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

Dongming Zhu

Materials and Structures Division
NASA Glenn Research Center
Cleveland, Ohio 44135, USA

40th Jubilee Symposium: Engineered Ceramics — Current Status and Future Prospects
International Conference and Exhibition on Advanced Ceramics and Composites
January 25, 2016
Acknowledgements

- The work was supported by NASA Fundamental Aeronautics Program (FAP) Transformational Tools and Technologies (TTT) Project and NASA Environmentally Responsible Aviation (ERA) Project

In particular, the contributions from the following:
- Ron Phillips (Vantage Partners, LLC) and Ralph Pawlik (University of Toledo), mechanical testing
- Mike Cuy, Furnace Cyclic Oxidation Testing
- Don Humphrey (Vantage Partners, LLC), TGA Testing
- Terry McCue (SAIC/NASA GRC, SEM/EDS)
- Joy Buehler (Vantage Partners, LLC, Met Lab)

NASA EBC-CMC Team, In particular, Jim DiCarlo, Jim Smialek, Dennis Fox, Bryan Harder, Robert A. Miller, Janet Hurst, Martha Jaskowiak, Ram Bhatt, Mike Halbig, Valerie Wiesner, Nate Jacobson, Narottam Bansal, Francisco Sola-Lopez, and Serene Farmer (NASA GRC)

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

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

**Component strength and toughness requirements**

<table>
<thead>
<tr>
<th>Temperature Capability</th>
<th>2800°F comb</th>
<th>2500°F TBC</th>
<th>2700°F (1482°C)</th>
<th>3000°F SiC/SiC CMC airfoil and combustor technologies</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>combustor TBC</td>
<td>TBC</td>
<td></td>
<td>2700°F SiC/SiC thin turbine EBC systems for CMC airfoils</td>
</tr>
</tbody>
</table>

* 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 SiO$_2$ activity EBC system development emphasis
- Advanced rig testing and modeling
  More complex recession behavior of CMC and EBCs in High Pressure Burner Rig

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

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

Combustion gas

Cooling gas
Fundamental Recession Issues of CMCs and EBCs - Continued

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

- Early generations of environmental barrier coatings - EBC systems

Robinson and Smialek, J. Am. Ceram Soc. 1999

- HfO$_2$ based low k - APS
- HfO$_2$ based low k - EB-PVD

Combustor coating
Turbine coating
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.]

- Stability and temperature capability improvements through coating composition and architecture innovations

**NASA EBC development stability goal**

**Gas pressure 6 atm**

**Gas velocity 30m/s**

**Gas velocity 200m/s**

- SiC, 20m/s, 6 atm; Robinson and Smialek, J. Am. Ceram Soc. 1999;
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 mⁱ/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-Siliacte (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

BaO-Al₂O₃-SiO₂ ternary phase diagram

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
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 interface temperature vs. cycles to failure](image1)

Two layer ytterbium mono- and di-silicates

Si

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

![Micrographs of EBC interface](image2)

Delamination of EBC under heat flux test

Mullite

Mullite+BSAS

Si

YSZ

Mullite+

BSAS

Si
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 HfO₂ and ZrO₂ -RE₂O₃-SiO₂/RE₂Si₂-xO₇-2x environmental barrier systems
  • More advanced composition and composite EBC systems focusing temperature capability, strength and toughness

• Advanced HfO₂-Si and Rare Earth-Silicon based EBC bond coat systems
  • Develop HfO₂-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., ($\text{Gd},\text{Yb},\text{Y}$)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>Generation</th>
<th>Period</th>
<th>Engine Components</th>
<th>Top Coat</th>
<th>Interlayer</th>
<th>EBC</th>
<th>Bond Coat</th>
<th>Challenges overcome by advancements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gen I (EPM)</td>
<td>1995-2000 R&amp;D Award</td>
<td>Combustor</td>
<td>BSAS (APS)</td>
<td>--</td>
<td>Mullite + BSAS</td>
<td>Si</td>
<td>Improved temperature capability, sintering, recession resistance, and high temperature strength</td>
</tr>
<tr>
<td>Gen II (UEET)</td>
<td>2000-2004</td>
<td>Combustor/ (Vane)</td>
<td>RE₂Si₂O₇ or RE₂SiO₅ (APS)</td>
<td>--</td>
<td>BSAS + Mullite</td>
<td>Si</td>
<td></td>
</tr>
<tr>
<td>Gen III (UEET)</td>
<td>2000-2005 R&amp;D Award (2007)</td>
<td>Combustor/ Vane</td>
<td>- (Hf,Yb,Gd,Y)₂O₃</td>
<td>RE-HfO₂-ZrO₂/ZrO₂ aluminosilicate layered systems</td>
<td>RE silicates or RE-Hf mullite</td>
<td>Oxide + Si bond coat</td>
<td></td>
</tr>
<tr>
<td>Gen IV (FAP)</td>
<td>2005-2011 R&amp;D Award (2007)</td>
<td>Vane/ Blade</td>
<td>RE-HfO₂-Alumino silicate</td>
<td>Nanocomposite graded oxide/silicate</td>
<td>RE doped mullite-HfO₂ or RE silicates</td>
<td>HfO₂-Si-X, doped mullite/Si SiC nanotube</td>
<td></td>
</tr>
<tr>
<td>Gen V-VI (FAP - ERA)</td>
<td>2007 – 2012 to present</td>
<td>- Vane/Blade EBCs</td>
<td>RE-HfO₂-X advanced top coat</td>
<td>Gen IV interlayer not required (optional)</td>
<td>Multi-component RE silicate systems</td>
<td>Optimized Gen IV HfO₂-Si-X bond coat 2700°F bond coats</td>
<td></td>
</tr>
</tbody>
</table>

