Advanced Thermal Barrier and Environmental Barrier Coating Development at NASA GRC

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Durable Thermal and 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

Hybrid Electric Propulsion Aircraft
NASA Advanced Turbine Thermal and Environmental Barrier Coating Development Goals

- Develop innovative coating technologies and life prediction approaches
- 2500°F Turbine TBCs with high toughness, and improved impact erosion resistance
- 2700°F (1482°C) EBC bond coat technology for supporting next generation turbine engines
- 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
  - Improve impact/erosion and CMAS resistance

Temperature Capability

- 2850°F combuster TBC
- 2500°F Turbine TBC
- 3000°F+ (1650°C+) 3000°F SiC/SiC CMC airfoil and combustor technologies
- 2700°F (1482°C) 2700°F SiC/SiC thin turbine EBC systems for CMC airfoils
- 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)

Step increase in the material’s temperature capability

Increase in ∆T across T/EBC

Ceramic Matrix Composite

Single Crystal Superalloy

Gen I – Current commercial

Gen II

Gen III

Gen IV

Year

* Recession: <5 mg/cm² per 1000 hr (40-60 atm., Mach 1~2)
** Component strength and toughness requirements
NASA Turbine Thermal and Environmental Barrier Coatings for CMC-EBC Systems

- Emphasize temperature capability, performance and durability for next generation turbine engine systems
- Increase Technology Readiness Levels (TRLs) for component system demonstrations

Baseline metal temperature

Current metal turbine airfoil system

State of the art metal turbine airfoil system 2500°F TBCs

2700-3000°F CMC turbine airfoil systems
NASA Environmental Barrier Coating (EBC) - Ceramic Matrix Composite (CMC) Development Needs

- **Advanced Component Development Programs (particularly under the Environmentally Responsible Aviation Program):** Advanced environmental barrier coatings for SiC/SiC CMC combustor and turbine vane components, technology demonstrations in engine tests
  - N+2 (2020-2025) generation with 2400°F CMCs/2700°F EBCs (cooled)

- **NASA Aeronautics Program (FAP-SUP*, SRW/Aero Sciences/TTT** Projects):** Next generation high pressure turbine airfoil environmental barrier coatings with advanced CMCs
  - N+3 (2020-2025) generation with advanced 2700°F CMCs/2700-3000°F EBCs (uncooled/cooled)

- **Turbine TBC development under NASA Partner Collaborative Programs**

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* SUP: NASA Supersonics Project; SRW: NASA Subsonic Rotary Wing Project; TTT**: NASA Transformational Tools and Technologies Project.
Outline

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

— Advanced thermal and environmental barrier coating systems (EBCs) for CMC airfoils and combustors
  • NASA turbine and combustor EBC coating systems
  • Performance and modeling
  • Advanced EBC development: processing, testing and durability

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

— Summary
### NASA Turbine Environmental Barrier Coating Development: Major Emphases and Milestones

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<td>2.5D CVI CMC rig demo</td>
<td>2700°F turbine EBC on 2400-2500°F CMC turbine vanes Rig Demo (TRL 4-5)</td>
<td>NASA and NASA-ARFL for Embedded Sensor for TBC-EBC/CMCs</td>
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<td>NASA-NAVY Turbine TBC developments for Next-Gen high steam resistance</td>
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<td>FY 13</td>
<td>Develop creep resistant turbine EBC with advanced 2700°F bond coats</td>
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<td>2700°F EBC Advanced architecture hollow CMC blade subelements</td>
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Thermal and Environmental Barrier Coating Development: Challenges and Limitations

- Thermal barrier coatings and, in particular, environmental barrier coatings are limited in their temperature capability, water vapor stability and long-term durability
  - Prime-reliant coatings are critical for future 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

- Turbine airfoil coating low thermal conductivity critical (half k thermal and environmental barrier)

- Thermal and environmental barrier coating need improved impact, erosion and calcium-magnesium-alumino-silicate (CMAS) resistance
Development of Advanced Defect Cluster Low Conductivity Thermal Barrier Coatings

