



DEVELOPMENT OF ADVANCED ENVIRONMENTAL BARRIER COATINGS FOR SIC/SIC COMPOSITES At NASA GRC: PRIME-RELIANT DESIGN AND DURABILITY PERSPECTIVES

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Advanced Ceramic Matrix Composites:
Science and Technology of Materials, Design, Applications, Performance and Integration
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NASA's Advanced 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

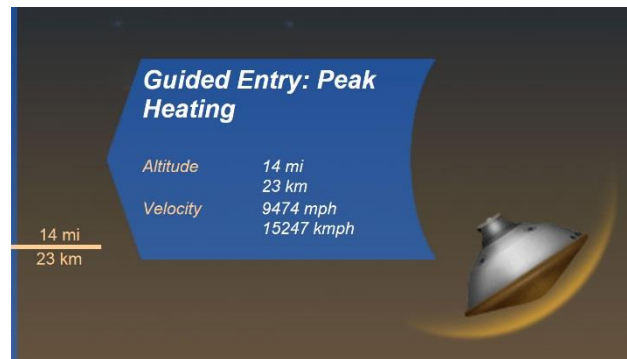
- Help achieve future engine temperature and performance goals
- Ensure system durability – towards prime reliant coatings and material systems
- Establish database, design tools and coating lifing methodologies
- Improve technology readiness



Fixed Wing Subsonic and Supersonics Aircraft



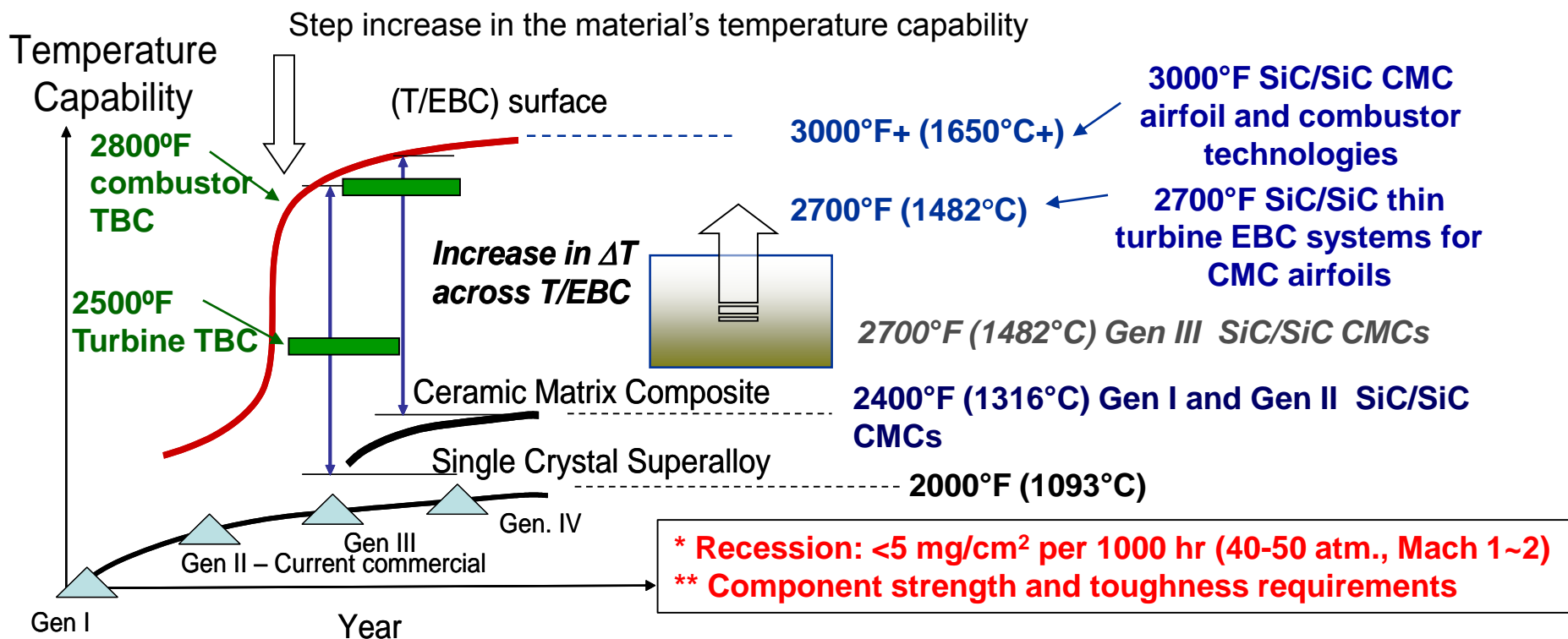
Hybrid Electric Propulsion Aircraft



Entry, Descending and Landing: Ultra High Ceramics and Coatings (UHTCC)

NASA Environmental Barrier Coating Development Goals

- Emphasize temperature capability, performance and durability
- Develop 2700°F environmental barrier coating technologies
 - For 2400°F (1316°C) and 2700°F (1482°C) Ceramic Matrix Composites (CMCs) in support of next generation turbine engines
 - Focus on advanced CMC combustors and highly loaded turbine airfoils
 - Particularly also emphasize highly loaded turbine blades





Outline

- **Environmental barrier coating systems: towards Prime-Reliant systems**
 - Thermomechanical, environment and thermochemical stability of EBCs
 - Prime-reliant EBCs, initial design approach and requirements

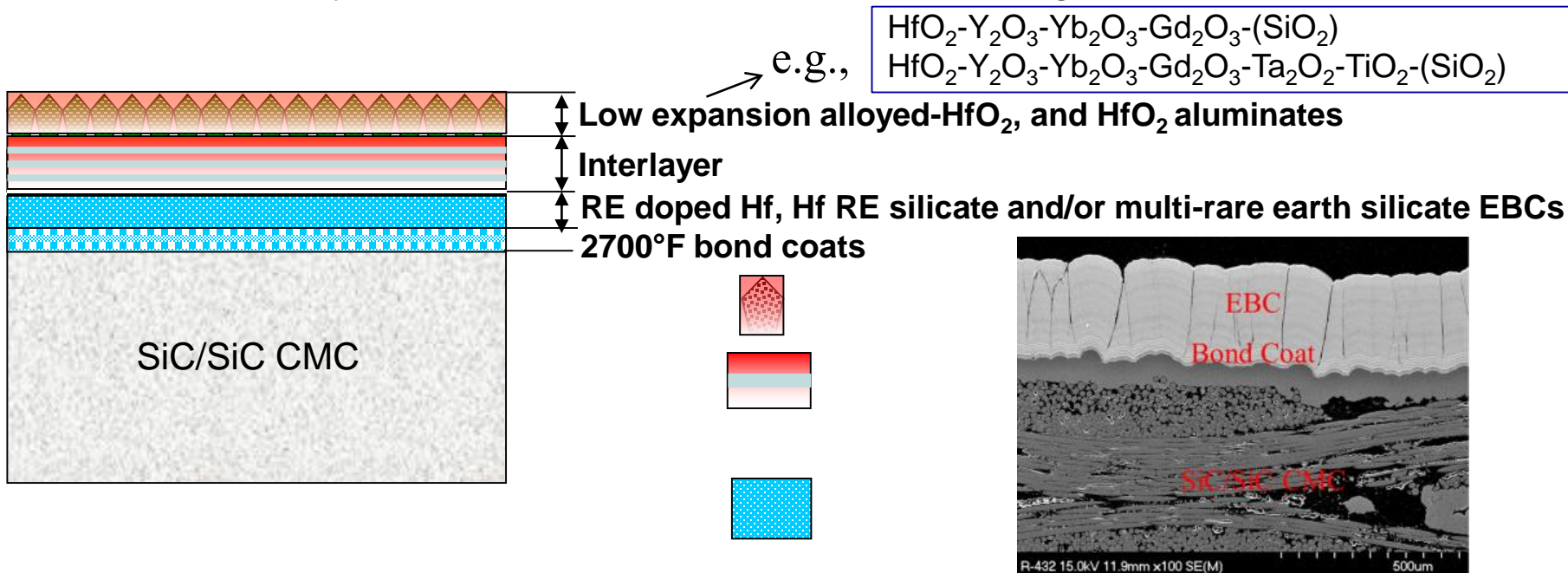
 - **Advanced environmental barrier coating systems (EBCs) for CMC airfoils and combustors**
 - NASA advanced turbine EBC coating system status
 - Development and testing, durability perspectives

 - **Design tool and life prediction perspectives of coated CMC components**
 - Advanced Testing
 - Emphasizing thermomechanical, environment and thermochemical interactions

 - **Summary and future directions**
-

Prime-Reliant EBC Systems for Si-Based Ceramic Matrix Composites

- NASA's advanced environmental barrier and bond coat development: Prime-reliant designs
 - High toughness and low conductivity EBC top coat
 - Alternating Composition Layered Coatings (ACLCs) and composite coatings, including improving impact resistance
 - Advanced doped multicomponent EBCs
 - Prime-reliant designed bond coats
 - Ultimately environmental resistant CMCs with high thermal conductivities



Fundamental Recession Issues of CMCs and EBCs

- Recession of Si-based Ceramics

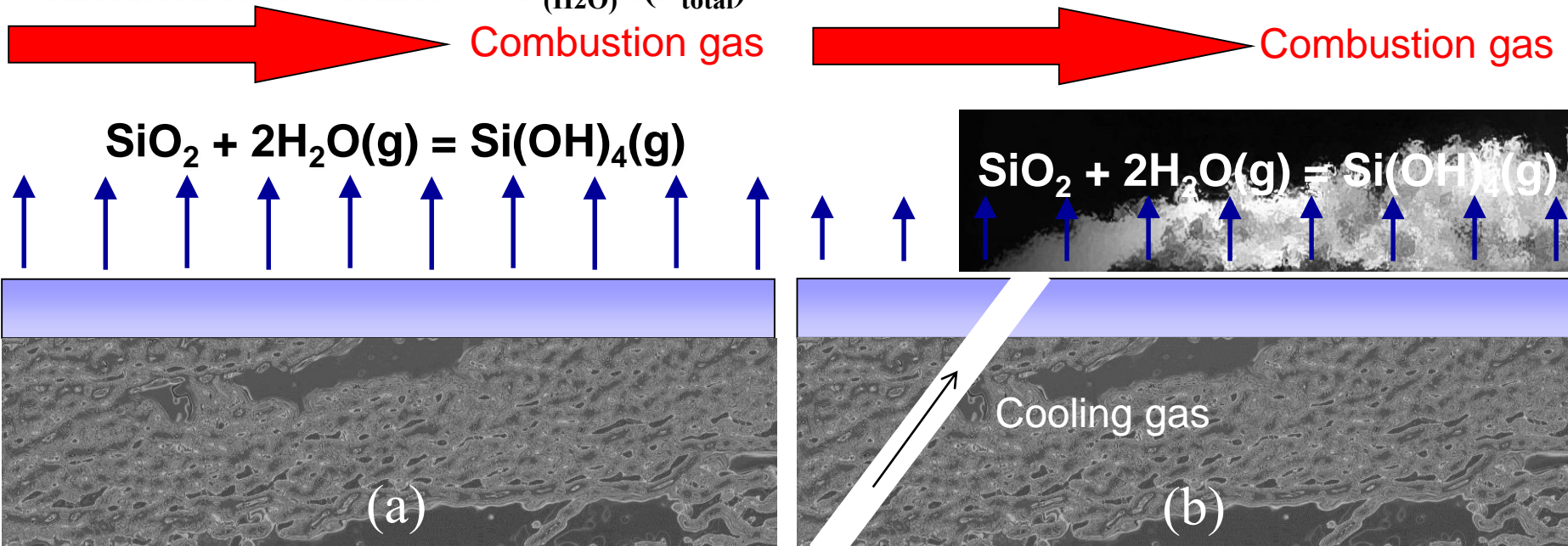
(a) Convective; (b) Convective with film-cooling

- Low SiO_2 activity EBC systems

- Advanced rig testing and modeling

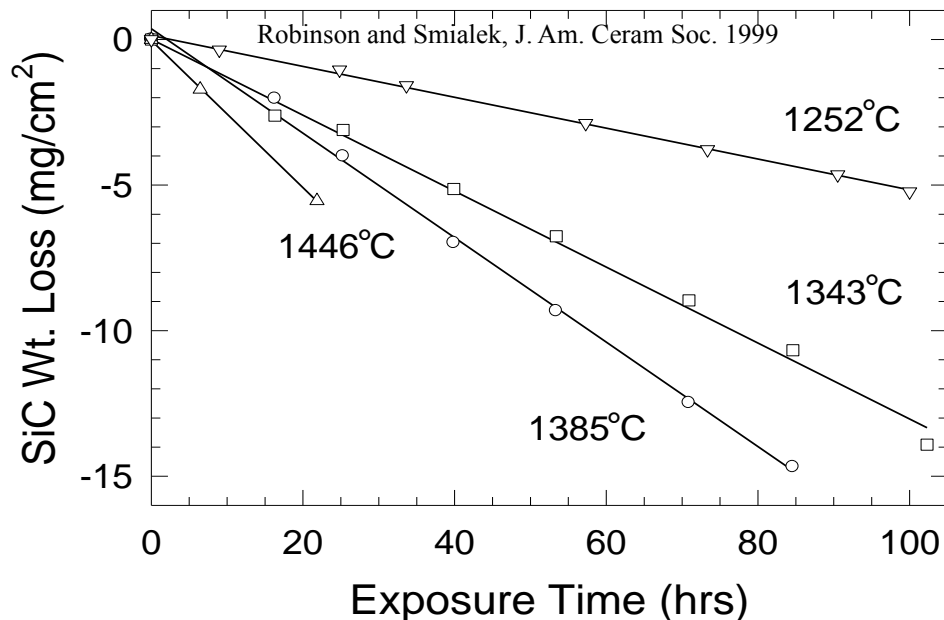
Complex recession behavior of CMC and EBCs studied in NASA High Pressure Burner Rig, included film-cooled recession