**Engine Components:**
- Combustor
- Combustor/ (Vane)
- Combustor/ Vane
- Vane/ Blade

**Top Coat:**
- BSAS (APS) RE₂Si₂O₇ or RE₂SiO₅ (APS)
- RE-HfO₂-Alumino silicate

**Interlayer:**
- RE-HfO₂/ZrO₂ aluminosilicate layered systems

**EBC:**
- Mullite + BSAS
- BSAS + Mullite
- RE silicates or RE-Hf mullite
- RE doped mullite-HfO₂ or RE silicates

**Bond Coat:**
- Si
- Oxide + Si bond coat
- HfO₂-Si-X, doped mullite/Si SiC nanotube

**Surface T:**
- Up to 2400°F
- 2400°F
- 3000°F/2400CMC
- 2700°F/2400F CMC
- 3000°F

**Bond Coat T:**
- Limited to 2462°F
- Limit to 2462°F
- Limit to 2642°F
- Proven at 2600°F +; Advancements targeting 2700°F
- 2700°F (2011 Goal)

**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

HfO₂-Si bond coat

NASA EBC processed by Triplex pro

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
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 low 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

Nozzle section view  Mid section view  End section (sample side) view

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 the relationship between temperature and strength for different EBC bond coat constituents.](chart.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.

![Graphs showing the effects of Si content on creep rates and strength at 1400°C.](image)
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

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

![Microstructure Image]

EDS A

EDS C

EDS B
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 measureable 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

<table>
<thead>
<tr>
<th>PVD-CVD</th>
<th>EB-PVD</th>
<th>APS*</th>
<th>FurnaceLaser/CVD/PVD</th>
</tr>
</thead>
<tbody>
<tr>
<td>YSi</td>
<td>YbGdYSi</td>
<td>HfO2-Si; REHfSi</td>
<td>REHfSi</td>
</tr>
<tr>
<td>ZrSi+Y</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
<td></td>
</tr>
<tr>
<td>ZrSi+Y</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
<td></td>
</tr>
<tr>
<td>ZrSi+Ta</td>
<td>YbGdYSi</td>
<td>GdYSi</td>
<td></td>
</tr>
<tr>
<td>HfSi + Si</td>
<td>YbGdSi</td>
<td>GdYSi-X</td>
<td></td>
</tr>
<tr>
<td>HfSi + YSi</td>
<td>YbGdSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HfSi+Ysi+Si</td>
<td>YbGdSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>YbSi</td>
<td>YbSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HfSi + YbSi</td>
<td>YbSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GdYbSi(Hf)</td>
<td>YbYSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>YYbGdSi(Hf)</td>
<td>YbYSi</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Process and composition</td>
<td>Hf-RESilicate</td>
<td>Used in ERA</td>
</tr>
<tr>
<td></td>
<td>transitions</td>
<td></td>
<td>components as part</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>of bond coat system</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Hf-RE-Al-Silicate</td>
<td>Used also in ERA</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>components as part</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>of bond coat system</td>
</tr>
</tbody>
</table>


*APS*: or plasma spray related processing methods
NASA EBC Bond Coats for Airfoil and Combustor EBCs

Continued

- 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

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
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

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

**Kp as a function of silicon content**

An oxidized bond coat after 1500°C 100 h creep testing

Oxidation kinetics of a YbGdSi(O) bond coat
Advanced EBC developments – Some Hybrid APS-PVD Systems and Qualification Tests

- EB-PVD HfO$_2$-RE$_2$O$_2$ (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

Some surface spallation

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

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
Advanced environmental barrier coatings – Prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 ksi at $T_{EBC}$ 2700°F (1482°C), $T_{CMC}$ interface ~2500°F (1371°C)

- The HfO$_2$-Si based bond coat showed excellent durability in the long term creep tests
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

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

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**

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

**Fatigue Tested**

- 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

![Graph showing fatigue or creep hot time, hours](image)

Creep-fatigue durability test summary

Tested, SA Tyrannohex with bond coat only

Tested, SA Tyrannohex with EBC system 188

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

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

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).

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

- Hot streaks with possible gas temperature over 2000°C, with minimum back cooling.

- Some minor coating spalling at hot streak impingement.

![Swirl jet flows](image)
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