— Multi-component oxide defect clustering approach (Zhu et al, US Patents No. 6,812,176, No.7,001,859, and No. 7,186,466)
  e.g.: $\text{ZrO}_2$-$\text{Y}_2\text{O}_3$-$\text{Nd}_2\text{O}_3(\text{Gd}_2\text{O}_3,\text{Sm}_2\text{O}_3)$-$\text{Yb}_2\text{O}_3(\text{Sc}_2\text{O}_3)$ systems
  Primary stabilizer
  Oxide cluster dopants with distinctive ionic sizes

— Defect clusters associated with dopant segregation
— The nanometer sized clusters for reduced thermal conductivity, improved stability, toughness, CMAS resistance and mechanical properties

Plasma-sprayed $\text{ZrO}_2$-($\text{Y, Nd,Yb})_2\text{O}_3$

EB-PVD $\text{ZrO}_2$-($\text{Y, Nd,Yb})_2\text{O}_3$

EELS elemental maps of EB-PVD $\text{ZrO}_2$-($\text{Y, Gd,Yb})_2\text{O}_3$
Advanced Multi-Component Erosion Resistant Turbine Blade Thermal Barrier Coating Development

— Rare earth (RE) and transition metal oxide defect clustering approach (*US Patents No. 6,812,176, No.7,001,859, and 7,186,466; US Patent 7,700,508 NASA-Army*) specifically by additions of $\text{RE}_2\text{O}_3$, $\text{TiO}_2$ and $\text{Ta}_2\text{O}_5$

— Significantly improved toughness, cyclic durability and erosion resistance while maintaining low thermal conductivity

— Improved thermal stability due to reduced diffusion at high temperature

$\text{ZrO}_2$-$\text{Y}_2\text{O}_3$ - RE1 {e.g.,$\text{Gd}_2\text{O}_3$, $\text{Sm}_2\text{O}_3$}-RE2 {e.g.,$\text{Yb}_2\text{O}_3$, $\text{Sc}_2\text{O}_3$} – TT{$\text{TiO}_2$+$\text{Ta}_2\text{O}_5$} systems

◀ Primary stabilizer ▶ ▶ ▶ Toughening dopants

Oxide cluster dopants with distinctive ionic sizes
Thermal Conductivity of Multi-Component Thermal Barrier Coatings – Recent Developments with The Air Force Programs

- Rare earth (RE) and transition metal oxide defect clustering approach (US Patents No. 6,812,176, No.7,001,859, and 7,186,466; US Patents 7,700,508 - TBC and 7,740,960 - EBC; NASA-Army) specifically by additions of RE$_2$O$_3$, TiO$_2$ and Ta$_2$O$_5$.
Thermal Conductivity of NASA EB-PVD Low Thermal Conductivity Thermal Barrier Coatings

- Turbine TBC development focusing on toughness and CMAS resistance
- The systems are applicable to advanced environmental barrier coatings for ceramic matrix composites
Thermal Conductivity Optimization of A Series of NASA EB-PVD Processed Low Conductivity Thermal Barrier Coatings

- A $\text{ZrO}_2-m_1 \text{Y}_2\text{O}_3-m_2 \text{Gd}_2\text{O}_3-m_3 \text{Yb}_2\text{O}_3$ System Composition Optimization
- Low thermal conductivity and low rare earth design criteria, including the commercial coating alloy 206A

![Graph showing thermal conductivity data with markers at various dopant concentrations.](image-url)
Thermal Conductivity of ZrO$_2$-(Y,Gd,Yb)$_2$O$_3$ and ZrO$_2$-(Y,Gd,Yb)$_2$O$_3$ + TT( TiO$_2$-Ta$_2$O$_5$) Systems – Compared with Low k + Gd$_2$Zr$_2$O$_7$ Composite Systems

- The six-component low conductivity coating systems, for toughness and CMAS resistance, have lower TRLs

