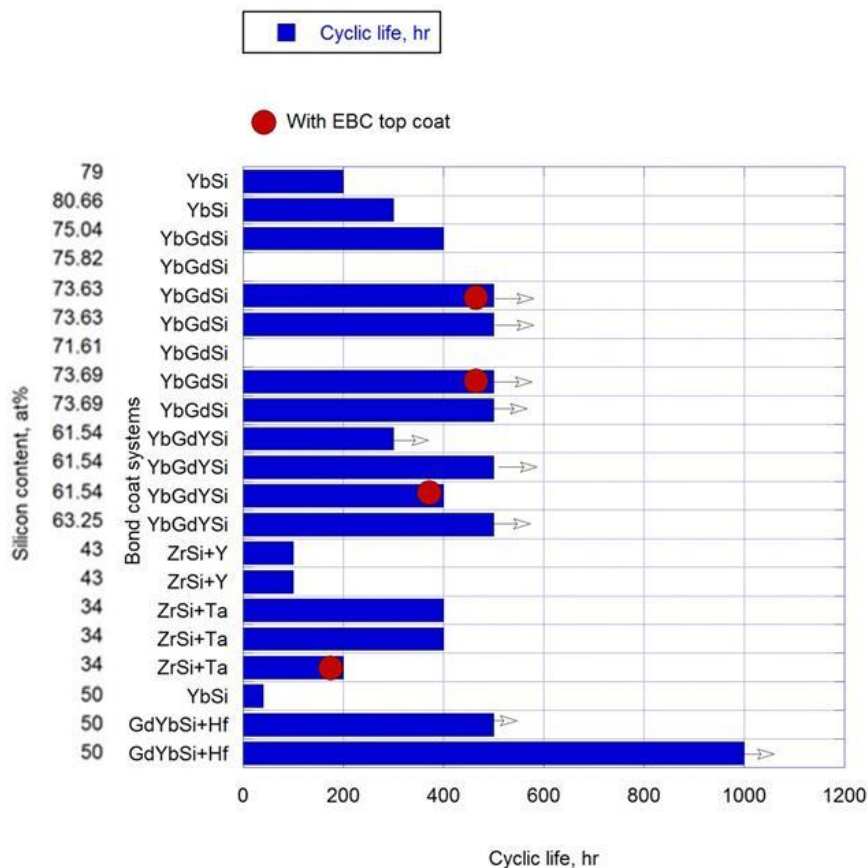
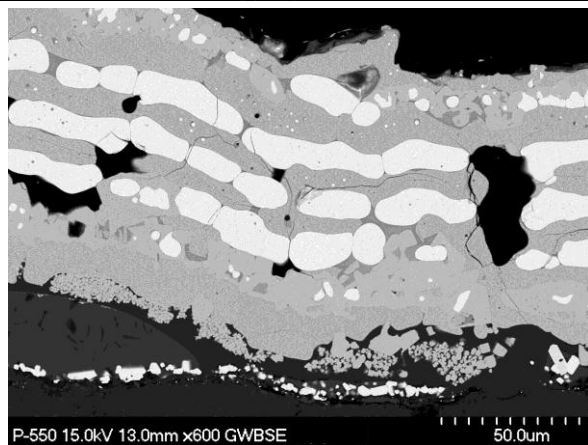
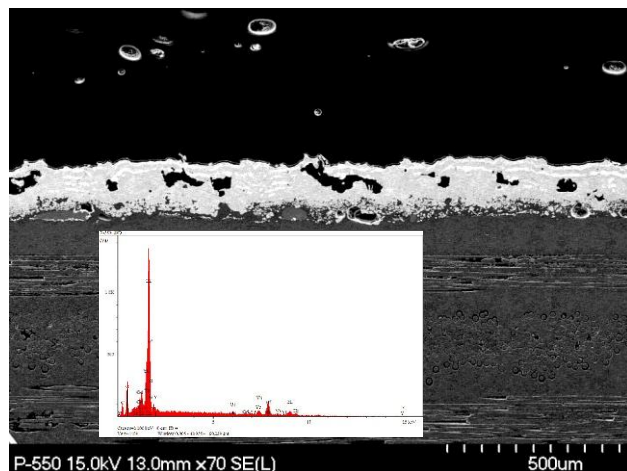
Oxidation rate constant vs. Si content



