Environmental Barrier Coating Development for SiC/SiC Ceramic Matrix Composites: Recent Advances and Future Directions

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   Praxair Surface Technologies – John Anderson and Li Li
   Southwest Research Institute – Ronghua Wei (PVD coating processing)
in supporting the coating processing

Engine OEM Companies including GE Aviation, Rolls Royce (Kang Lee), Honeywell, Pratt & Whitney
Durable Environmental Barrier Coating Systems for Ceramic Matrix Composites (CMCs):
Enabling Technology for Next Generation 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
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
  - Meet 1000 h for subsonic aircraft and 9,000 h for supersonics/high speed aircraft hot-time life requirements

Temperature Capability

- 2800°F combusted TBC
- 2500°F Turbine TBC

Increase in $\Delta T$ across T/EBC

- 2700°F (1482°C) Gen III SiC/SiC CMCs
- 2400°F (1316°C) Gen I and Gen II SiC/SiC CMCs
- 2000°F (1093°C)

* Recession: <5 mg/cm² per 1000 hr (40-50 atm., Mach 1~2)
** Component strength and toughness requirements
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/SiO$_2$ 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
  - Economical
Outline

— Environmental barrier coating system development: challenges and limitations
  • Thermomechanical, environment and thermochemical stability issues
  • Prime-reliant EBCs for CMCs, a turbine engine design requirement

— Advanced environmental barrier coating systems (EBCs) for CMC airfoils and combustors
  • NASA EBC systems and material system evolutions
  • Current turbine and combustor EBC coating emphases
  • Advanced EBC development: processing, testing and durability

— Design tool and life prediction perspectives of coated CMC components

— Advanced CMC-EBC performance demonstrations
  • Fatigue – Combustion and CMAS environment durability
  • Component demonstrations

— Summary and future directions
Fundamental Recession Issues of CMCs and EBCs

- **Recession of Si-based Ceramics**
  (a) Convective; (b) Convective with film-cooling
  - Low SiO₂ activity EBC system development emphasis

- **Advanced rig testing and modeling**
  More complex recession behavior of CMC and EBCs in High Pressure Burner Rig

\[
\text{Recession rate} = \text{const. } V^{1/2} \frac{P_{(H_2O)}^2}{(P_{\text{total}})^{1/2}}
\]

\[
\text{SiO}_2 + 2\text{H}_2\text{O}(g) = \text{Si(OH)}_4(g)
\]

(a)

(b)
Fundamental Recession Issues of CMCs and EBCs - Continued

Weight Loss of SiC in High Pressure Burner Rig
6 atm 20 m/s

![Graph showing weight loss of SiC](image1)

- Early generations of environmental barrier coatings - EBC systems

![Images of coating structures](image2)

Robinson and Smialek, J. Am. Ceram Soc. 1999
Environmental Stability of Selected Environmental Barrier Coatings Demonstrated in NASA High Pressure Burner Rig

- EBC stability evaluated on SiC/SiC CMCs in high velocity, high pressure burner rig environment
- More stable turbine coatings developed under NASA programs
- HfO$_2$-Rare Earth (RE) silicate-based coatings showed significantly improved stability and durability

![Graph showing stability and temperature capability improvements through coating composition and architecture innovations]
EBC Bond Coat: Degradation Mechanisms for Current State of the Art Silicon Bond Coat

- Silicon bond coat melts at 1410°C (melting point)
- Fast oxidation rates (forming SiO₂) and high volatility at high temperature
- Low toughness at room temperature (0.8-0.9 MPa m^{1/2}; Brittle to Ductile Transition Temperature about 750°C)
- Low strength and high creep rates at high temperatures, leading to coating delamination
- Interface reactions leading to low melting phases
  - A significant issue when sand deposit Calcium- Magnesium – Alumino-Silicate (CMAS) is present
- Si and SiO₂ volatility at high temperature (with and without moisture)
Degradation Mechanisms for Si Bond Coat – Interface reactions

- Significant interfacial pores and eutectic phases formation due to the water vapor attack and Si diffusion at 1300°C
- Heat flux condition further limit the use temperatures

SEM images Interface reactions at 1300°C; total 200 hot hours

Si bond coat after 1350°C, 50 hr furnace test in air; 1” dia plasma sprayed EBC button specimen

Hot pressed BSAS+Si button specimen after 1350°C, 50 hr furnace test in air

Interface Si bond coat melting of selected coating systems, under laser heat flux tests, 1” dia button specimen