Thermal conductivity of EB-PVD erosion TBCs
Thermal Conductivity of $\text{ZrO}_2-(\text{Y,Gd,Yb})_2\text{O}_3$ and $\text{ZrO}_2-(\text{Y,Gd,Yb})_2\text{O}_3 + \text{TT( TiO}_2-\text{Ta}_2\text{O}_5)$ Systems – Compared with Low $k$ + $\text{Gd}_2\text{Zr}_2\text{O}_7$ Composite Systems - Continued

- The EB-PVD processing of low $k$ $t'$, low $k$ cubic phased and Gadolinium Zirconate coatings
Initial Furnace Cyclic Behavior of Advanced Multi-Component Rare Earth Oxide Cluster Coatings

— The dopant concentration and coating architecture have been optimized and developed to significantly improve the cyclic durability
— Some composition showed exceptional durability even at higher dopant concentrations
— Lower rare earth concentration was found to be preferred for better durability

(a) Plasma-sprayed coatings

(b) EB-PVD coatings
Furnace Cyclic Behavior of Advanced Multicomponent Thermal Barrier Coatings with an Interface $t'$ Coating layer

- $t'$ low $k$ TBCs had good cyclic durability
- The cubic-phase low conductivity TBC durability generally improved by an $7YSZ$ or low $k$ $t'$-phase interlayer

![Graph showing cycles to failure vs. total dopant concentration for different TBC systems at 2075°F (1135°C).]

- Low-$k$ $t'$-phase region
- Low-$k$ cubic phase region

With the interlayer

Without interlayer

EB-PVD low $k$ coatings
Furnace Cyclic Behavior of $\text{ZrO}_2-(\text{Y},\text{Gd},\text{Yb})_2\text{O}_3$ and with Co-doped with $\text{TiO}_2$ and $\text{Ta}_2\text{O}_5$

— Low to moderate concentration $\text{TiO}_2$ and $\text{Ta}_2\text{O}_3$ dopants significantly improve cyclic life and thus the coating toughness
Furnace Cyclic Behavior of ZrO$_2$-(Y,Gd,Yb)$_2$O$_3$ and with Co-dopant TiO$_2$-TaO$_5$ Thermal Barrier Coatings

1150°C Cyclic Oxidation of Low $k_T$ RE-doped PVD TBC, Pt-Al Bond Coat on Rene'N5

With J. Smialek

- Six component system
- Six component system

With J. Smialek
Advanced Ballistic Impact Resistant NASA Four-Component Low Thermal Conductivity Turbine Coatings

- 10X improvements in Impact Resistance; experience learnt for also being used for developing advanced EBC systems

![Graph showing spalled area vs. energy and projectile velocity](Image)

- 7YSZ and t lowk baseline TBCs
- Zr-RE-Ti-Ta t' oxide systems
- Impact resistant series (four-component systems)

Tested at 2100°F
Advanced Ballistic Impact Resistant NASA Four-Component Low Thermal Conductivity Turbine Coatings - Continued

— 10X improvement in Impact Resistance; experience learnt for also being used for developing advanced EBC systems

Four-component and six-component coating systems showed excellent impact (10X improvement) and erosion resistance (up to 2X) compared to 7YSZ baseline.
Erosion and Impact Aspects: Early Mach 0.3 Ballistic Impact Tests of HfO$_2$-Si Bond Coat EBC Systems

- Advanced EBCs on par with best TBCs
- More advanced EBC compositions in developments

![Graph showing spalled area vs. energy and projectile velocity](image)

- 7YSZ and low k TBCs
- ZrRETT
- ATES series
- ND2
- ND3
- ND6
- ND7
- SUP-ERA7.4 EBC
- t'-ZrO2/NASA EBC
- Cubic low k ZrO$_2$ k-NASA EBC

![Images of impacted coatings](image)
High Heat Flux CO$_2$ Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development with CMAS

- Direct Laser heat flux infiltrated thermal barrier coatings (Conventional CMAS)
- Low conductivity thermal barrier coatings may have improved “non-wetting” performance compared to baseline 7YSZ