NG3_O-407 15.0kV 11.8mm x5.00k SE(L) 4/16/2013 10.0um

High Stability Rare Earth Silicon Bond Coat with High Melting Point Coating Compositions: Designed with Improved Temperature Capability and CMAS Resistance

- Furnace cyclic or high heat flux test life evaluated at 1500°C up to 1000 hours

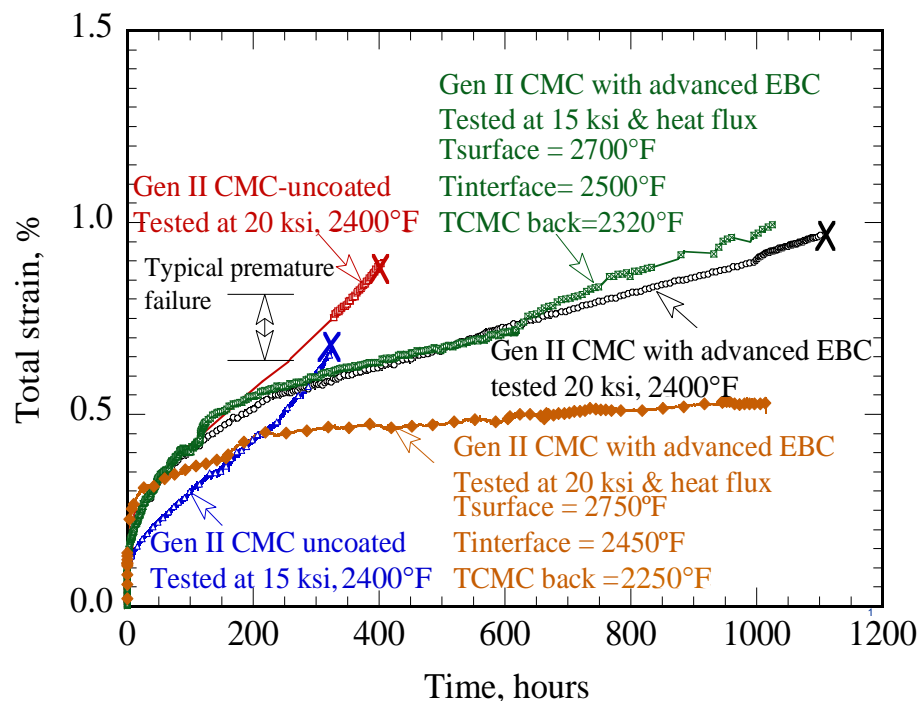
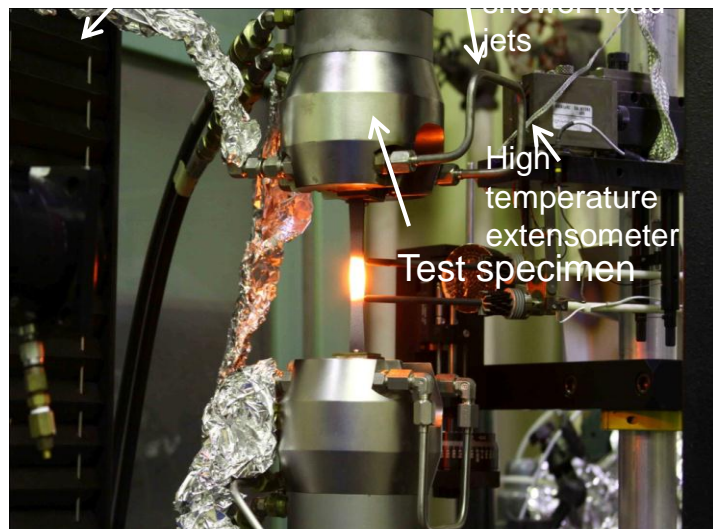
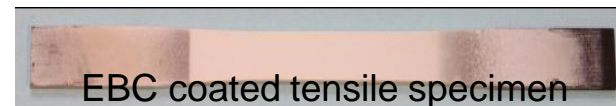


An Yb-Gd 2700°F EBC bond coat showed 500hr cyclic durability

FCT life of NASA RE-Si (O) series coatings

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

- Advanced high stability multi-component hafnia-rare earth silicate based turbine environmental barrier coatings being successfully tested for 1000 hr creep rupture
- EBC-CMC fatigue and environmental interaction currently being emphasized