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

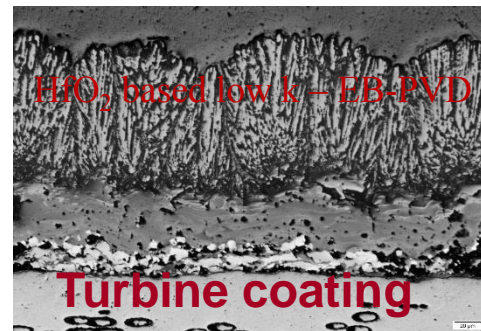
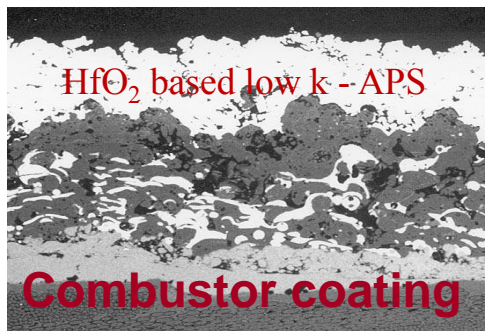
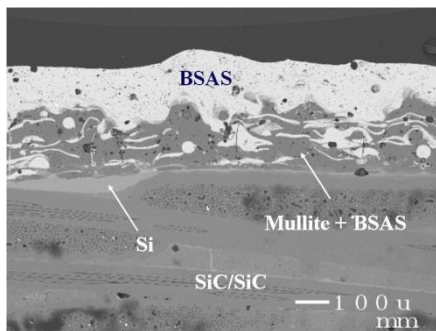


Fundamental Recession Issues of CMCs and EBCs - Continued

- EBC stability evaluated on SiC/SiC CMCs in high velocity, high pressure burner rig environment

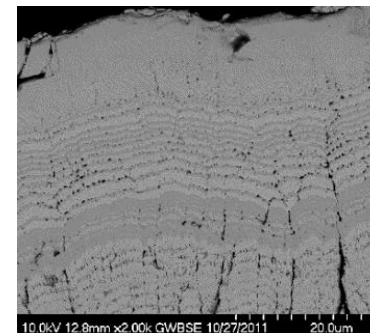
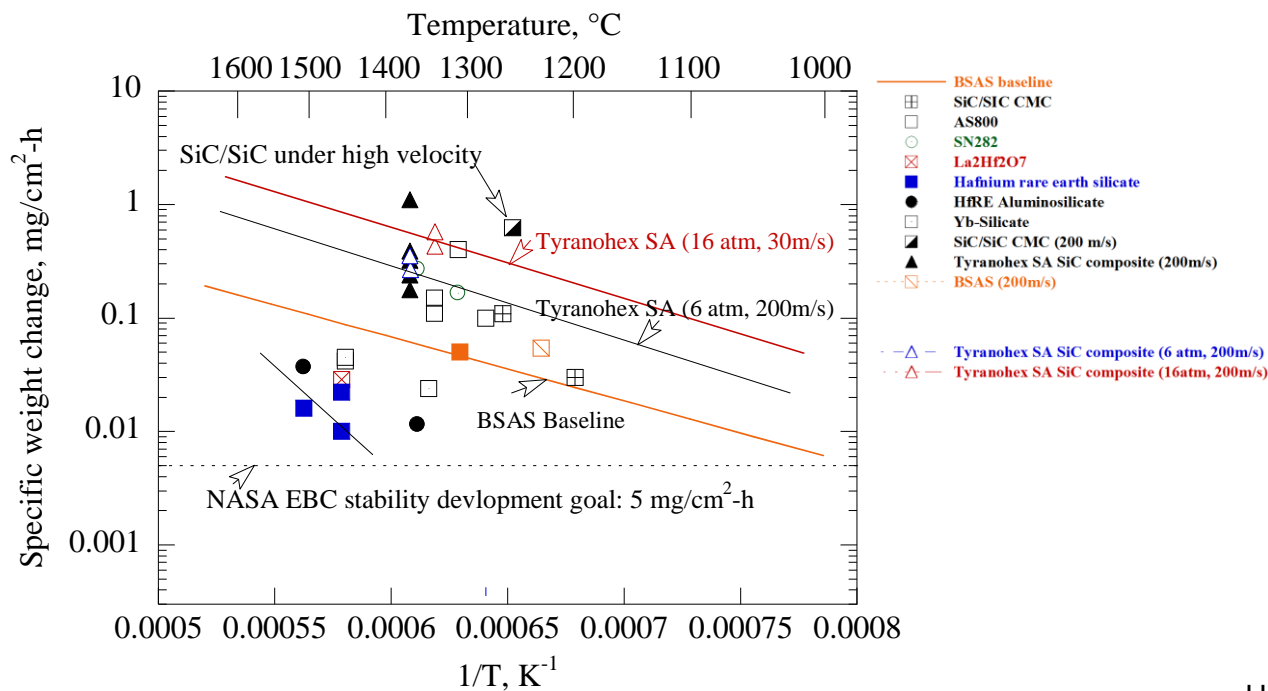


- Early generations of environmental barrier coatings - EBC systems, improved stability in turbine environments



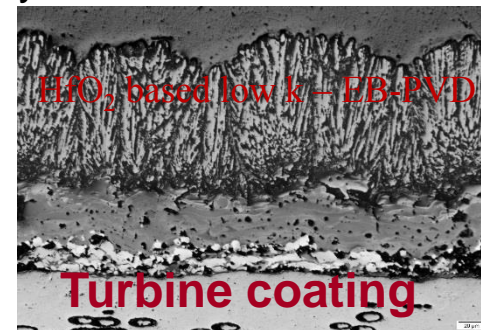
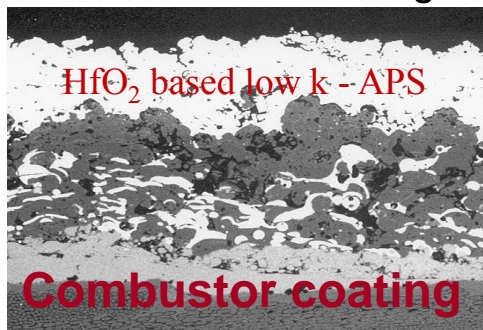
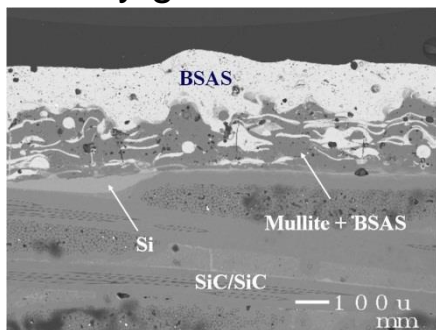
Fundamental Recession Issues of CMCs and EBCs - Continued

— EBC stability evaluated on SiC/SiC CMCs in high velocity, high pressure burner rig environment



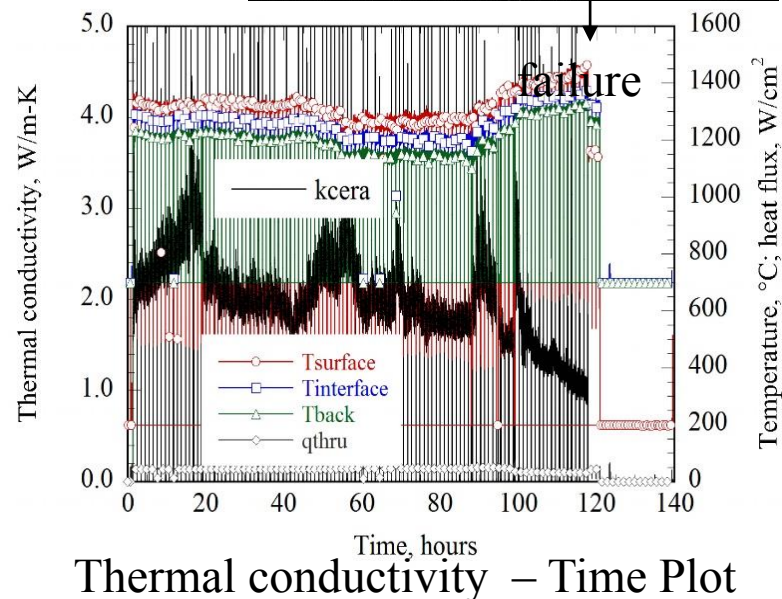
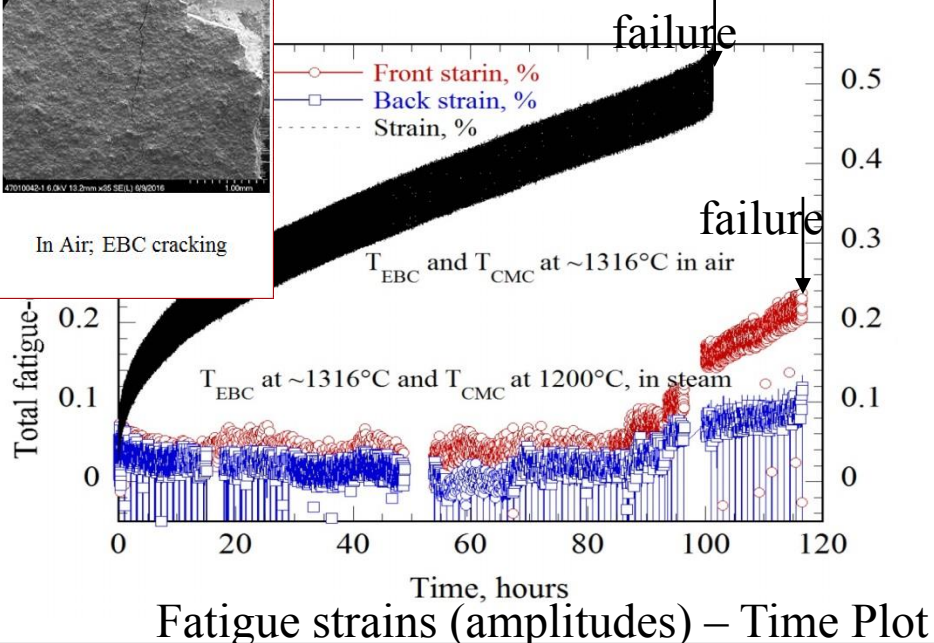
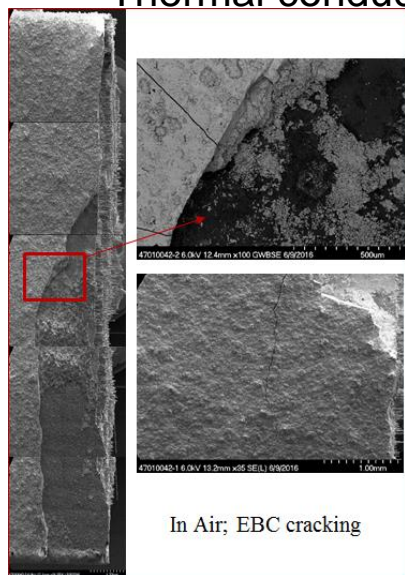
High pressure burner rig, 16 atm, 31 hr