ZrO$_2$-1.0Y$_2$O$_3$-1.5Gd$_2$O$_3$-1.5Yb$_2$O$_3$

ZrO$_2$-1.6Y$_2$O$_3$-1.2Gd$_2$O$_3$-1.2Yb$_2$O$_3$

7YSZ

- Direct Laser heat flux infiltrated thermal barrier coatings (Air Force/PTI CMAS)

EB-PVD Low k ZrO$_2$-4mol%Y$_2$O$_3$-3mol%Gd$_2$O$_3$-3mol%Yb$_2$O$_3$
/PtAl/Rene N5 (Howmet Processing-Run 3844, ID 15H1)
CMAS Related Erosion Failure (CMAS+Erosion)

- CMAS Tested Specimen in Burner Rig
High Heat Flux CO$_2$ Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development with CMAS

- Heat flux cyclic failure of a thick Gd$_2$Zr$_2$O$_7$ system tested at 1300°C, due to low toughness and the formation of a reaction layer

Typical cyclic failure due to the reaction layer within a few cycles in a Gd$_2$Zr$_2$O$_7$ system.
High Heat Flux CO₂ Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development with CMAS

- Low k ZrO₂-2.25mol%Y₂O₃-9mol%Gd₂O₃-2.25mol%Yb₂O₃ tested at 1300°C
- Limited CMAS spreading or penetration, suggesting the coating have resistance to CMAS
- Top and reacted layers had some spallation after 170h cyclic tests
- Preliminary effects of heat flux on baseline coatings determined, further studies planned
- Establish the life database and will compare with those of advanced systems
CMAS Reaction Studies for Advanced TEBCs: Advanced Low k and HfO<sub>2</sub> showed Potential Benefits

- CMAS reactions studied for selected coating candidate materials
- Preliminary results showed 7YSHf, ZrO<sub>2</sub>-9.6Y<sub>2</sub>O<sub>3</sub>-2.2Gd<sub>2</sub>O<sub>3</sub>-2.1Yb<sub>2</sub>O<sub>3</sub>, and 30YSZ had the highest CMAS resistance

CMAS resistance of selected coating systems

SEM cross – sectional electron images ceramic coating reacted with CMAS at 1300 °C for 5 h

With Gustavo Costa et al
Advanced Environmental Barrier Coatings Developments

• Fundamental studies of environmental barrier coating materials and coating systems, stability including recession in rig environments, temperature limits and failure mechanisms

• Focus on high performance and improving technology readiness levels (TRL), including high stability \(\text{HfO}_2\) and \(\text{ZrO}_2\)-RE\(_2\)\(\text{O}_3\)-SiO\(_2\)/RE\(_2\)Si\(_2\)-xO\(_7\)-2x environmental barrier systems, including processing optimizations for improved composition control and process robustness

• Advanced NASA \(\text{HfO}_2\)-Si and Rare Earth-Silicon based EBC bond coat systems
  • More advanced, multicomponent composition and composite EBC systems to improve the temperature capability, strength and toughness
  • Develop \(\text{HfO}_2\)-Si based + X (dopants) bond coat systems for \(2700^\circ\text{F} (1482^\circ\text{C})\) long-term applications
  • Develop *prime-reliant* \(2700^\circ\text{F}+ (1482^\circ\text{C})\) Rare Earth (RE)-Si + X (dopants) bond coat systems for advanced integrated EBC-CMC systems, improving bond coat temperature capability and durability
Developing 3000°F (1650°C) EBCs

- Hybrid 3000°F EBC system
  - High stability multicomponent HfO₂ Top Coat (Hf-RE-SiO₂ systems, tetragonal t’ ZrO₂ toughened rare earth silicate EBC; Ta, Ti additions)
  - Graded and Layer graded interlayers
  - Advanced HfO₂-Rare Earth-Alumino-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)
Also available alloys such as Metco AE 10155 and AE 9892 in APS systems