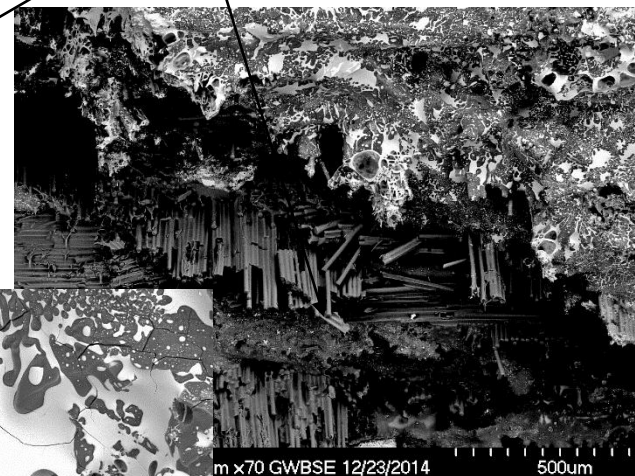
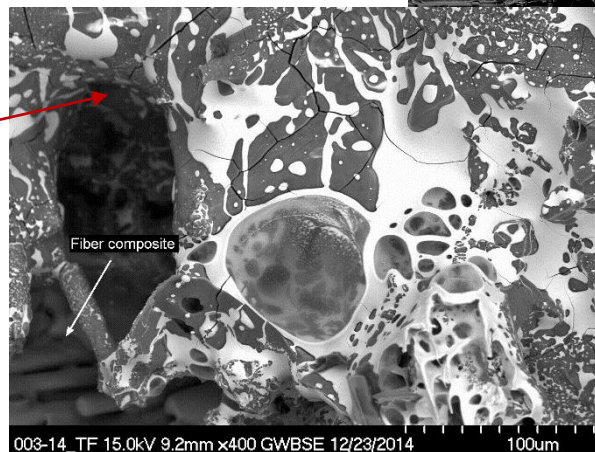
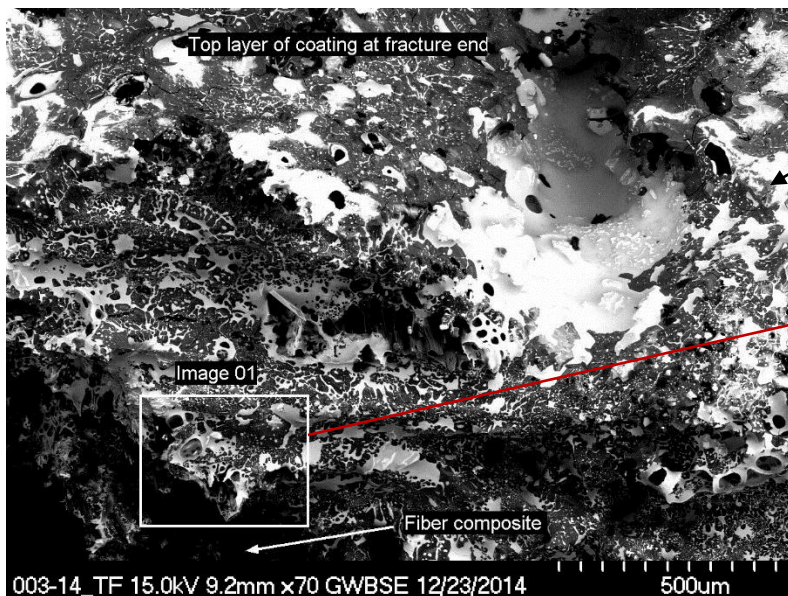
Thermal Gradient Tensile Creep Rupture Testing of Advanced Environmental Barrier Coating SiC/SiC CMCs

- Controlling CMAS wetting, viscosity, stability and melting points
- Providing better EBC protections for CMCs in CMAS environments
- EBC durability initially validated under long-term CMAS-mechanical loading

400 hr, 69 MPa creep rupture at EBC surface temperature 1400°C

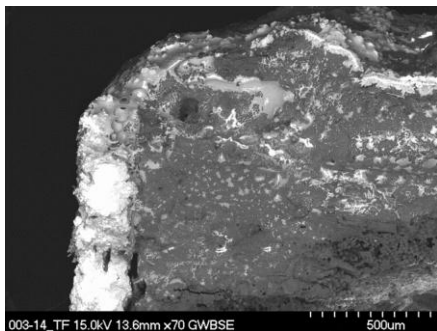


202 hr, 69 MPa creep rupture at EBC surface temperature 1540°C EBC, 1650°C+; CMC failure