- Early generations of environmental barrier coatings - EBC systems



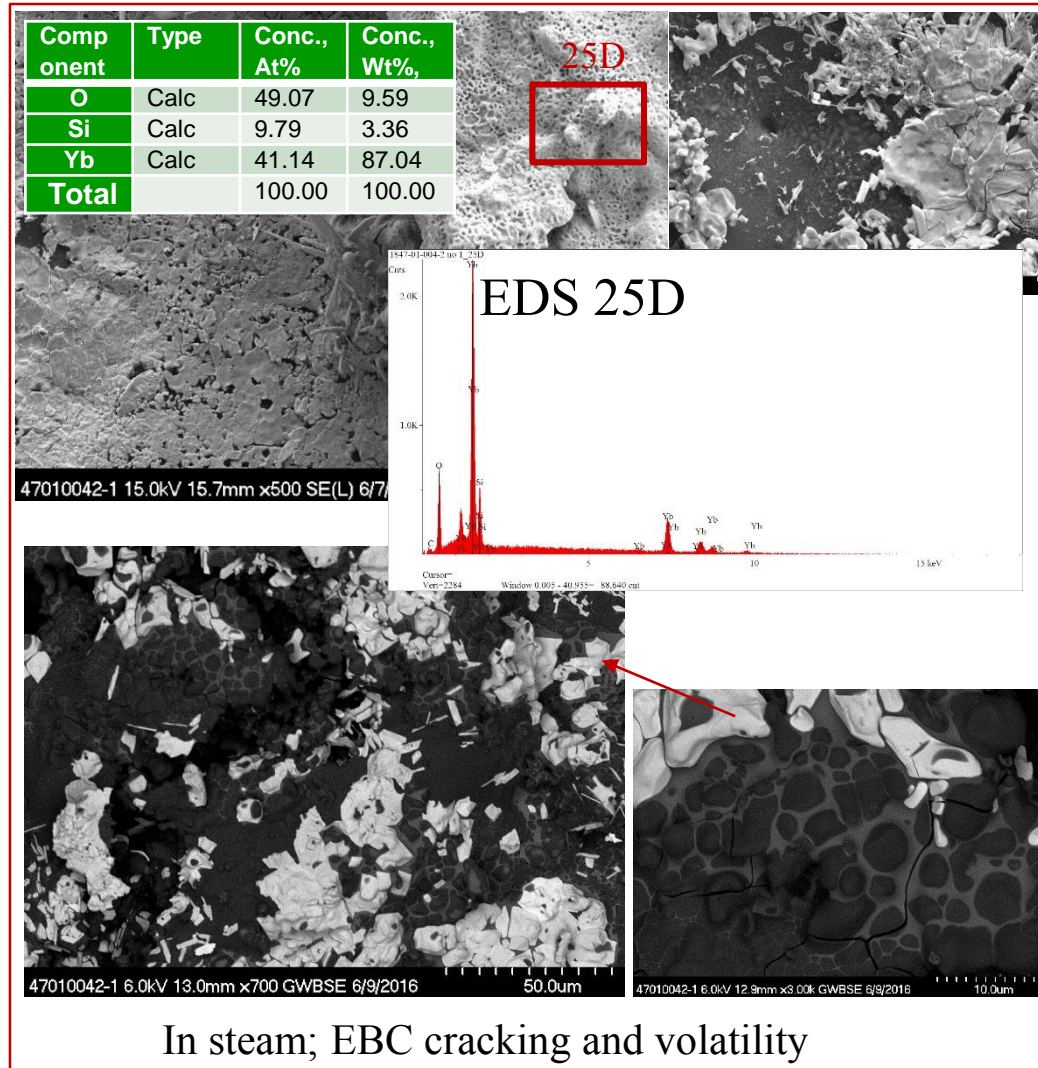
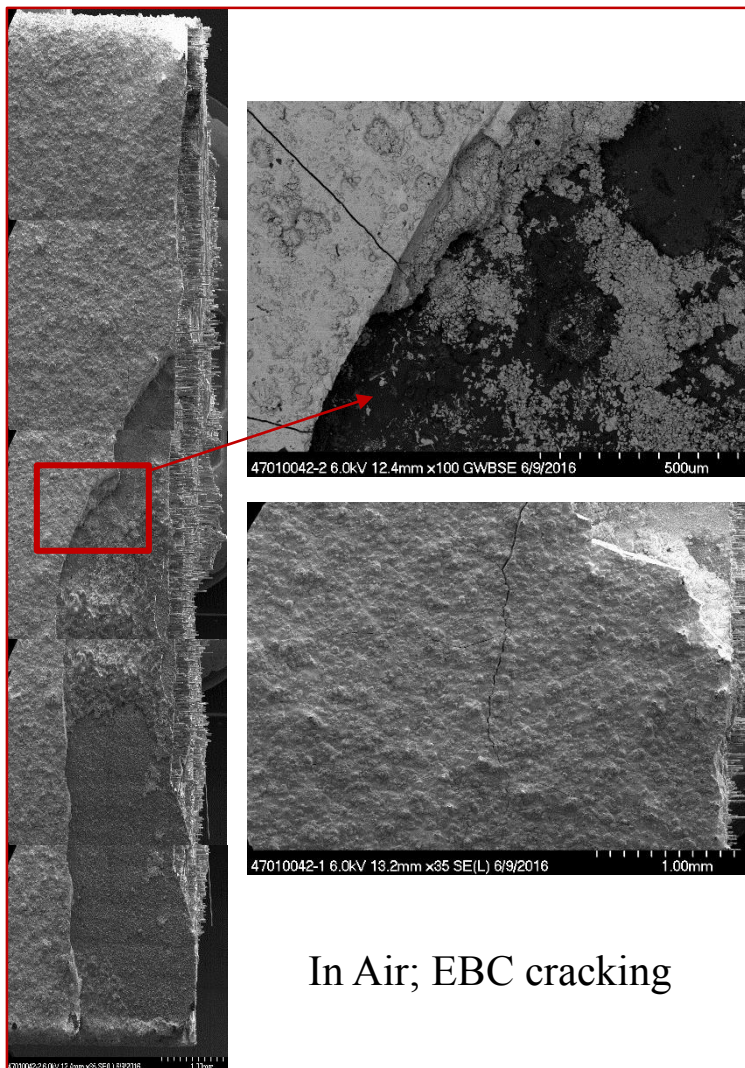
Environmental Barrier Coating in Simulated Load-Fatigue Testing Environments (Laser High Heat Flux Rig)

- EBC $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7/\text{Si}$ on Melt Infiltrated (MI) Prepreg SiC/SiC CMC substrates
- Tested in air, furnace isothermal at 1316°C ; and in heat flux steam, at $T_{\text{EBC}} 1316^\circ\text{C}$, T_{CMC} at $\sim 1200^\circ\text{C}$
- Lower CMC failure strain observed in the steam-heat flux test environment
- Ytterbium mono-silicate recession observed in the test
- Thermal conductivity for monitoring coating delamination



Environmental Barrier Coating in Simulated Load-Fatigue Testing Environments (Laser High Heat Flux Rig) - Continued

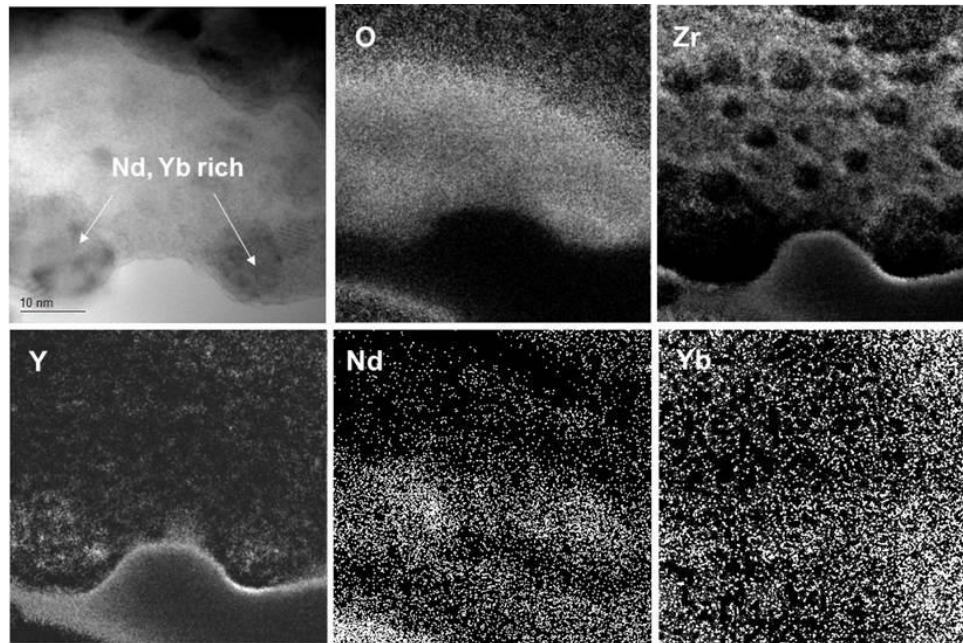
- Crack and recession failure in air and steam tests



Advanced NASA EBC Developments

NASA advanced EBC systems emphasizing high stability HfO_2 - and ZrO_2 - RE_2O_3 - SiO_2 EBC systems and HfO_2 -Si and Rare Earth – Silicon bond coat systems

- NASA multicomponent Rare Earth $\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$, such as $(\text{Yb,Gd,Y})_2\text{Si}_{2-x}\text{O}_{7-2x}$ environmental barrier systems
- Rare earth-doped HfO_2 - ZrO_2 , and HfO_2 -doped Rare Earth (RE) silicates are among the advanced EBC systems
 - Improved high temperature stability and creep strength;
 - Facilitating nano-cluster formation with high concentration rare earth compound phases;
 - Improving CMAS resistance

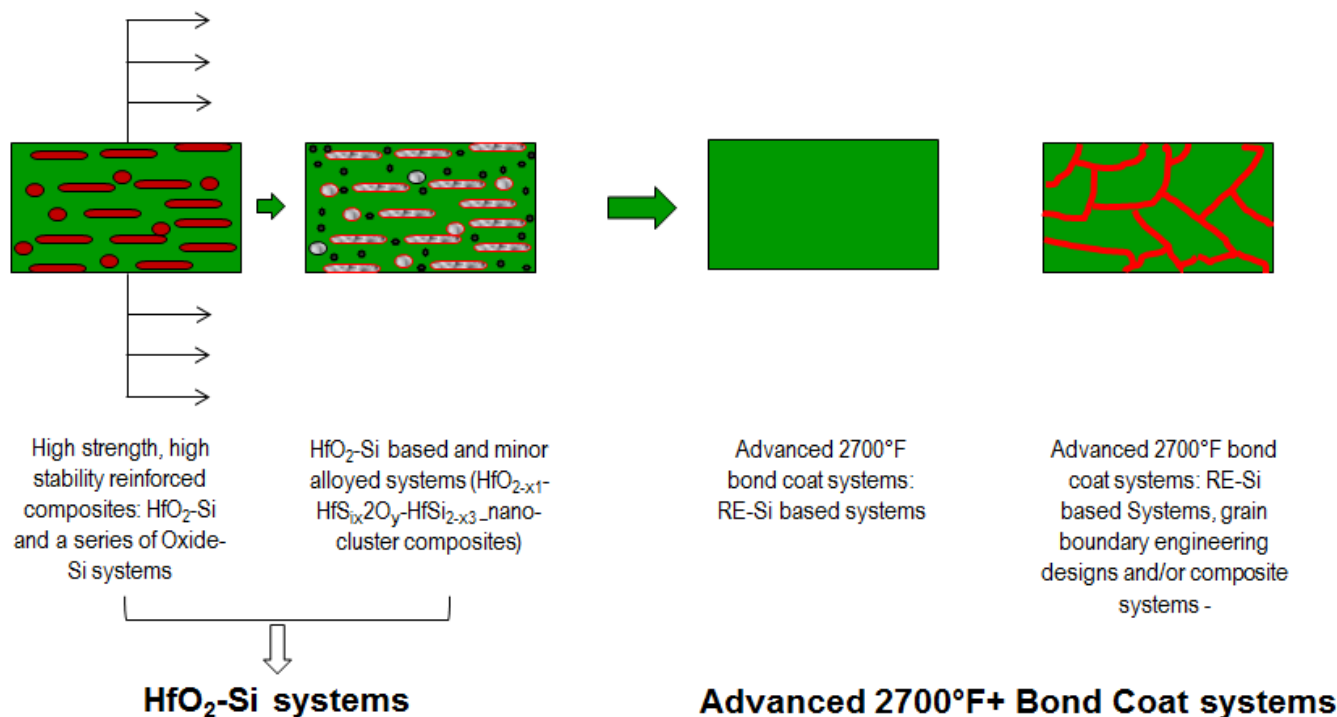


TEM-EELS
Composition map

NASA EBC Bond Coat Systems

NASA EBC Systems

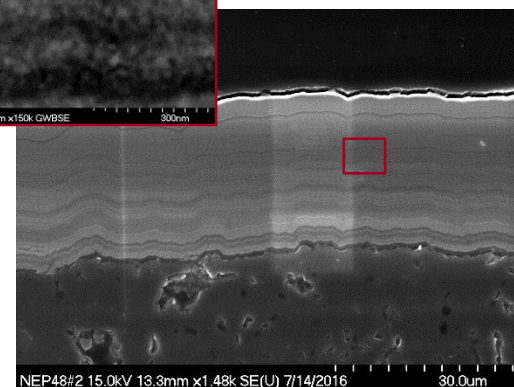
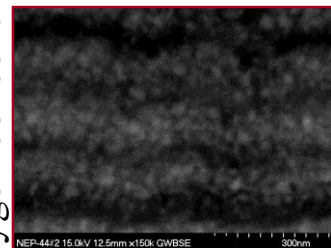
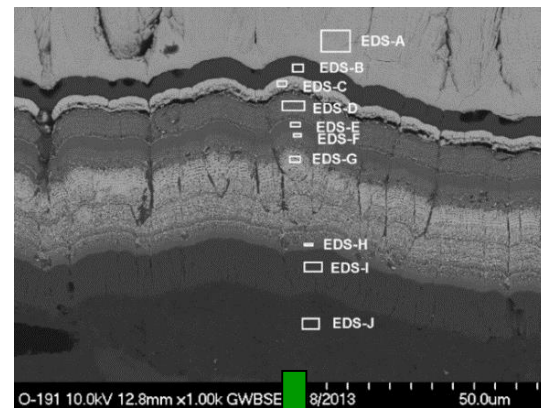
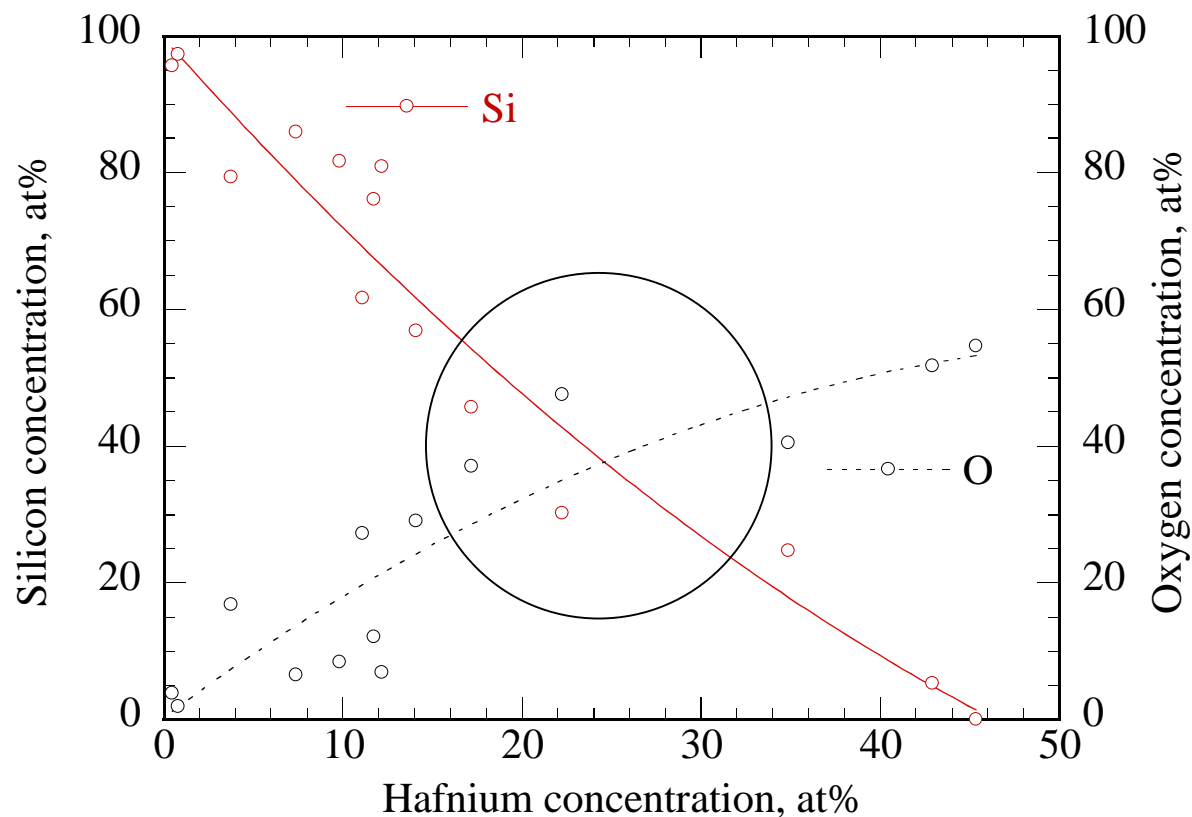
- HfO_2 - RE_2O_3 - SiO_2 / $\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$ environmental barrier systems
 - Controlled silica content and rare earth dopants to improve EBC stability, toughness, erosion and CMAS resistance
 - HfO_2 -Si based bond coat, controlled oxygen partial pressure
 - Advanced rare earth-Si composition systems for 2700°F+ long-term applications
- Early RE_2O_3 - SiO_2 - Al_2O_3 or YAG Systems
- Develop prime-reliant composite EBC-CMCs, HfSiRE(CN) systems