Strain tolerant interlayer
HfO₂-Rare Earth-Alumino-Silicate EBC
(e.g., Metco 10157)

HfO₂-Si or RE modified mullite bond coat
(e.g., Metco 10219 in APS systems)
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 a High Pressure Burner Rig simulated Turbine Environment

Recession rate = const. \( V^{1/2} \frac{P_{(H2O)}^2}{(P_{total})^{1/2}} \)

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

(a) Combustion gas | (b) Combustion gas

Cooling gas
NASA High Pressure High Velocity and High Heat Flux SiC/SiC and EBC Recession Studies Under Film Cooling Conditions

— Determined recession under complex, and realistic High Pressure Burner Rig and Laser Rig simulated turbine steam conditions

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

High temperature recession kinetics for film-cooled and non-film cooled Gen II SiC/SiC CMCs

Graph showing recession rates for different conditions:
- Film cooled recession at 2400°F
- Non-film cooling recession at 2400°F (model extrapolated to 300 m/s gas velocity)
- Film cooled recession at 2100°F
- Non-film cooling recession at 2100°F

**Specimen recession and durability testing under air cooled, heat flux conditions:**
- 1" and 2" discs, 600 psi cooling chamber

**Steam heat flux test rig:**

- Y2Si2O7, laser rig 1550°C steam
- Yb2SiO5/Yb2Si2-xO7, HPBR 1200°C, 20 m/s
- Yb2SiO5/Yb2Si2-xO7, HPBR 1350°C, 200 m/s
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

Interface Si bond coat melting of selected coating systems, under laser heat flux tests, 1” dia button specimen
The Yb$_2$SiO$_5$/Yb$_2$Si$_2$O$_7$ EBC Delamination Crack Propagation Tests under Heat Flux Thermal Gradient Test Conditions

- Penney-shaped crack initially size 1.5 mm in diameter, tested in air at 1350°C
- Crack propagated from 1.5 mm to 7.5 mm 60, 1 hr cyclic testing
- SiO$_2$ loss (volatility) accelerated crack propagation

After 60 hr, 1 hr cyclic testing
Ytterbium Mono-/Di-Silicate EBC Tested in Laser High Heat Flux Steam Rig

- Observed mudflat cracking after 1400°C test
- Loss of Silica and increased porosity observed after the testing
Fatigue Testing using a Laser High-Heat-Flux Approach for Environmental Barrier Coated Prepreg SiC/SiC CMCs

- Environmental Barrier Coatings Yb$_2$SiO$_5$/Yb$_2$Si$_2$O$_7$/Si on MI Prepreg SiC/SiC CMC substrates
- One specimen tested in air, air testing at 1316°C
- One specimen tested in steam, steam testing at $T_{\text{EBC}}$ 1316°C, $T_{\text{CMC}}$ at ~1200°C
- Lower CMC failure strain observed in steam test environments

Fatigue strains (amplitudes) – Time Plot

Thermal conductivity – Time Plot
Fatigue Testing using a Laser High-Heat-Flux Approach for EBC Coated Prepreg SiC/SiC CMCs - Continued

- Crack and recession failure in the laser rig air and steam environment tests.
Advanced High Temperature and 2700°F+ Bond Coat and EBC Development

NASA Advanced EBC Development:
- Advanced compositions ensuring environmental and mechanical stability
- Bond coat systems for prime reliant EBCs; capable of self-healing
- Corresponding oxygen containing EBCs with high toughness and CMAS resistance
- Composition further being developed for Ultra-High Temperature Ceramics and Coatings

High strength, high stability reinforced composites: HfO$_2$-Si and a series of Oxide-Si systems

HfO$_2$-Si based and minor alloyed systems for improved strength and stability, e.g., rare earth dopants

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

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

Temperature capability increase

Rare Earth – Si + Hf coating systems

Hf – Rare Earth – Si coating systems
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