BaO-Al₂O₃-SiO₂ ternary phase diagram
Degradation Mechanisms for Si Bond Coat – Interface reactions

- Continued

- Significant interfacial pores and eutectic phases formation due to the water vapor attack and Si diffusion at 1300°C

- Heat flux condition further limits the use temperatures

![Graph showing cycles to failure versus interface temperature](image)

![Image of delamination of EBC under heat flux test](image)

Two layer ytterbium mono- and di-silicates

Biaxial heat flux tested ytterbium silicate / Si EBC: surface cracking and interface reaction

Delamination of EBC under heat flux test
Advanced EBC Developments

• Fundamental studies of environmental barrier coating materials and coating systems, stability, temperature limits and failure mechanisms

• Focus on high performance and improving technology readiness levels (TRL), high stability HfO$_2$ and ZrO$_2$ -RE$_2$O$_3$-SiO$_2$/RE$_2$Si$_{2-x}$O$_{7-2x}$ environmental barrier systems
  • More advanced composition and composite EBC systems focusing temperature capability, strength and toughness

• Advanced HfO$_2$-Si and Rare Earth-Silicon based EBC bond coat systems
  • Develop HfO$_2$-Si based + X (dopants) and more advanced bond coat systems for 1482°F (2700°F)+ long term applications
  • Develop prime-reliant Rare Earth (RE)-Si systems for advanced integrated EBC-bond coat systems, improving bond coat temperature capability and reducing silicon/silica-rich phase separations

• Processing optimization for improved composition control and process robustness
Major development milestones:

- 1995-2000: BSAS/Mullite+BSAS/Si

- 2000-2004: $\text{RE}_2\text{Si}_2\text{O}_7$ or $\text{RE}_2\text{SiO}_5$/BSAS+Mullite/Si

- 2000-2004 - 3000°F EBC systems: HfO$_2$ systems (*HfO$_2$ version four-component low k – no silicon containing*) / $\text{RE}_2\text{Si}_2\text{O}_7$ or $\text{RE}_2\text{SiO}_5$ / BSAS+Mullite/Si and Oxide+Si bond coats; component demonstrations
  - Modified mullite (with transition metal and RE dopants) to replace BSAS+mullite
  - Many compound oxide top coat materials explored

- 2005-2011 - Turbine coating systems: Multi-component, graded HfO$_2$-Rare Earth Oxide-SiO$_2$/ multi-component Rare earth Silicate/ HfO$_2$-Si systems
  - RE-HfO$_2$-X/Multicomponent RE-silicate / HfO$_2$-Si +X (doped)

- 2009-present: Improved EBC compositions; RE-Si bond coats
  - e.g., (Gd,Yb,Y)Si bond coat and top coat
# Evolution of NASA EBC Technology for SiC/SiC Ceramic Matrix Composites: Current State of the Art

<table>
<thead>
<tr>
<th>Engine Components:</th>
<th>Combustor</th>
<th>Combustor/ (Vane)</th>
<th>Combustor/ Vane</th>
<th>Vane/ Blade</th>
<th>- Vane/Blade EBCs - Equivalent APS combuster EBCs</th>
<th>Airfoil components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top Coat:</td>
<td>BSAS (APS)</td>
<td>RE$_2$Si$_2$O$_7$ or RE$_2$SiO$_5$ (APS)</td>
<td>- (Hf, Yb, Gd, Y)$_2$O$_3$ - ZrO$_2$/HfO$_2$+RE silicates - ZrO$_2$/HfO$_2$+BSAS (APS and EB-PVD)</td>
<td>RE-HfO$_2$-Alumino silicate (APS and/or 100% EB-PVD)</td>
<td>RE-HfO$_2$-X advanced top coat RE-HfO$_2$-graded Silica (EB-PVD)</td>
<td>Advanced EBC</td>
</tr>
<tr>
<td>Interlayer:</td>
<td>--</td>
<td>--</td>
<td>RE-HfO$_2$/ZrO$_2$- aluminosilicate layered systems</td>
<td>Nanocomposite graded oxide/silicate</td>
<td>Gen IV interlayer not required (optional)</td>
<td></td>
</tr>
<tr>
<td>EBC:</td>
<td>Mullite+ BSAS</td>
<td>BSAS+Mullite</td>
<td>RE silicates or RE-Hf mullite</td>
<td>RE doped mullite-HfO$_2$ or RE silicates</td>
<td>Multi-component RE silicate systems</td>
<td>Multicomponent RE-silicate /self grown</td>
</tr>
<tr>
<td>Bond Coat:</td>
<td>Si</td>
<td>Si</td>
<td>Oxide+Si bond coat</td>
<td>HfO$_2$-Si-X, doped mullite/Si SiC nanotube</td>
<td>Optimized Gen IV HfO$_2$-Si-X bond coat 2700°F bond coats</td>
<td>RE-Si+X systems</td>
</tr>
<tr>
<td>Thickness</td>
<td>10-15 mil</td>
<td>10-15 mil</td>
<td>15-20 mil</td>
<td>10 mil</td>
<td>5 mil</td>
<td>1 - 3 mils</td>
</tr>
<tr>
<td>Surface T:</td>
<td>Up to 2400°F</td>
<td>2400°F</td>
<td>3000°F/2400CMC</td>
<td>2700°F/2400F CMC</td>
<td>3000°F</td>
<td></td>
</tr>
<tr>
<td>Bond Coat T:</td>
<td>Limited to 2462°F</td>
<td>Limit to 2462°F</td>
<td>Limit to 2642°F</td>
<td>Proven at 2600°F +; Advancements targeting 2700°F</td>
<td>2700°F (2011 Goal)</td>
<td></td>
</tr>
</tbody>
</table>