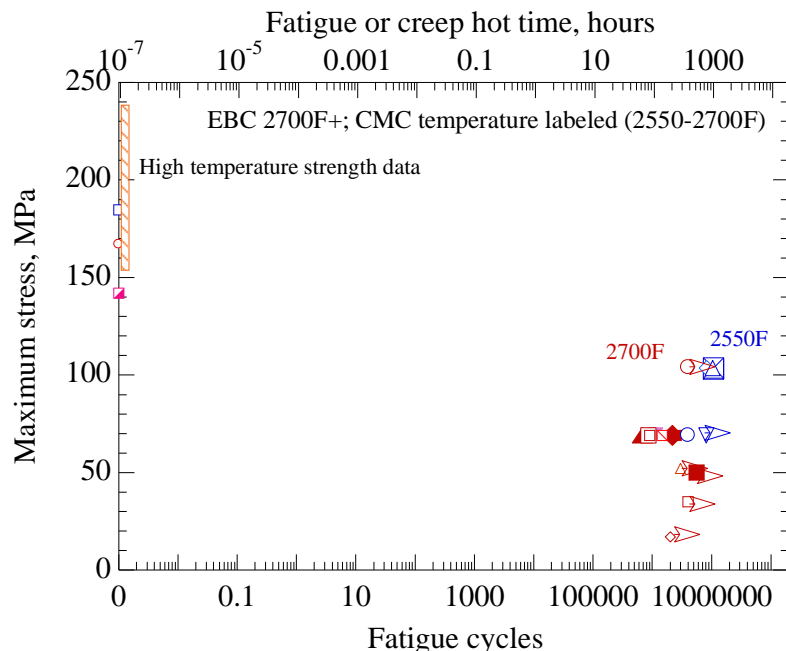


Creep-Fatigue of EBCs-CMCs in Complex Heat Flux and Simulated Engine Environments

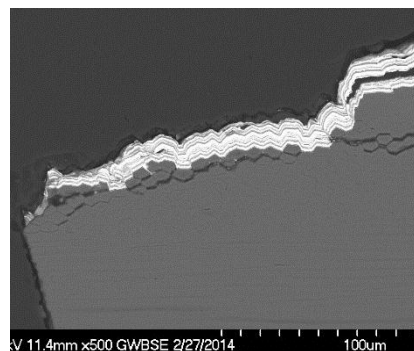
- Long-term creep and fatigue used to validate EBCs at various loading levels
- Demonstrated 2700°F EBC and bond coat capability in complex environments



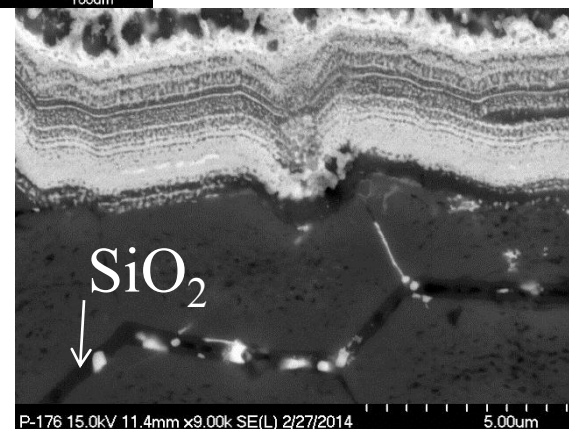
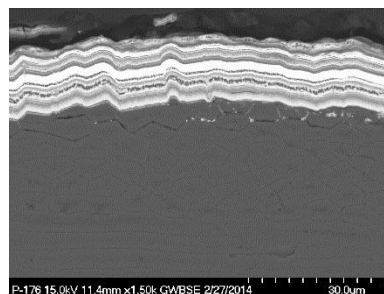
Fracture surface; 200+ hr at 2700°F+ (1482°C) creep rupture testing with CMAS; Advanced EBC protected CMCs



Stress-oxidation and stress-CMAS environmental testing



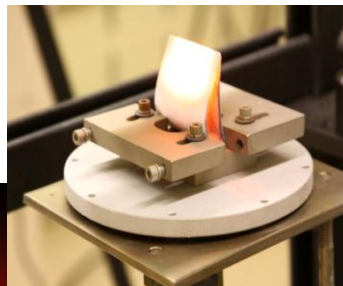
Advanced Bond Coat on CMC – intact after fatigue test with 15 ksi load and 2600-2700°F surface temperature for 460 hot hours