Bond coat systems for prime reliant EBCs; capable of self-healing

Development of Advanced HfO₂-Si Bond Coats

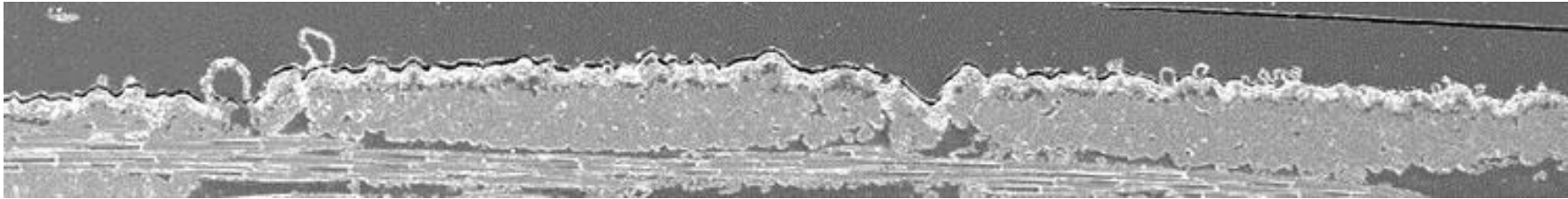
- Coating architecture and HfO₂ contents can be effectively controlled and optimized
- Si:Hf atomic ratio preferably 2:1 – 1:1
- Reported Spinodal decomposition of hafnium-silicate systems



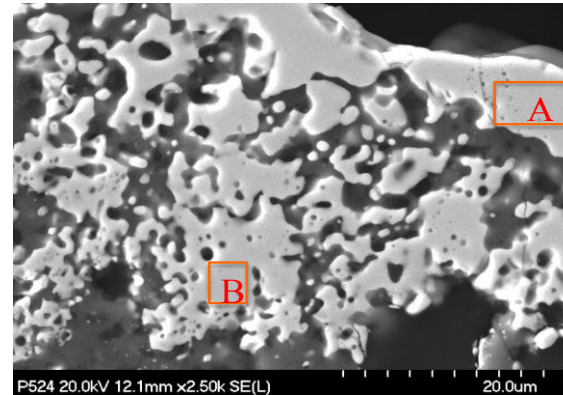
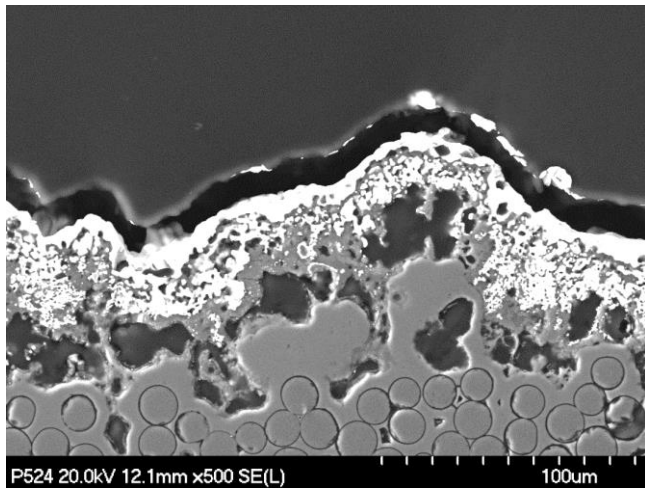
Microstructures of Furnace Cyclic Tested GdYbSi(O) EBC Systems



- Cyclic tested cross-sections of PVD processed YbGdSi(O) bond coat
- Self-grown rare earth silicate EBCs and with some RE-containing SiO₂ rich phase separations
- Relatively good coating adhesion and cyclic durability



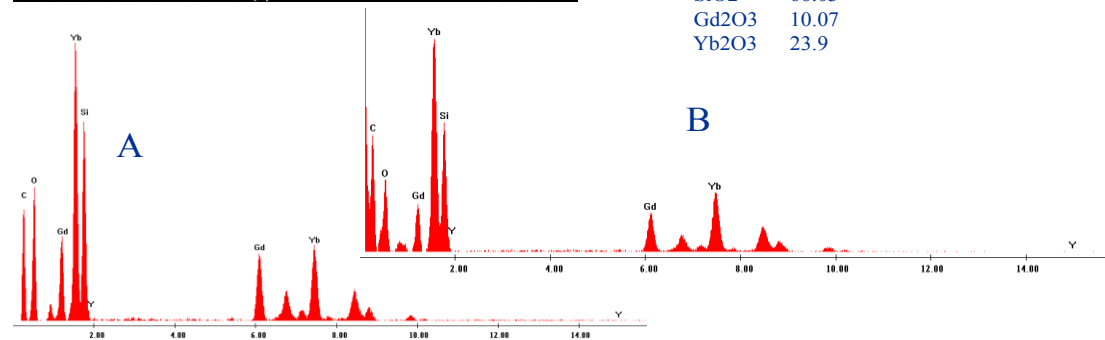
1500°C, in air, 500, 1 h cycles



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

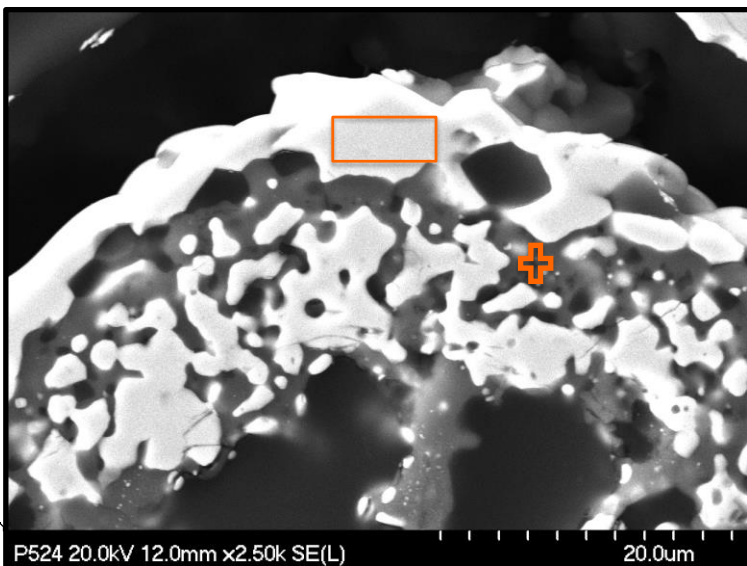
Composition (mol%) spectrum Area #1	
SiO ₂	67.98
Gd ₂ O ₃	11.95
Yb ₂ O ₃	20.07

Composition (mol%) spectrum Area #2	
SiO ₂	66.03
Gd ₂ O ₃	10.07
Yb ₂ O ₃	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 SiO₂ rich phase separations
- Interface growth instability



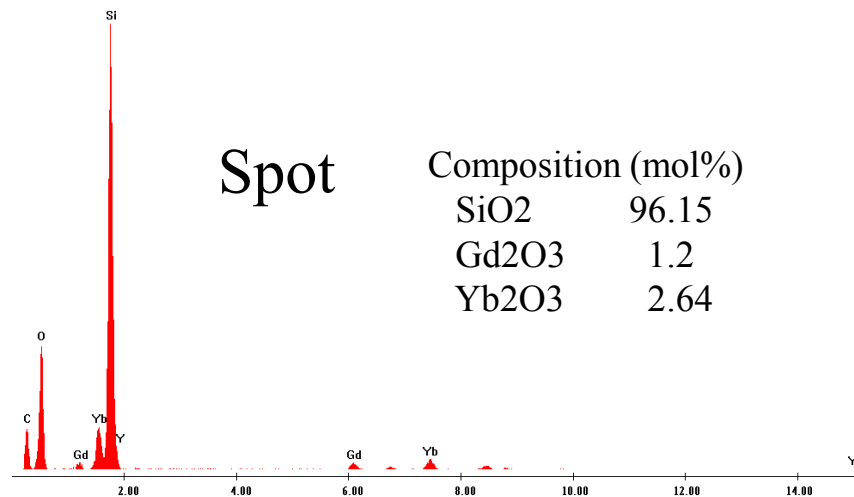
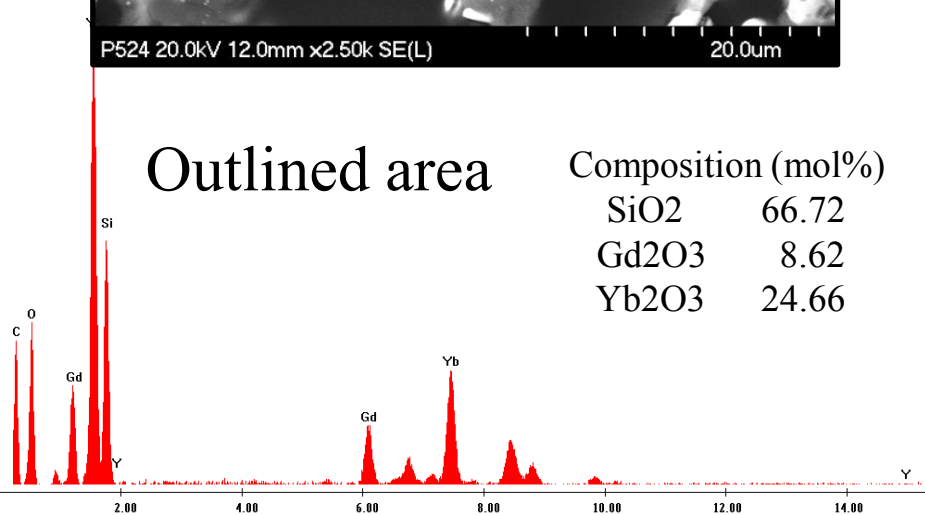
1500°C, in air, 500, 1 hr cycles

Outlined area

Composition (mol%)	
SiO ₂	66.72
Gd ₂ O ₃	8.62
Yb ₂ O ₃	24.66

Spot

Composition (mol%)	
SiO ₂	96.15
Gd ₂ O ₃	1.2
Yb ₂ O ₃	2.64



Advanced EBC developments – Some Hybrid Air-Plasma Spray/EB-PVD Turbine Combustor EBC Systems and Qualification Tests

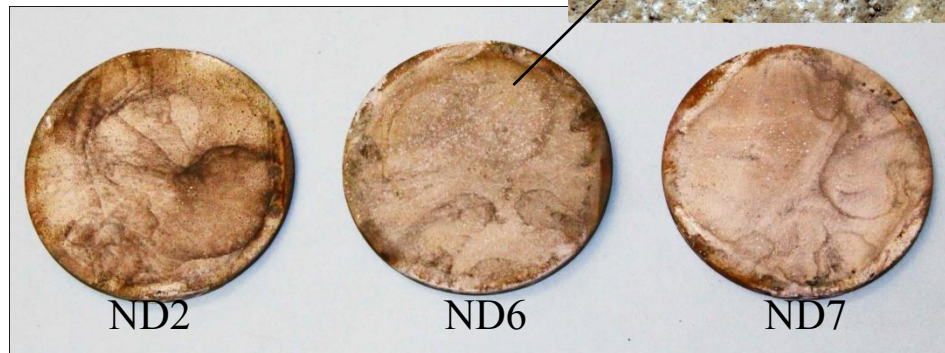
- Achieved low thermal conductivity ranging from 1.0 - 1.7 W/m-K
- Demonstrated high pressure environmental stability at 2600-2650°F, 12-20 atm. in the high pressure burner rig
- Surface recession or micro-spallation for less tougher coating systems



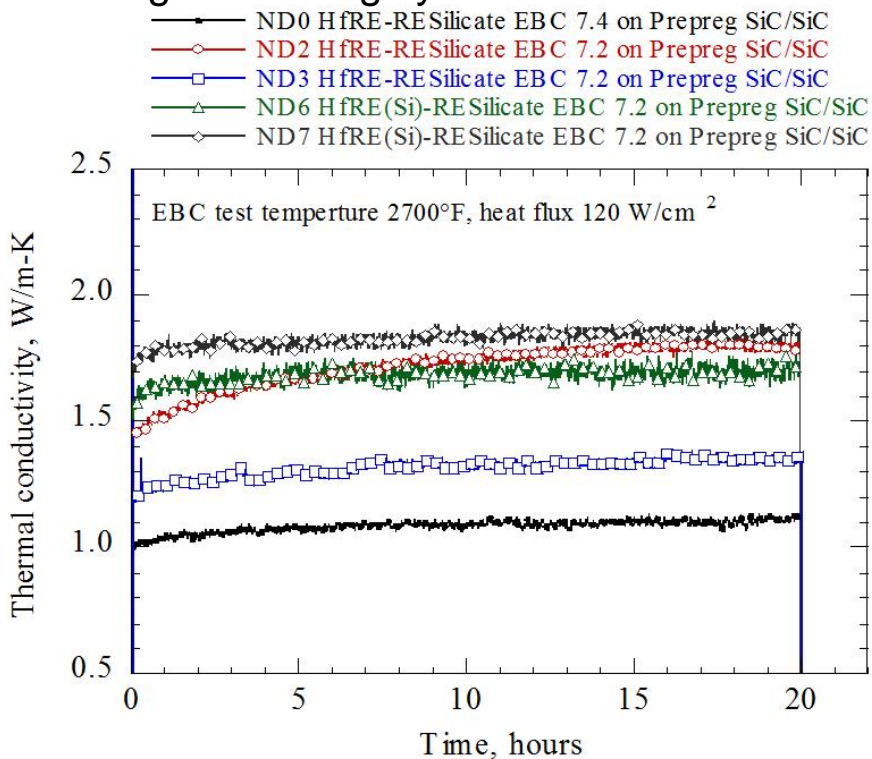
2" diameter ND3 EBC/SiC/SiC specimen after testing in the high pressure burner rig At 2600°F+, 200 m/s