**Challenges overcome by advancements:**
- Improved temperature capability, sintering phase stability, recession resistance, and high temperature strength
- Advanced compositions & processing for combined thermomechanical loading and environments, higher stability and increased toughness towards prime-reliant
NASA EBC Processing Developments for SiC/SiC Ceramic Matrix Composites: Current State of the Art - Continued

- Develop processing capabilities, experience and demonstrate feasibilities in various techniques: air plasma spray, Electron Beam - Physical Vapor Deposition (EB-PVD), Plasma Sprayed-Physical Vapor Deposition (PS-PVD)

- Efforts in developing turbine EBC coatings with Directed Vapor Technologies using Directed Vapor EB-PVD: Turbine Airfoils
- NASA APS, and Triplex Pro APS (with Sulzer/Oerlikon Metco) - for Combustor applications
- Cathodic arc and Magnetron PVD processes: bond coat developments
- NASA PS-PVD
- Some planned EBCs DVM/DVC coatings (with Praxair): aiming at combustor EBC

- Other processing techniques such as Polymer Derived Coating composite coatings (Ceramtec), and laser processing for improved stability
EBC Processing using Plasma Spray and EB-PVD

Oerlikon Metco Triplex Processed Advanced NASA Multilayered EBCs

Directed Vapor EB-PVD Processed Advanced NASA EBCs
Air Plasma Spray Processing of Environmental Barrier Coatings for Combustor Liner Components

— Focused on advanced composition and processing developments using state-of-the-art techniques
— Improved processing envelopes using high power and higher velocity, graded systems processing for advanced TEBCs and thermal protection systems

Advanced Multilayer EBCs

HfO$_2$-Si bond coat

Sulzer Triplex Pro system having high efficiency and high velocity processing

EBC coated SiC/SiC CMC Inner and Outer Liner components

Inner and outer liner articles

NASA EBC processed by Triplex pro
Development and Processing of Directed Vapor Electron Beam - Physical Vapor Deposition (EB-PVD)

- NASA programs in supporting processing developments and improvements with Directed Vapor Technologies International, Inc.
  - Multicomponent thermal and environmental barrier coating vapor processing developments
  - High toughness turbine coatings
  - Affordable manufacture of environmental barrier coatings for turbine components

Advanced multi-component and multilayer turbine EBC systems

Directed Vapor Processing systems

Processed EBC system
Plasma Sprayed-Physical Vapor Deposition (PS-PVD) Processing of Environmental Barrier Coatings

— NASA PS-PVD and PS-TF coating processing using Sulzer newly developed technology
  
  • High flexibility coating processing – PVD - splat coating processing at lo pressure (at ~1 torr)
  • High velocity vapor, non line-of-sight coating processing for complex-shape components
  • Significant progress made in processing the advanced EBC and bond coats

100 kW power, 1 torr operation pressure

NASA PS-PVD coater system

Processed coating systems
Advanced EBC Coating Material Strength Evaluations

- EBC and bond coat constituents are designed with high strength and high toughness to improve coating durability
  - Advanced EBC 150-200 MPa strength achieved at high temperature
  - Multicomponent silicates showed excellent high temperature properties
  - Toughness 3-4 MPa m$^{1/2}$ also achieved (tested at room temperature)
- HfO$_2$-Si based systems showed promising strength and toughness
- More advanced bond coats showed higher temperature capabilities and improved strength