Advanced Bond Coat on CMC – intact after fatigue test with 15 ksi load and 2600-2700°F surface temp for 460 hot hours

Advanced Rig Tests for SiC/SiC CMC EBC Demonstrations

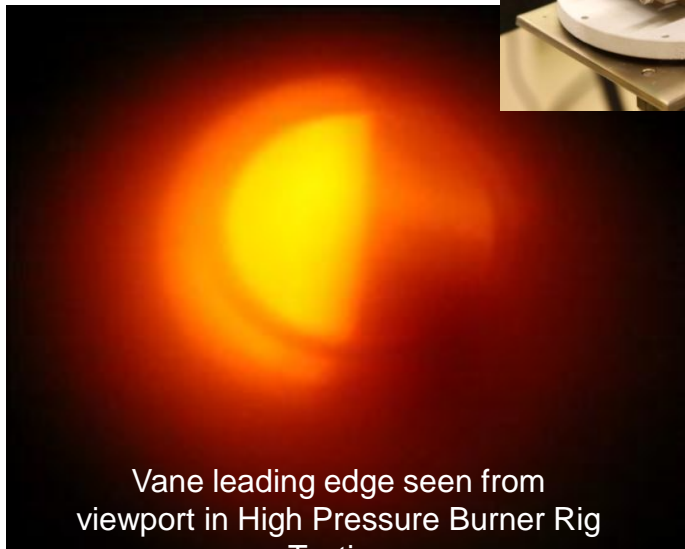
- Advanced EBC coated turbine airfoils and subelements demonstrated in high pressure burner rig and high heat flux laser rig simulated engine environments



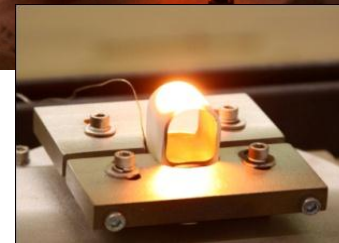
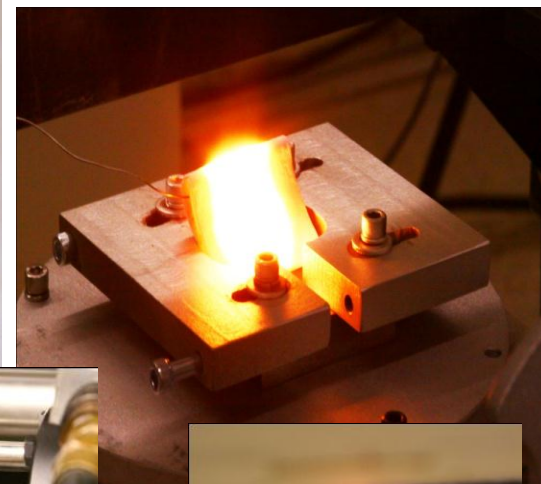
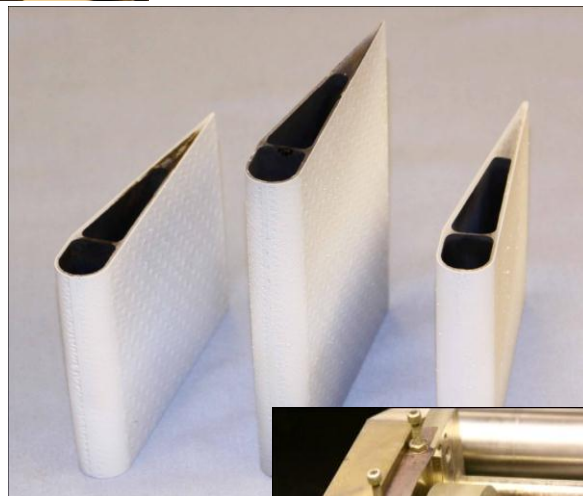
*50 hr EBC-CMC
vane laser rig
testing*



*50 hr EBC-2.5D
CMC Sub-element
demo in HPBR*



Vane leading edge seen from
viewport in High Pressure Burner Rig



NASA EBC coated turbine airfoils and testing

The Advanced EBC on SiC/SiC CMC Turbine Components Successfully Tested for Rig Durability in NASA High Pressure Burner Rig