Surface spallation

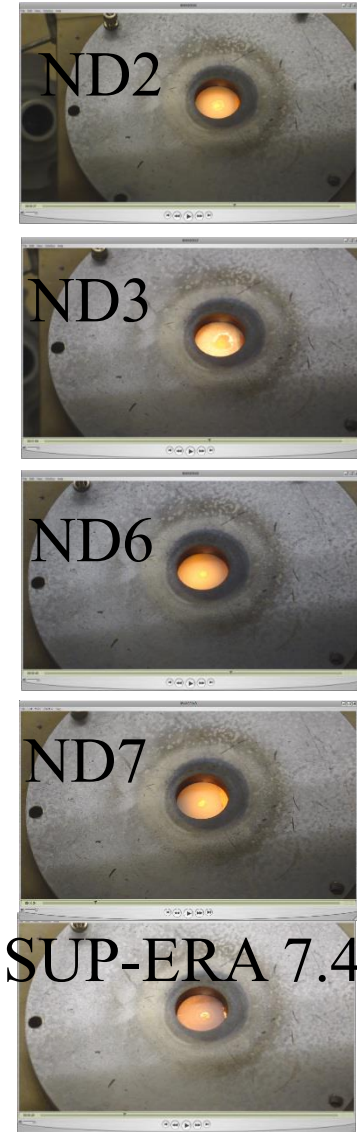
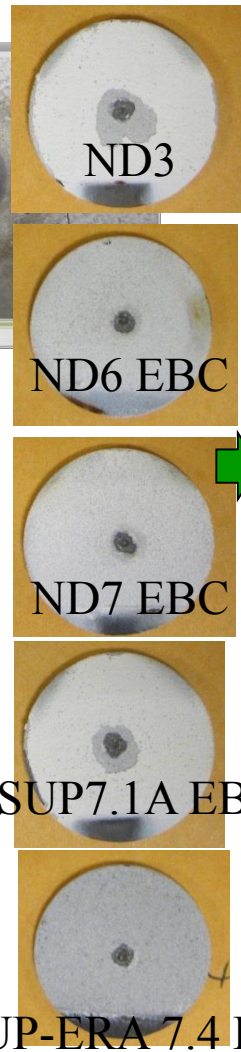
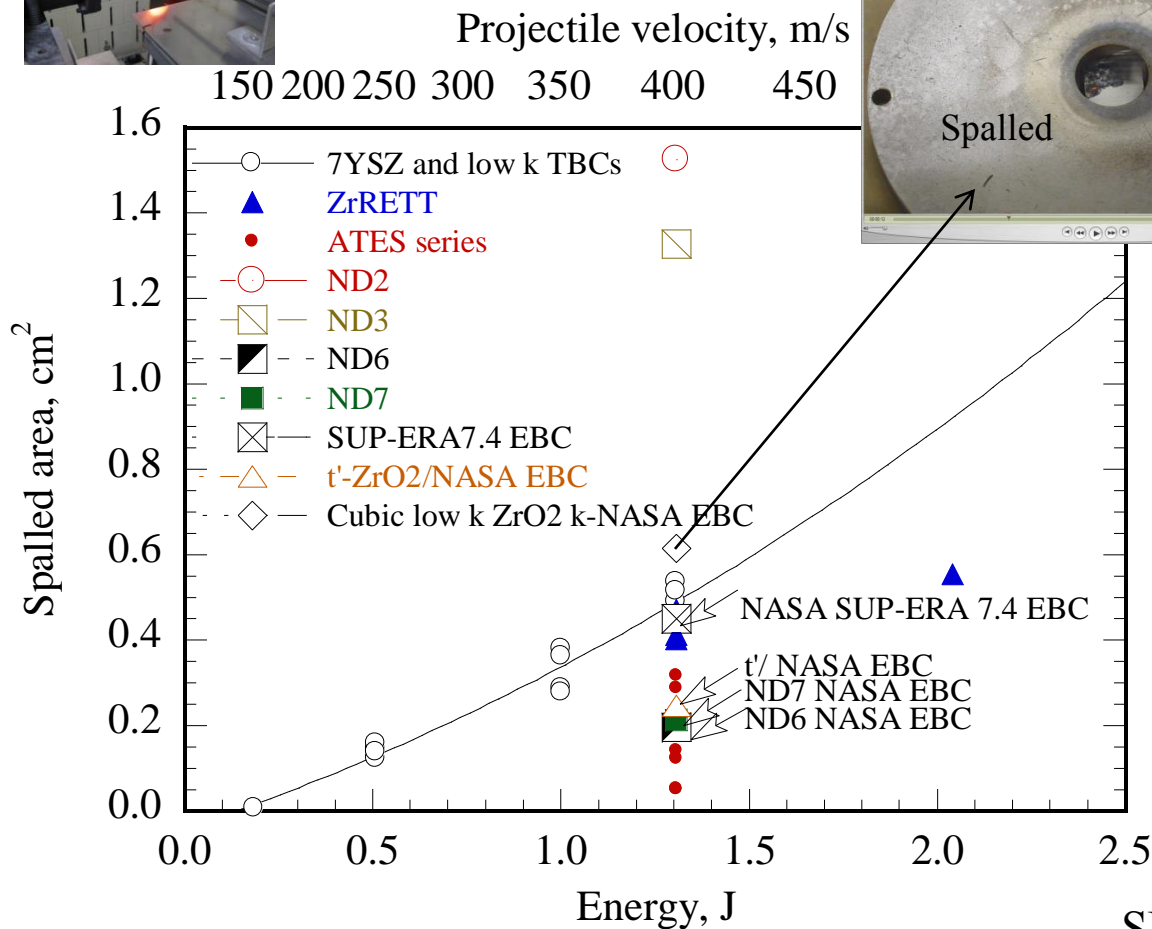


High pressure burner rig tested new ND series Hybrid EBC systems coated on 2" diameter Gen II Prepreg SiC/SiC CMCs



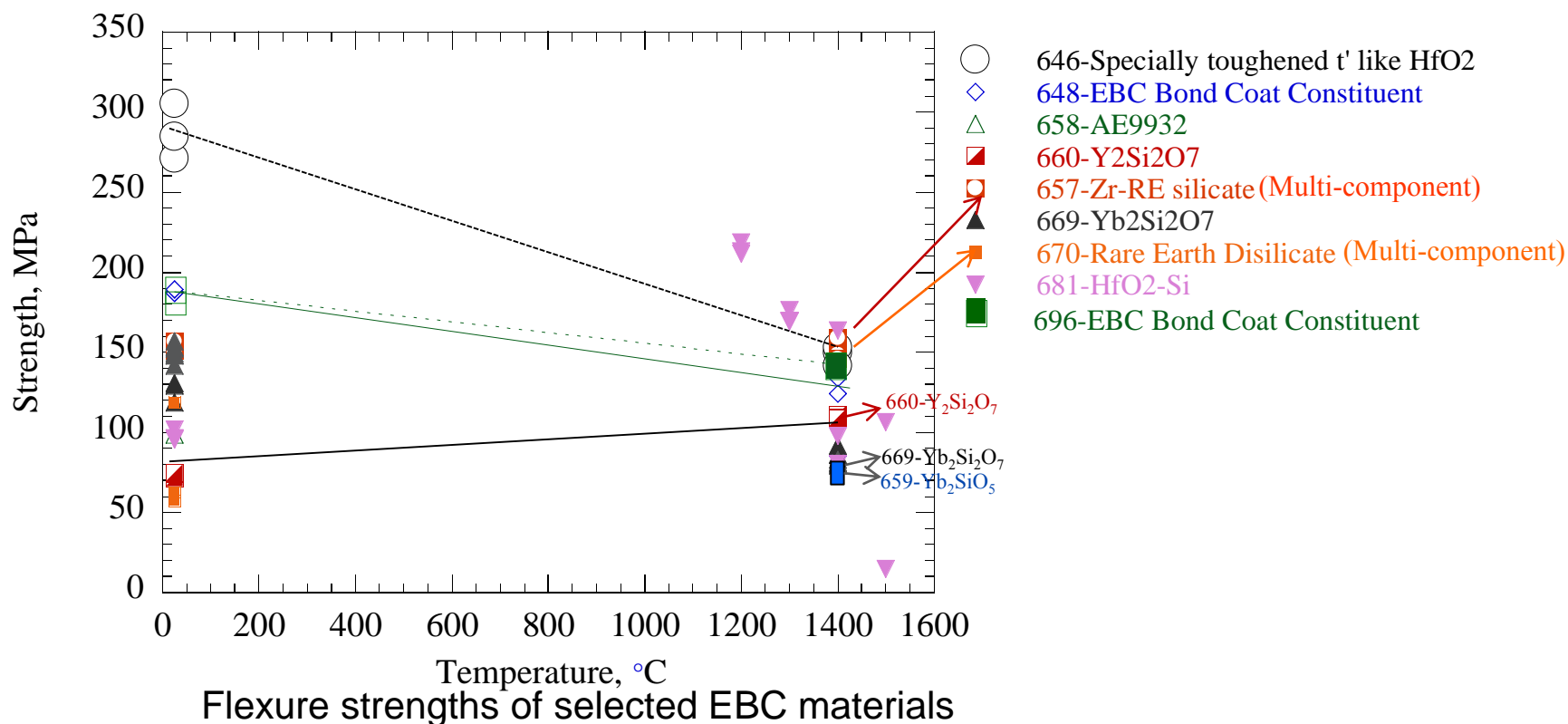
Erosion and Impact Aspects: Early Mach 0.3 Ballistic Impact Tests of $\text{HfO}_2\text{-Si}$ Bond Coat EBC Systems

- Advanced high toughness EBCs tested with comparable performance of best TBCs
- More advanced EBC compositions currently also in developments



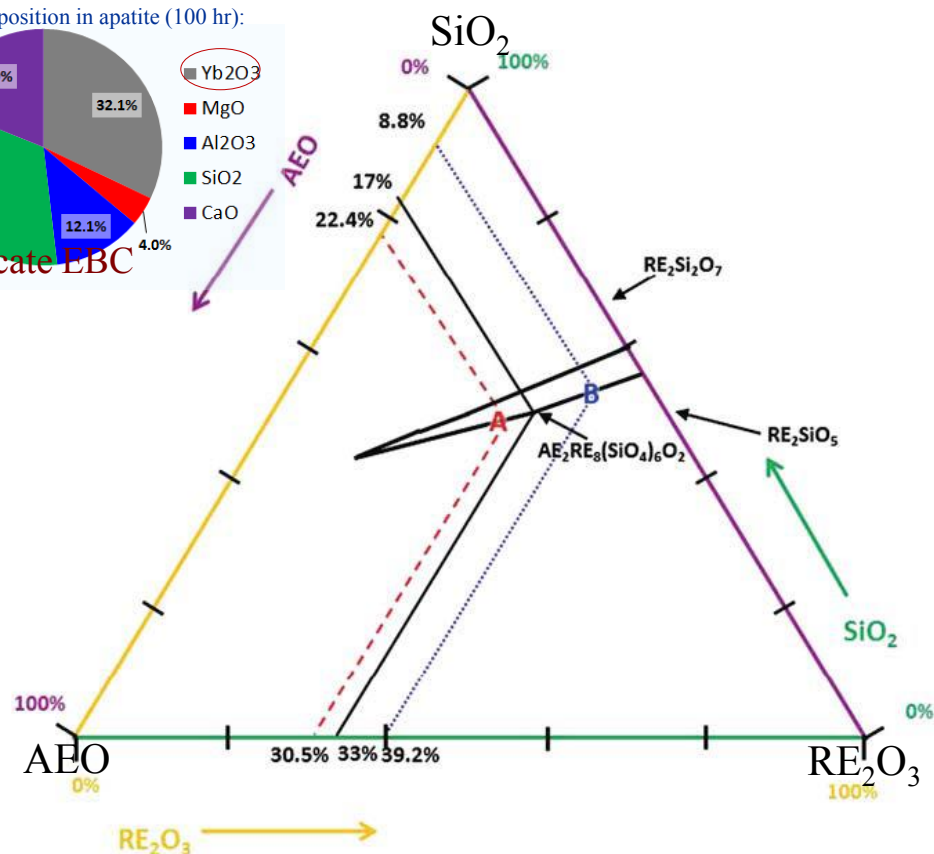
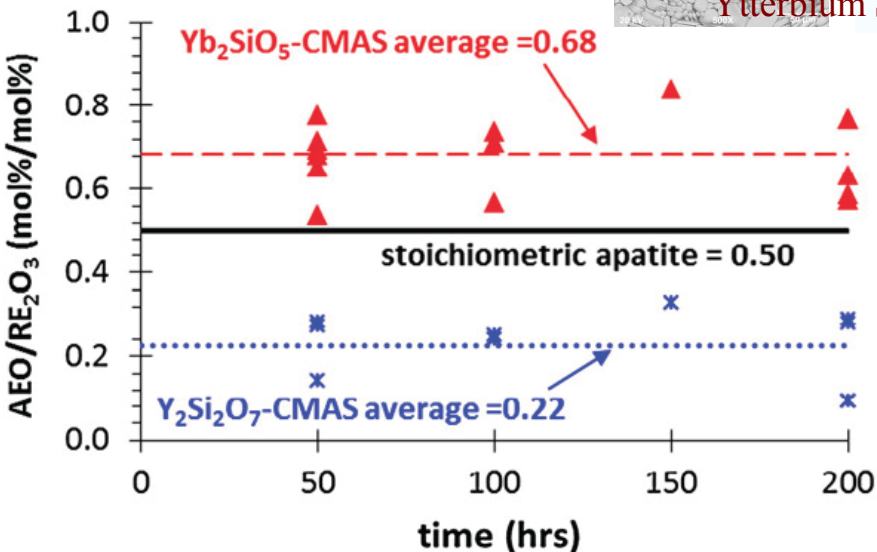
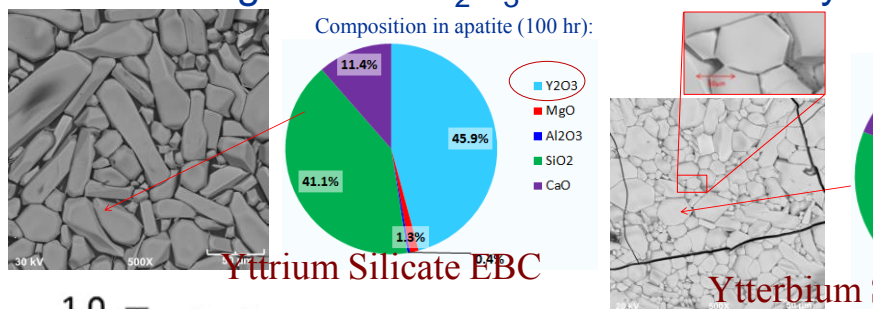
Advanced EBC Coating Material Strength Evaluations