![Graph showing strength vs. temperature for various EBC bond coat constituents.](graph.png)
Advanced HfO$_2$-Si Bond Coats: Effects of Compositions on Strength and Creep Rates

- The HfO$_2$-Si composite coatings showed high strength, and improved creep resistance at high temperatures
- Increased HfO$_2$-HfSiO$_4$ contents improve high temperature strength and creep resistance

![Graph showing the relationship between Si content and creep rates at 1400°C, 30 MPa.](image1)

![Graph showing the relationship between Si content and strength at 1400°C.](image2)
Developing 3000°F (1650°C) EBCs

- NASA Hybrid 3000°F EBC system (2007 R&D 100 Award)
  
  Highlighted coating material systems:
  - High stability multicomponent HfO₂ Top Coat (Patented Hf-RE-SiO₂ systems)
  - Graded and Layer graded interlayers
  - Advanced HfO₂-Rare Earth-Alumino-Silicate EBC (tetragonal t’ ZrO₂ toughened rare earth silicate EBC)
  - Ceramic HfO₂-Si composite bond coat capable up to 2700°F

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Multicomponent Rare Earth (RE) doped HfO₂
(HfO₂-11Y₂O₃-2.5Gd₂O₃-2.5Yb₂O₃)

**Strain tolerant interlayer**
HfO₂-Rare Earth-Alumino-Silicate EBC

HfO₂-Si or RE modified mullite bond coat
Advanced HfO$_2$-Si+X Bond Coats

- Coating architecture and HfO$_2$ contents can be effectively controlled and optimized
- Low oxygen activity in the HfO$_2$-Si bond coats
Advanced HfO$_2$-Si+X Bond Coats - Continued

- Microstructure of a HfO$_2$-doped (Yb,Y)Si(O) bond coat
NASA Turbine Environmental Barrier Coating Developments – Environmental Testing Validations

- Advanced NASA EBCs tested in coupons under laser heat flux cyclic rigs up 1650°C+
- Coated subelements coating tested up 1500°C under laser thermal gradient for 200 hr
- EBC systems show high stability in High Pressure Burner Rig Tests
- Low thermal conductivity of 1.2 W/m-K for optimized turbine airfoil coatings

High pressure burner rig, 16 atm, 31 hr – no measurable weight loss
NASA EBC Bond Coats for Airfoil and Combustor EBCs

- Advanced systems developed and processed 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

<table>
<thead>
<tr>
<th>YSi</th>
<th>YbGdYSi</th>
<th>GdYSi</th>
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<tbody>
<tr>
<td>ZrSi+Y</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
</tr>
<tr>
<td>ZrSi+Y</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
</tr>
<tr>
<td>ZrSi+Ta</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
</tr>
<tr>
<td>HfSi + Si</td>
<td>YbGdSi</td>
<td>GdYSi-X</td>
</tr>
<tr>
<td>HfSi + YSi</td>
<td>YbGdSi</td>
<td></td>
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<tr>
<td>HfSi+Ysi+Si</td>
<td>YbGdSi</td>
<td></td>
</tr>
<tr>
<td>YbSi</td>
<td>YbGdSi</td>
<td></td>
</tr>
<tr>
<td>HfSi + YbSi</td>
<td>YbSi</td>
<td></td>
</tr>
<tr>
<td>GdYbSi(Hf)</td>
<td>YbSi</td>
<td></td>
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<tr>
<td>YYbGdSi(Hf)</td>
<td>YbYSi</td>
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<tr>
<td>YbYSi</td>
<td></td>
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<tr>
<td>YbHfSi</td>
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<td>YbHfSi</td>
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<td>YbSi</td>
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### EB-PVD

<table>
<thead>
<tr>
<th>HfO2-Si; REHfSi</th>
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<tbody>
<tr>
<td>YSi+RESilicate</td>
</tr>
<tr>
<td>GdYSi</td>
</tr>
<tr>
<td>GdYbSi</td>
</tr>
<tr>
<td>GdYb-LuSi</td>
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<tr>
<td>NdYSi</td>
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### APS*