- NASA advanced EBC coated turbine vane subcomponents tested in the NASA High Pressure Burner Rig simulated engine environments (up to 240 m/s gas velocity, 10 atm), reaching TRL of 5
- Combustor EBC-CMC also demonstrated



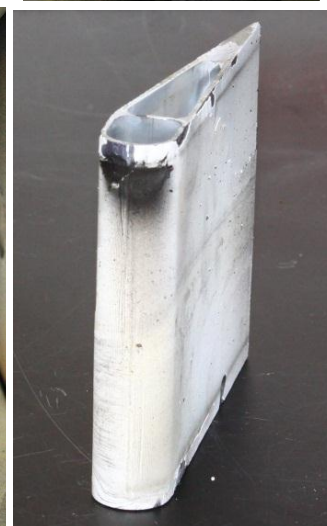
EBC coated SiC/SiC CMC inner and outer liner components



EBC Coated CVI SiC/SiC vane after 31 hour testing at 2500°F+ coating temperature



EBC Coated Prepreg SiC/SiC vane after 21 hour testing at 2500°F

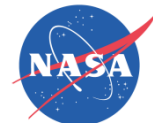


EBC Coated Prepreg SiC/SiC vane tested 75 hour testing at 2650°F



Summary

- **Advanced high temperature SiC/SiC CMC environmental barrier coatings development**
 - Developed new compositions for meeting current coating and component performance requirements
 - Emphasized advanced thinner coating configurations with long-term stability and durability
 - Demonstrated higher temperature capability, improved environmental stability and coating thermal - mechanical stress and creep-rupture resistance
 - Focused on coating composition developments and architecture designs to improve stability and durability at 2700-3000°F
- **Advanced high temperature SiC/SiC CMC environmental barrier coatings Testing Developments**
 - Developed advanced coating and subelement testing methods relevant to turbine CMC combustors and vanes, establishing initial property database, degradation and lifing prediction models
 - Developed advanced combustor and turbine vane EBC component technologies, and demonstrating the full feature EBC - CMC sub-components in relevant rig simulated engine environments



Advanced Environment Barrier Coating Material System Development - Emerging Opportunities

- High stability, low expansion top coat development
 - Advanced high stability nano-phase composite designs
 - Rare earth dopants and silica clusters for improved thermal stability
 - Transition metal dopants for phase stability and temperature capability of EBCs
 - Reducing interface reactions – self-forming diffusion and reaction barriers
 - Minimizing grain boundary Si segregation, SiO_2 phase formation, and low melting phase formation
 - Implementing new architecture PVD composite columnar structures to achieve high stability, high strength, low expansion, low conductivity and high erosion resistance
 - Thin coating configurations emphasized for both turbine CMC airfoil and advanced combustor applications
 - Advanced non-line-of-sight hybrid, high efficiency PVD and CVD processing for economical airfoils inner and outer surfaces, cooling holes with high coat EBC compositions
 - High adhesion and intergraded EBC/CMC interfaces
- Low stress, strain tolerant interlayer and high strength bond coats
 - Thin, nano-layered layered *high toughness* coatings with minimum thickness
 - Novel compositional and architectural designs to achieve maximum energy dissipation and durability
 - High strength and high toughness, combined with optimized strain tolerance for superior erosion and impact resistance
- Environmental barrier and high strength bond coats
 - Low expansion, high stability, low diffusivity, high strength and strain tolerance to allow a few micron thick coating designs
 - Self repairing and/or self-growing of slow growth adherent protective coatings, i.e., Design of alloys, intermetallic and composites capable of self growing EBCs needed
- Multifunctional compositions for high temperature sensing, health monitoring, and reduced heat transfer



Acknowledgements

- **The work was supported by NASA Environmentally Responsible Aviation (ERA) Project and Fundamental Aeronautics Program (FAP) Aeronautical Sciences Project**

NASA EBC-CMC Team, In particular, Jim DiCarlo, Jim Smialek, Dennis Fox, Robert A. Miller, Janet Hurst, Martha Jaskowiak, Ram Bhatt, Bryan Harder, Mike Halbig

Collaborators include:

Sulzer Metco (US) - Mitch Dorfman; Chis Dambra

Directed Vapor Technologies, International – Derek Hass and Balvinder Gogia

Praxair Surface Technologies – John Anderson and Li Li

Southwest Research Institute – Ronghua Wei (PVD coating processing)
in supporting the coating processing