- High strength EBCs and bond coats are critical for prime-reliant designs
 - Multicomponent EBCs and first-generation $\text{HfO}_2\text{-Si}$ bond coat achieved 150-200 MPa strength at high temperature ($1400^\circ\text{C}+$)
 - Multicomponent silicates showed improved high temperature strengths compared to baseline yttrium and ytterbium silicates
 - High strength and high toughness are critical for erosion and fatigue cracking resistance



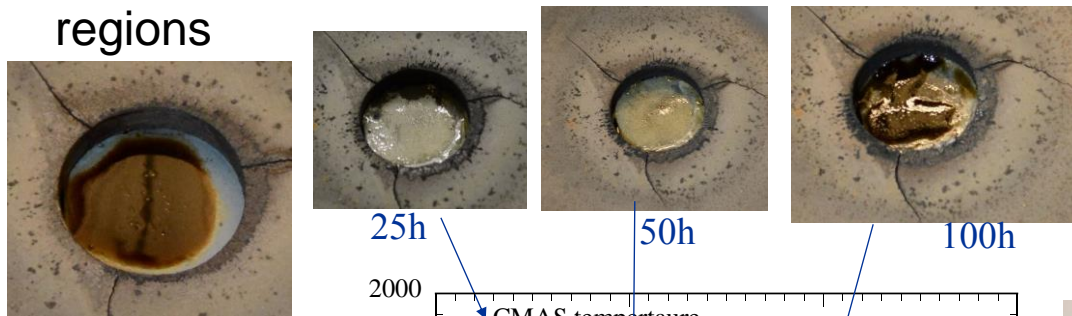
High Heat Flux and CMAS Resistance are Ensured by Advanced High Melting Point Coating, and Multi-Component Compositions

- Non stoichiometric characteristics of the CMAS – rare earth silicate reacted apatite phases – up to 200 h testing
- Difference in partitioning of ytterbium vs. yttrium in apatite
 - Average AEO/RE₂O₃ ratio ~ 0.68 for ytterbium silicate – CMAS system
 - Average AEO/RE₂O₃ ratio ~ 0.22 for yttrium silicate – CMAS system

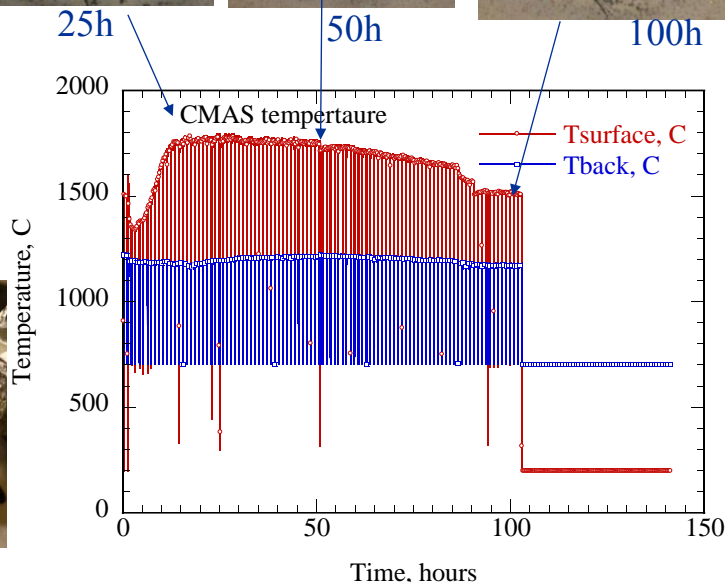


High Heat Flux and CMAS Resistance Tests of Advanced EBC Systems

- Multicomponent rare earth silicate EBC showed improved performance
- Silicate based coatings still sensitive to CMAS concentrations (estimated variation between average CMAS at 25 mg/cm² to more concentrated region CMAS at 75 mg/cm²)
- Some coating damage occurred for EBCs in JETS tests at higher CMAS loading regions



Initially infiltrated



4450 Cycles

Turbine vane EBC, CMAS JETS tests (100 h)

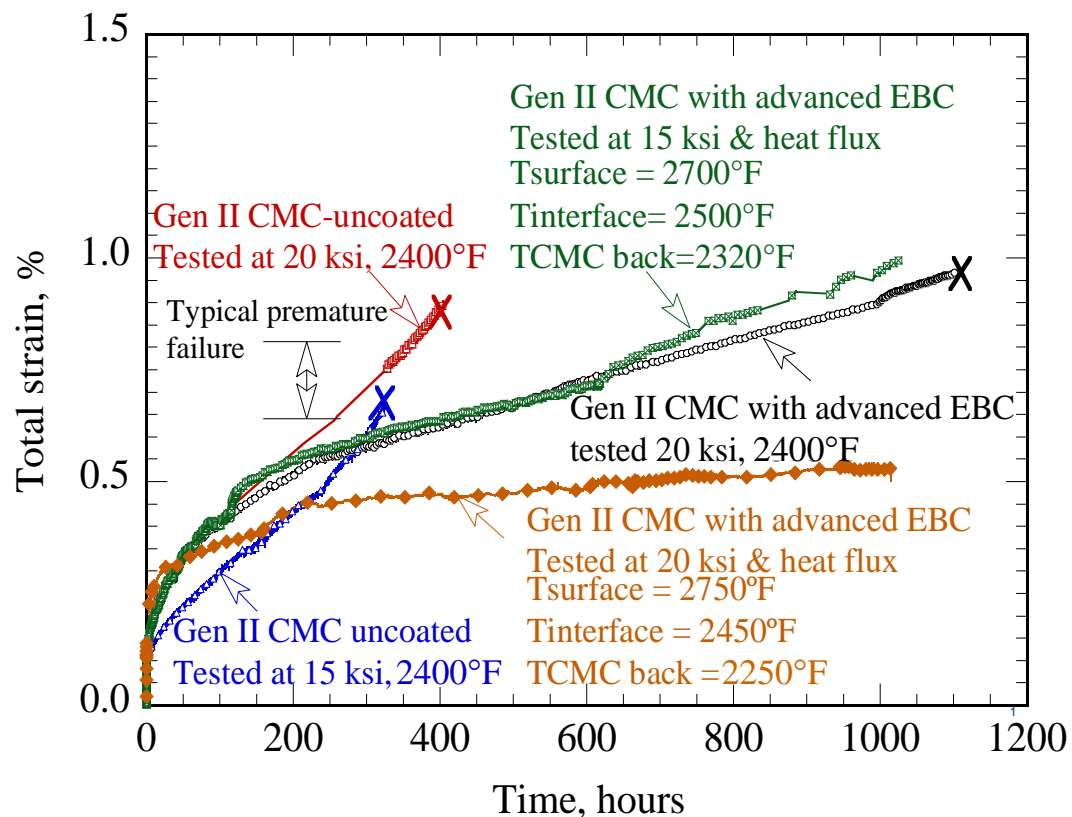
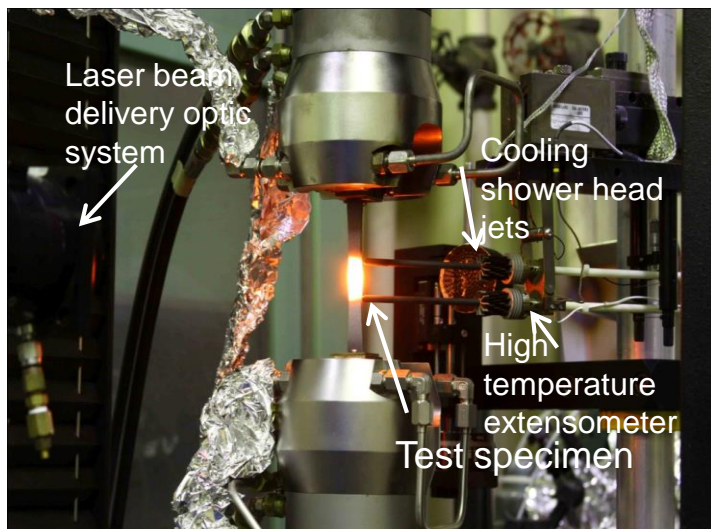
Laser rig test of advanced coating systems

Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine 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 creep, fatigue and environmental interaction is being emphasized

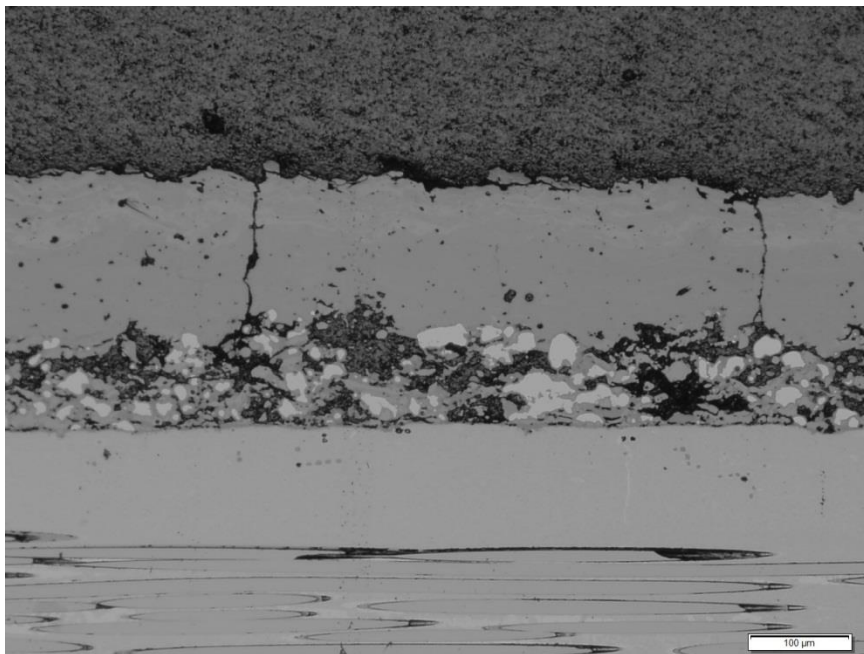


EBC coated tensile specimen

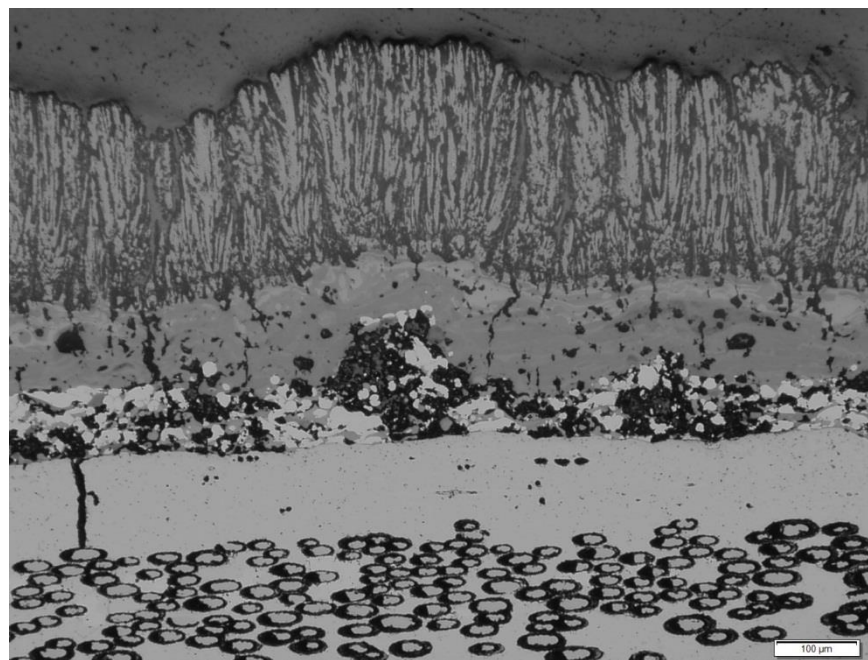


Thermal Gradient Fatigue-Creep Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued

- Advanced environmental barrier coatings – Prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 ksi at $T_{\text{EBC}} 2700^{\circ}\text{F}$, $T_{\text{CMC interface}} \sim 2500^{\circ}\text{F}$
- The HfO_2 -Si based bond coat showed excellent durability in the long term creep tests