<table>
<thead>
<tr>
<th>HfO2-Si</th>
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<tbody>
<tr>
<td>YSi+Hf-RESilicate</td>
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<tr>
<td>YSi+Hf</td>
</tr>
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### FurnaceLaser/CVD/PVD

<table>
<thead>
<tr>
<th>REHfSi</th>
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<tr>
<td>Hf-RESilicate</td>
</tr>
<tr>
<td>Used in ERA components as part of bond coat system</td>
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<tr>
<td>Hf-RE-Al-Silicate</td>
</tr>
<tr>
<td>Used also in ERA components</td>
</tr>
<tr>
<td>Used in ERA components as part of bond coat system</td>
</tr>
<tr>
<td>APS*: or plasma spray related processing methods</td>
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</table>
NASA EBC Bond Coats for Airfoil and Combustor EBCs

- 1500°C (2700°F) capable NASA RESi+X (X is dopants) 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 500 h durability.

- RESi-Hf, 100 hr
- RESi+Al, 50 hr
- RESi+Al, 50 hr

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.

Processed Subelement
Rare Earth (RE) Silicides/Silicates and Effect of the HfO$_2$ Dopant

- Dopants improving oxidation resistance, pesting, and SiO$_2$ separation

Undoped system shows separation of Si-rich/silica-rich phase

The Si-rich/silica-rich phases converted to more stable HfO$_2$ rich phases
RE Silicide Based Compositions without Multi-Dopants

- Advanced compositions improve high temperature stability and environmental resistance

\[ \text{YbSi}_x \]

1450-1500°C exposure for 100 hr

Yb silicate phase segregated after the long-term testing

Silica rich phase formed as a grain boundary “binding” phase
Furnace Cycle Test Results of Selected RESi and ZrSi + Dopant Bond Coats
- Testing in Air at 1500°C, 1 hr cycles

- Multi-component systems showed excellent furnace cyclic durability at 1500°C
Advanced Bond Coats for Turbine EBCs – Oxidation Resistance

- 1500°C (2700°F) capable RESiO+X series EBC bond coat compositions and related composite coatings developed for combustor and turbine airfoil applications
- Oxidation kinetics studied using TGA in flowing O₂
- Parabolic or pseudo-parabolic oxidation behavior observed

Oxidation kinetics of a YbGdSi(O) bond coat

An oxidized bond coat after 1500°C 100 h creep testing
Advanced EBC developments – Some Hybrid APS-PVD Systems and Qualification Tests

- EB-PVD HfO$_2$-RE$_2$O$_3$ (Silicate) top coat EBC with plasma-spayed multi-component advanced silicate sublayer EBC/HfO$_2$-Si bond coat systems
- Low thermal conductivity ranging 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

2” diameter ND3 EBC/SiC/SiC specimen after testing in the high pressure burner rig At 2600°F

Some surface spallation

High pressure burner rig tested new ND series Hybrid EBC systems coated on 2” diameter Gen II Prepreg SiC/SiC CMCs
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
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°F (1482°C), $T_{\text{CMC interface}}$ ~2500°F (1371°C)
– The HfO$_2$-Si based bond coat showed excellent durability in the long term creep tests

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

Hybrid EBCs on Gen II CMC after 1000 h low cycle creep fatigue testing
EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling

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

Finite Element Analysis (FEA) Modeling

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
- FEM models showed that a soft bond coat showed larger “spalling” displacements

MPa

0.383 μm

150 GPa Coating

CMC

0.369 μm

0.505 μm

150 GPa

50 GPa

CMC

0.461 μm

High Stability and CMAS Resistance: Improved by Advanced High Melting Point, and Multi-Component Coating Compositions

- Demonstrated CMAS resistance of the NASA RESi System at 1500°C, 100 hr
- Silica-rich phase precipitation in CMAS
- Rare earth element leaching into the melts (low concentration ~9 mol%)
High Stability and CMAS Resistance: Improved by Advanced High Melting Point, and Multi-Component Coating Compositions

- Non stoichiometric characteristics of the CMAS – rare earth silicate reacted apatite phases – up to 200 hr testing
- Difference in partitioning of ytterbium vs. yttrium in the apatite phases
  - Average AEO/RE$_2$O$_3$ ratio $\sim 0.68$ for ytterbium silicate – CMAS system
  - Average AEO/RE$_2$O$_3$ ratio $\sim 0.22$ for yttrium silicate – CMAS system

Fatigue Tests of Advanced RESi Bond Coats and EBC Systems

- APS and PVD processed 2700°F bond coats on CMCs: focus on fatigue testing at the temperature range of 2400 to 2700°F
- Incorporating CMAS and steam environments