EBCs on Gen II CMC after 1000 h fatigue testing



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

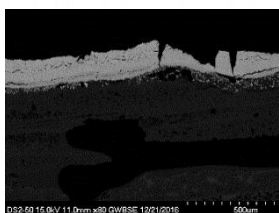
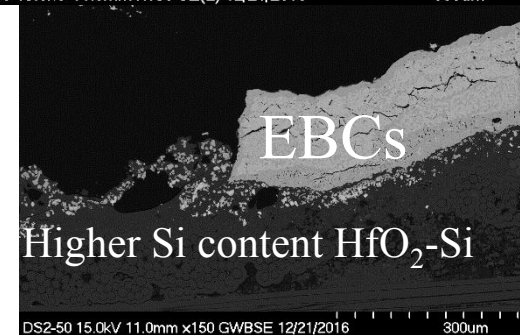
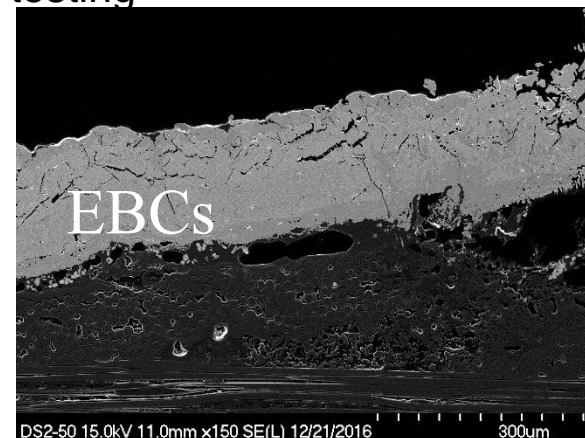
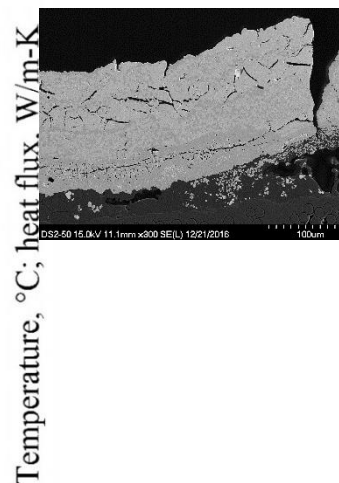
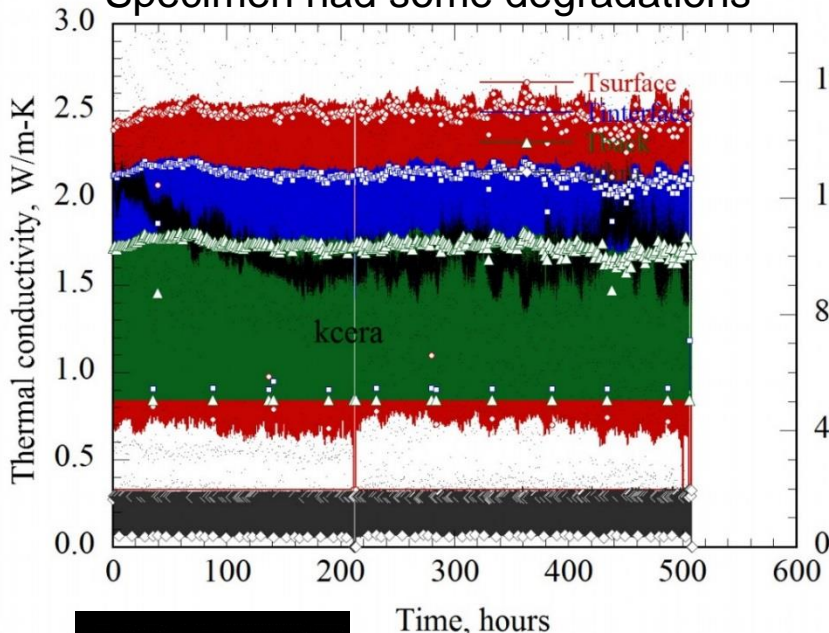
High Heat Flux Thermomechanical fatigue Tests of Advanced NASA EBC-Bond Coats Systems on CMCs

- Laser High Heat Flux thermomechanical fatigue testing of a HfO₂-Si and NASA advanced EBC baseline with steam at 3 Hz, 2600-2700°F, and 69 MPa maximum stress with stress ratio 0.05, completed 500 h testing



- T_{surface} = 1500-1600°C
- T = 1320-1350°C
- Heat Flux = 170 W/cm²
- Specimen had some degradations

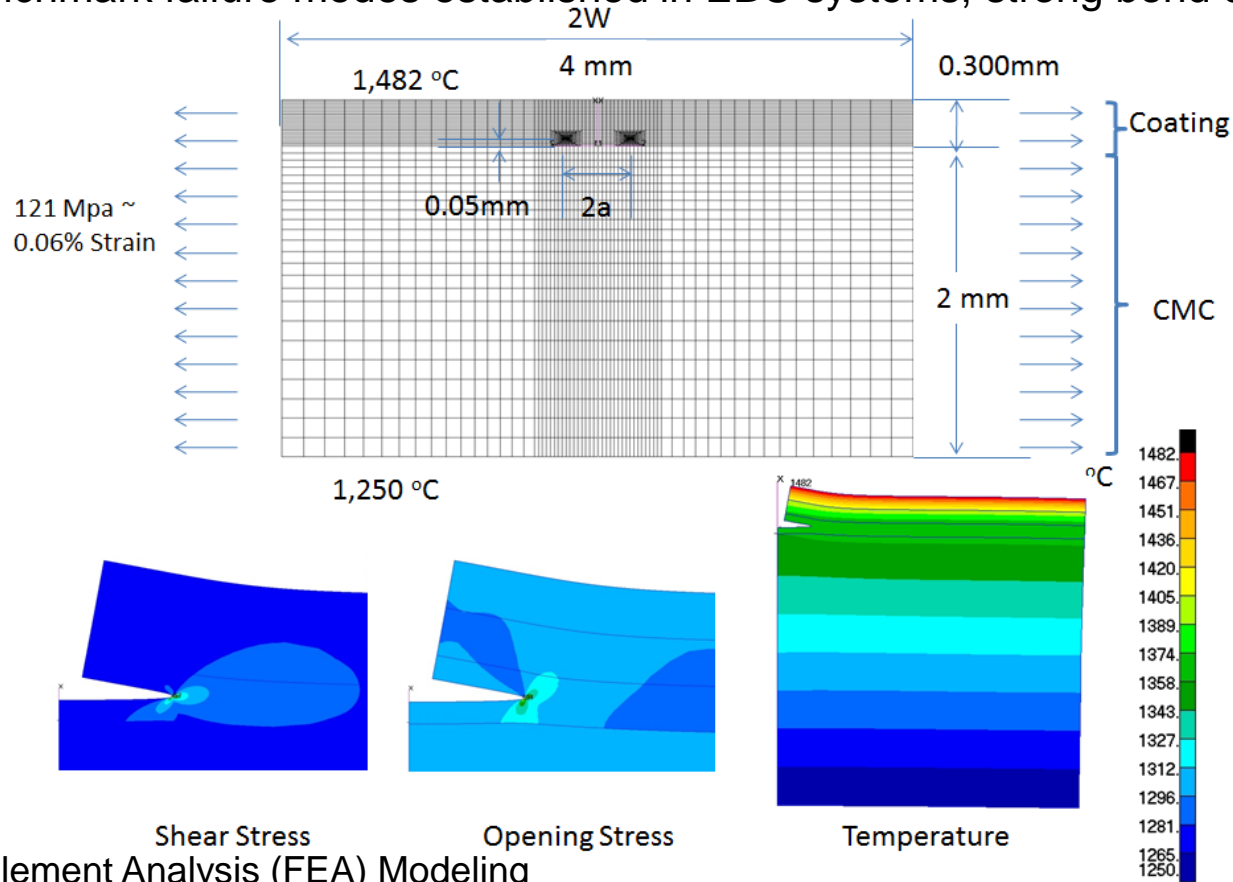
3 hz fatigue testing at 10 ksi loading
Completed 500 hr testing



Testing proving vital failure mechanisms in a simulated test environments

EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling

- An equivalent stress model is established for EBC multicroack stress intensity modeling: emphasize creep, thermal gradient and stress rupture interactions
- Benchmark failure modes established in EBC systems, strong bond coat beneficial

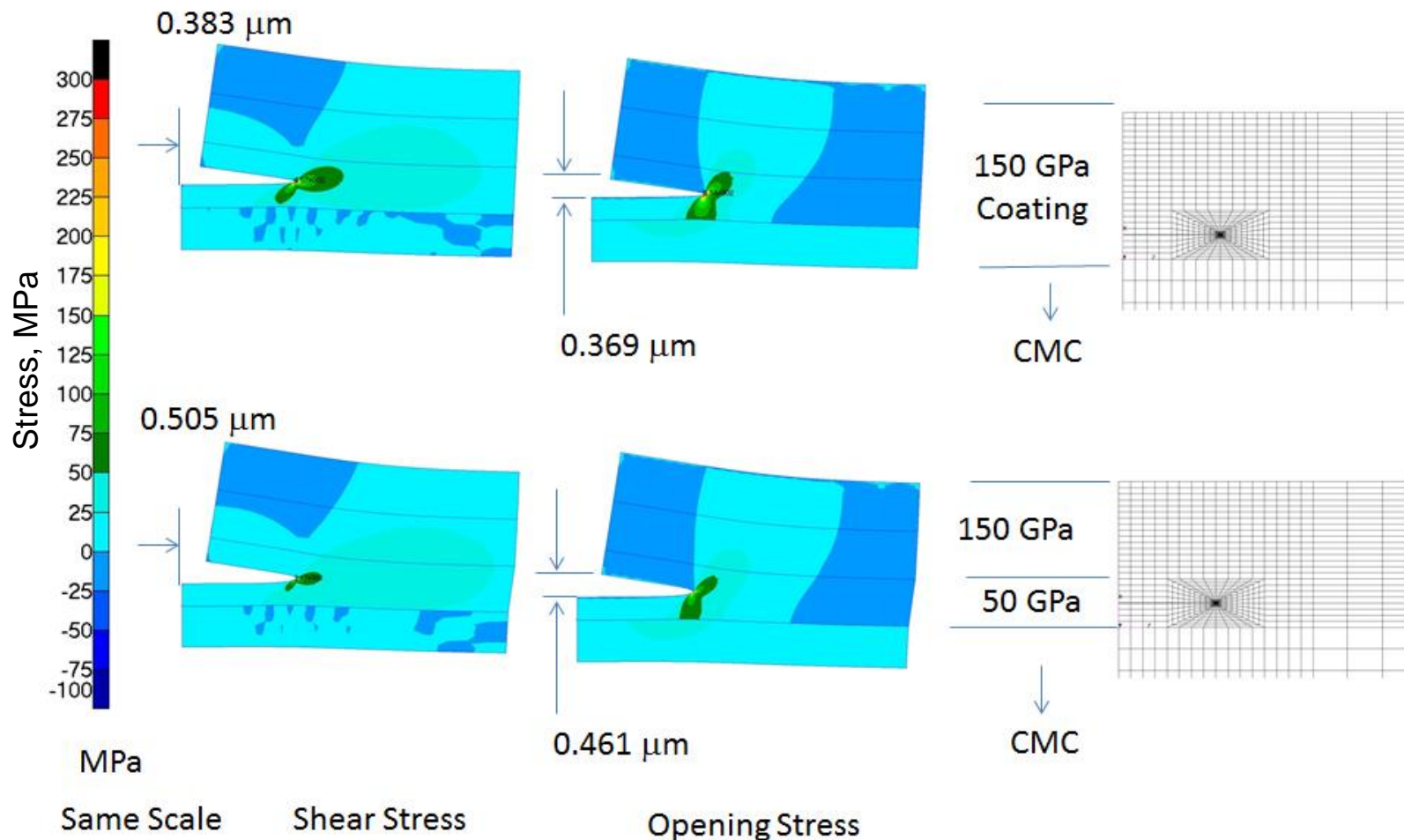


Finite Element Analysis (FEA) Modeling

D. Zhu and L. Ghosn, "Creep, Fatigue and Fracture Behavior of Environmental Barrier Coating and SiC-SiC Ceramic Matrix Composite Systems: The Role of Environment Effects", in *The 11th International Conference on Ceramic Materials and Components for Energy and Environmental Applications*, Vancouver, British Columbia, Canada, June 15-19, 2015.

EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling – Bond Coat Stiffness Effect

- Advanced EBCs designed with higher strength and stiffness to improve creep, fatigue, and cyclic durability



D. Zhu and L. Ghosn, "The Development of Environmental Barrier Coating Systems for SiC-SiC Ceramic Matrix Composites: Environment Effects on the Creep and Fatigue Resistance", in *Aerospace Coatings Conference & Exposition 2014: Development and Manufacturing Trend for the 21st Century*, Hartford, CT, USA, October 8, 2014

Fatigue Tests of Advanced RESi Bond Coats and EBC Systems

- APS and PVD processed 2700°F bond coats on CMC: focus on fatigue testing at temperatures 2400-2700°F
- EBC bond coats critical to prime-reliant coating system designs

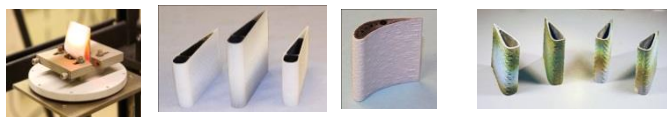
Creep and Fatigue Test with CMAS



Air Plasma Sprayed APS $YSi+Hf-RESi$ Silicate
EBC Bond Coat series on Royce Royce HTC
CVI-MI SiC/SiC (with CMAS)
1400°C, at 10 ksi, **400 h**



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)



Advanced EBC coated airfoil tests

Fatigue Tested

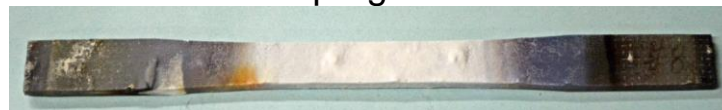


PVD GdYSi(O) coated on Hyper Them 12C-461-
002_#17

1316°C, 10ksi, **1000 h** fatigue (3 Hz, R=0.05)



PVD GdYbSi(O) bond coat, 1316°C, 15ksi,
1169 h fatigue (3 Hz, R=0.05) on GE
Prepreg SiC/SiC



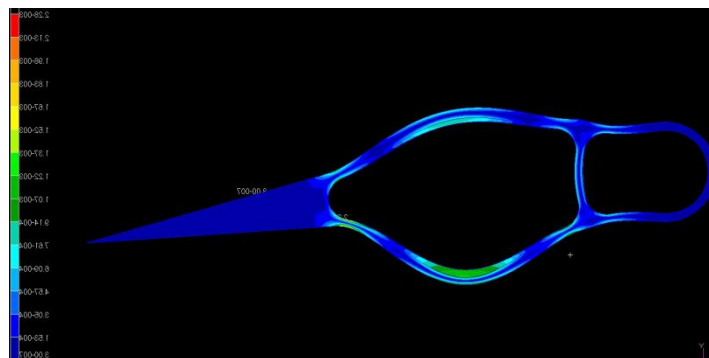
EB-PVD $RE_2Si_{2-x}O_{7-x}$ EBC/HfO₂-Si bond coat on 3D
CVI+PIP SiC/SiC 1482°C, 10ksi, SPLCF fatigue at 3
Hz, R=0.5 (**300 h** furnace tested, **500 h** in laser
thermal gradient)

EBC-CMC Turbine Element Fatigue Testing

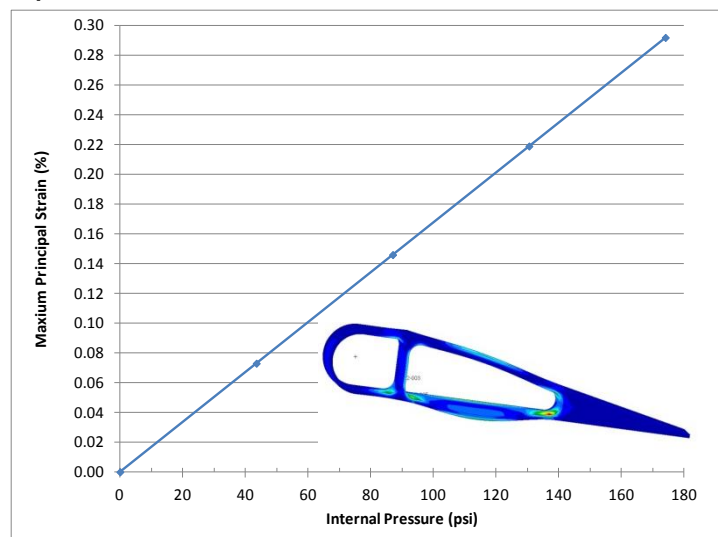
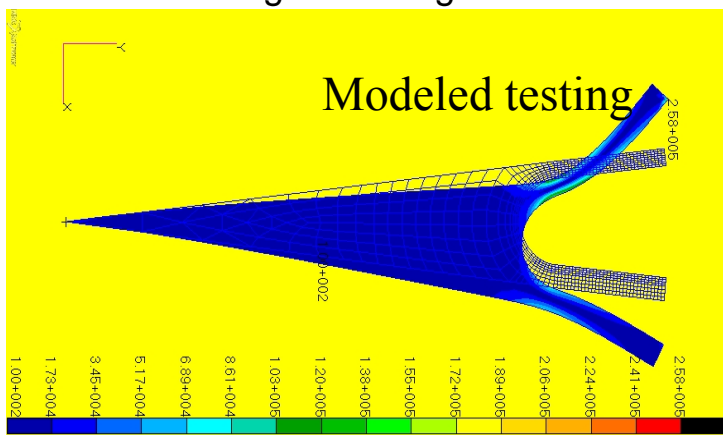
- Testing approaches developed for EBC-CMC trailing edge thermomechanical testing
- High heat flux capability to simulate required high thermal gradients and more complex temperature distributions in a turbine engine
- Mechanical loading to simulate the high pressure turbine airfoil pressure (ballooning) effects
- EBC-CMC durability being evaluated, planned incorporation of stream jet environments



EBC coated Trailing Edge (TE)
“wedge” testing in high heat flux and
mechanical fatigue loading

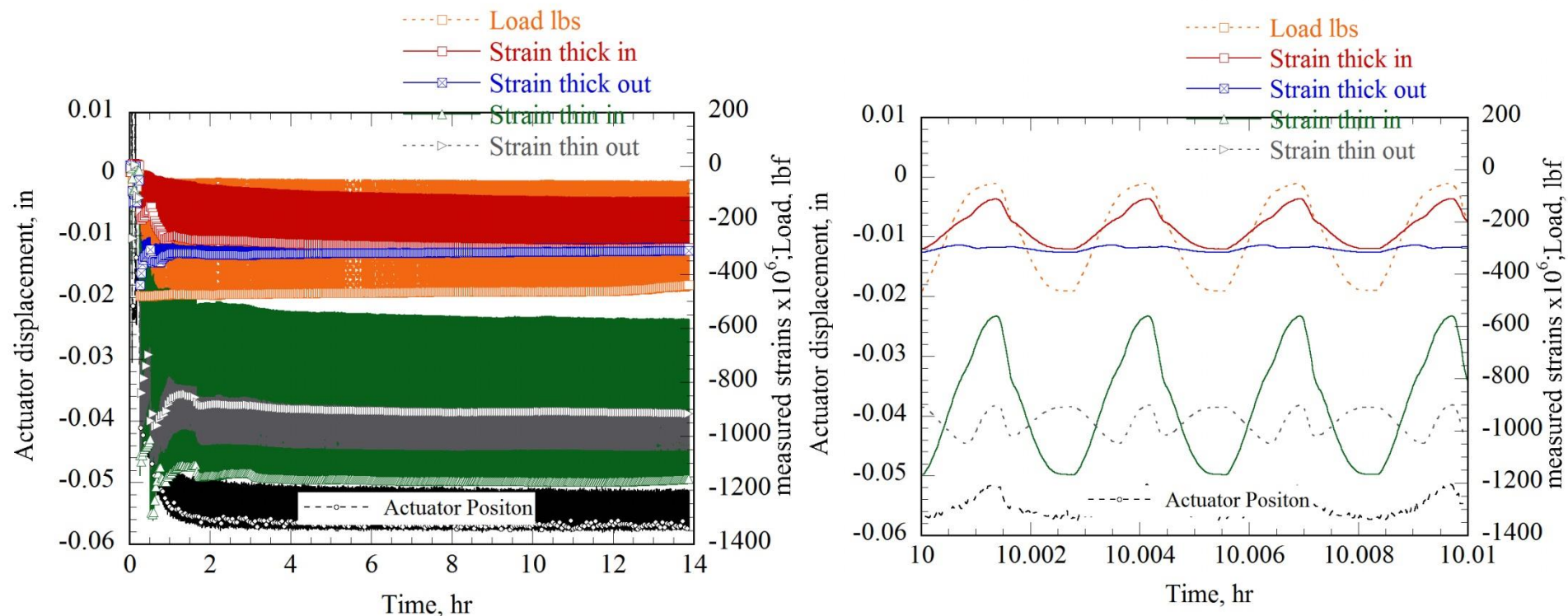


Maximum Principal Strain vs. Airfoil Internal Pressure



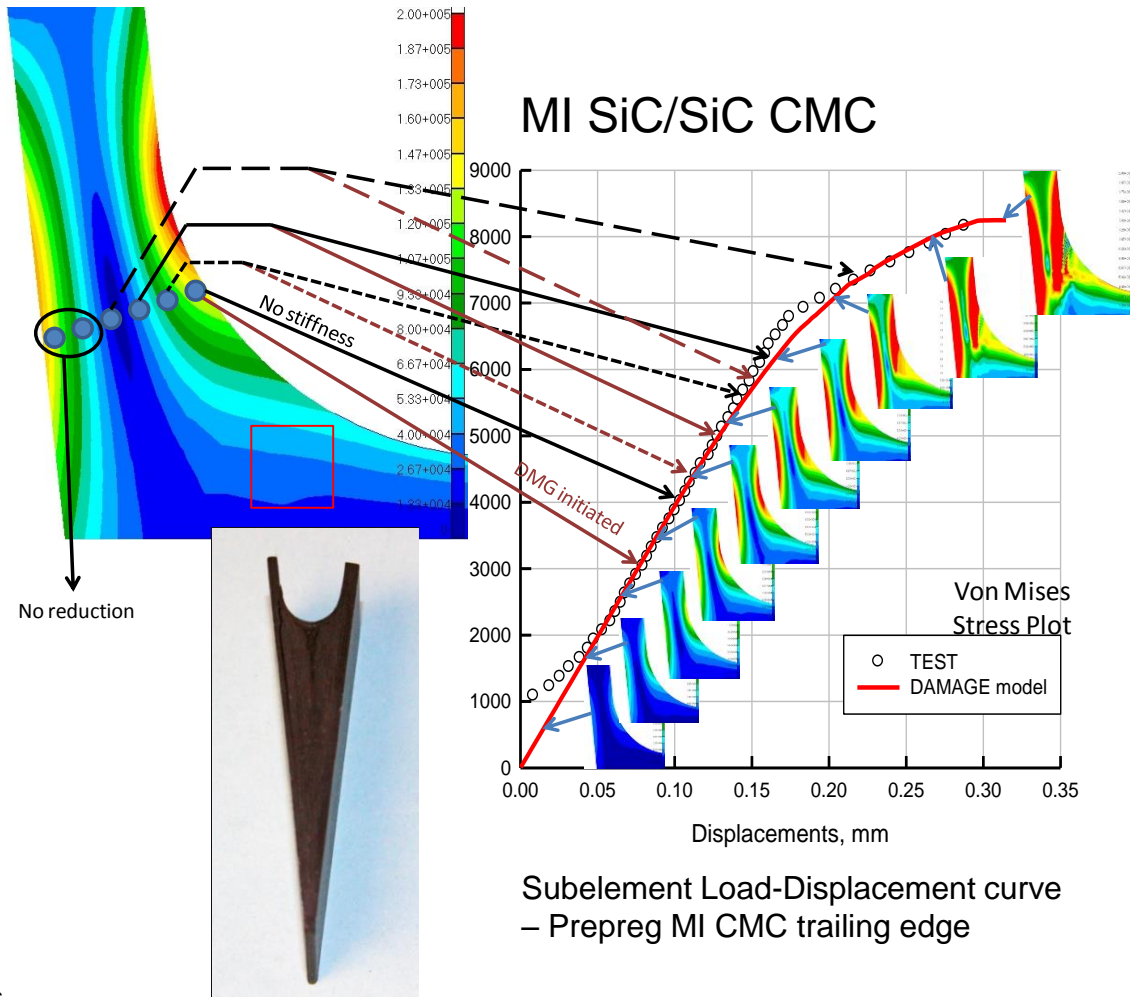
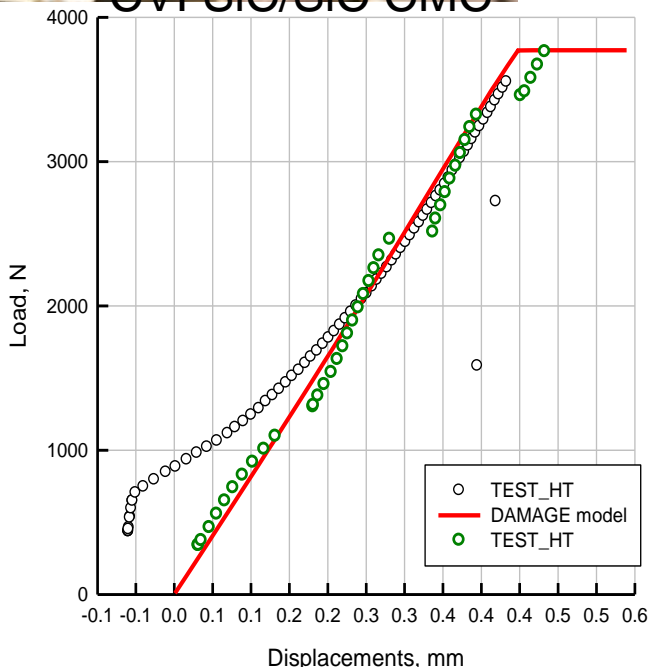
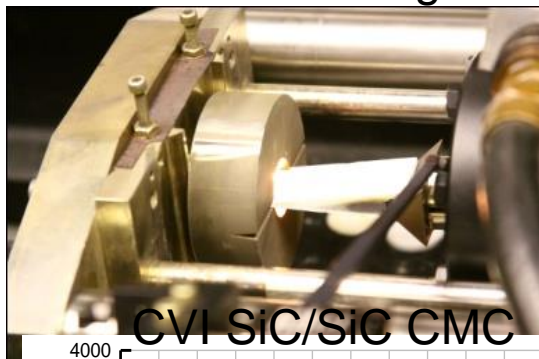
Strains Measurements for Coated Airfoil Trailing Edge Subelements at High Loads

- The results showed complex coating cycling behavior, and out of phase strain cycles also on the EBC coated sides
- Possibly changed neutral axes of the deflections of the CMC thin and thick walls
- Challenges for modeling along with thermal in-phase and out-of-phase loading



SiC/SiC Turbine Airfoil Trailing Edge Tests

- Subelement wedge testing and high temperature tests, aiming at understanding the CMC and EBC degradations



Subelement Load-Displacement curve – Prepreg MI CMC trailing edge

Subelement Load-Displacement curve – CVI CMC trailing edge



Summary

- **Prime-Reliant and durable EBCs are critical to emerging SiC/SiC CMC Hot-Section component technologies**
 - The EBC development built on a solid foundation from past experience, evolved with the current state of the art compositions with higher temperature capabilities and stabilities
 - Multicomponent EBC oxide-doped silicates showed promise with improved stabilities, strength and toughness, and durability in various tests
 - HfO₂-Si and RE-Si bond coats, along with RESiHfCN potentially for realizing prime-reliant EBC-designs
 - Advanced testing help scale-up for components and EVC-CMC modeling

Current emphases and future paths:

- Better understanding of the coating failure mechanisms, and helping develop coating property databases and validate life models, aiming at more robust EBC-CMC designs
- Continue to focus on coating composition and processing improvements, simulated engine environment testing and performance modeling
- Design high strength, strain tolerant, CMAS resistant top coat; and dense, low diffusion and high toughness EBC and bond coats
- Self-repairing and/or self-growing of slow growth adherent EBC coatings, minimizing silica separation



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