Creep Test with CMAS

Fatigue Tested

APS Bond Coat series on CVI-MI SiC/SiC EBC at 1400°C, 10 ksi, 400 hr

PVD GdYSi coated on Hyper Them 12C-461-002_#17
1316°C, 10ksi, 1000 h fatigue (3 Hz, R=0.05)

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

1537°C, 10ksi, 300 h fatigue (3 Hz, R=0.05) on CVI-MI SiC/SiC (with CMAS)
Thermomechanical Fatigue Tests of Validating Advanced RESi Bond Coats and EBC Systems

- Strength and Fatigue cycles in laser heat flux rigs in tension, compression and bending
- Fatigue tests at 3 Hz, 2600-2700°F, stress ratio 0.05, surface tension-tension cycles
- Total fatigue-CMAS durability demonstrated

- Achieved long-term fatigue lives (near 500 hr) with EBC at 2700°F

Creep-fatigue durability test summary

Example of fatigue test EBC systems on Tyrannohex SiC composites
The Advanced EBC on SiC/SiC CMC Turbine Airfoils Successfully Tested for Rig Durability in NASA High Pressure Burner Rig

- NASA advanced EBC coated turbine vane subcomponents tested in rig simulated engine environments (up to 240 m/s gas velocity, 10 atm), reaching TRL of 5
- Turbine EBCs generally intact (some minor partial coating top coat spalling for the coated Prepreg MI SiC/SiC vane)
- Some minor CMC vane degradations after the testing

Uncoated vane tested 15 hr

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
The EBC Coated SiC/SiC CMC Combustor Liner Successfully Demonstrated for Rig Durability in NASA High Pressure Burner Rig (First Inner Liner Processed at Sulzer with Triplex Pro)

- Tested pressures at 500 psi external for outliner, and up to 220 psi inner liners in the combustion chamber (16 atm), accumulated 250 hours in the high pressure burner rig
- Average gas temperatures at 3000°F (1650°C) based on CEA calculations, the liner EBCs tested at 2500°F (1371°C) with heat fluxes 20-35 W/cm², and the CMC liner component at 1800-2100°F (∼1000-1100°C)

![Graph showing Ideal Flame Temperature Calculation - Chemical Equilibrium Analysis Codes (CEA)-II]

- Hot streaks with possible gas temperature over 2000°C, with minimum back cooling
- Swirl jet flows
- Some minor coating spalling at hot streak impingement
Summary

- Durable EBCs are critical to emerging SiC/SiC CMC component technologies
- The NASA EBC development built on a solid foundation from past experience, evolved with the current state of the art compositions of higher temperature capabilities and stabilities
  - Multicomponent EBC oxide/silicates with higher stabilities
  - Improved strength and toughness
  - HfO$_2$-Si and RE-Si bond coats for realizing 1482°C+ (2700°F+) temperature capabilities and potentially prime-reliant EBC-designs
  - New EBC compositions improved combustion steam and CMAS resistance

- EBC processing and testing capabilities significantly improved, allowing more advanced compositions designed, validated and realized for more complex turbine components

- Improved the understanding of coating failure mechanisms, helping developing coating property databases and validated life models, also aiming at more robust EBC-CMC designs and developments

- Emphasized next generation turbine airfoil EBC developments, demonstrated component EBC technologies in simulated engine environments of TRL 5
Future Directions and Opportunities for EBC System Developments

- High melting point, high toughness, low expansion EBC top coat designs with advanced architectures and grain boundary phase designs to achieve exceptional environment stability and performance
- High stability nano-phase composite bond coat designs involving rare earth, hafnium and silicon-containing dopant alloy clusters for improved oxidation resistance and cyclic durability, minimizing silica separation and crystallization, at high temperature and in larger chemical potential gradients
- Self-repairing and/or self-growing of slow growth adherent EBC coatings
- Superior adhesion and intergraded EBC/CMC interfaces with reaction barriers, potentially integrated additive CMC-coating manufacturing
- High efficiency plasma spray, PVD and/or CVD cost effective and robust processing
- High strength and high toughness, combined with optimized strain tolerance for superior erosion and impact resistance
- Multifunctional compositions
  - High strength and high toughness, combined with optimized strain tolerance for superior erosion and impact resistance, self-healing
  - High temperature sensing, health monitoring, and reduced heat transfer