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<th>EB-PVD</th>
<th>APS*</th>
<th>FurnaceLaser/CVD/PVD</th>
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<td>GdYSi</td>
<td>REHfSi</td>
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<td>GdYbSi(Hf)</td>
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<td>HfRESilicate</td>
<td>Used in ERA components as part of bond coat system</td>
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<td>Hf-RE-Al-Silicate</td>
<td>Used also in ERA components as part of bond coat system</td>
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Process and composition transitions

HfO2-Si; REHfSi; YSi+RESilicate
GdYSi GdYbSi GdYb-LuSi NdYSi

HfO2-Si; YSi+RESilicate
YSi+Hf-RESilicate

Hf-RESilicate

Used in ERA components as part of bond coat system

Used also in ERA components as part of bond coat system

APS*: or plasma spray related processing methods
NASA EBC Processing Developments for SiC/SiC Ceramic Matrix Composites

- 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
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

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

– NASA PS-PVD and PS-TF coating processing using Sulzer (Oerlikon) 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 potentially suitable for complex-shape components
  • Significant progress made in processing the advanced EBC and bond coats

100 kW power, 1 torr operation pressure

HfO\textsubscript{2}-Si bond coat

Processed coating systems

NASA PS-PVD Coater System
HfO$_2$-Si Bond Coats Processing using EB-PVD compared with Early Hot-Press Coatings - Continued

- Capable of processing silicide dominant HfO$_2$-Si bond coats in EB-PVD coating and magnetron PVD
- Graded coatings being designed and used
- Processing nano-structured coatings

<table>
<thead>
<tr>
<th>HfO$_2$-Si</th>
<th>At%</th>
<th>Wt%</th>
<th>Units</th>
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<tr>
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<td>6.70</td>
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<tr>
<td>Si</td>
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<tr>
<td>Hf</td>
<td>24.82</td>
<td>71.15</td>
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<tr>
<td>Total</td>
<td>100.00</td>
<td>100.00</td>
<td>wt.%</td>
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HfO$_2$-Si (EB-PVD 48-6) Bond Coat

![Graph showing diffraction angle, 2θ vs. relative intensity with markers for different compositions and their corresponding intensities.](image)
Environmental Barrier Coatings and SiC/SiC DIC Testing – High Temperature (12C-470-08-11 and 12C-470-08-12)

- The uncoated and EBC HfO$_2$-Si coated CVI-MI specimen pre-tested in high pressure burner rig
- Uncoated specimen exposed showed severe degradation of composite properties
- Oxidation and embrittlement of the MI-CVI CMC in HPBR lead to the lowers strength of uncoated specimen

With Matt Appleby et al, Surface and Coatings Technology, 2015
Environmental Barrier Coatings and SiC/SiC DIC Testing – High Temperature (12C-470-08-11 and 12C-470-08-12)

- DIC studies of coated and uncoated CMC specimens
- Energy distribution of AE events compared in specimen gage section with corresponding DIC strain mapping at failure stress

**COATED 12C-470-08-11**
Nominal thermomechanical strain: 0.96%

**UNCOATED 12C-470-08-12**
Nominal thermomechanical strain: 0.71%

DIC studies of coated and uncoated CMC specimens

Energy distribution of AE events compared in specimen gage section with corresponding DIC strain mapping at failure stress

Nominal thermomechanical strain:
- 0.96% for COATED 12C-470-08-11
- 0.71% for UNCOATED 12C-470-08-12

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Advanced EBC-CMC Fatigue Test with CMAS: Successfully Tested 300 h Durability in High Heat Flux Fatigue Test Conditions - Continued

- Advanced Hf-NdYb silicate-NdYbSi bond coat EBC coatings on 3D architecture CVI-PIP SiC-SiC CMC (EB-PVD processing)

Surface view CMAS 35mg/cm²

Back view CMAS 35mg/cm²

EBC/CMC-CMAS Laser heat Flux Fatigue Tests
Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs

- A thin EB-PVD turbine airfoil EBC system with advanced HfO$_2$-(Yb,Gd,Y) silicate and (Yb,Gd)Si bond coat tested 300hr at $T_{\text{EBC-surface}}$ 1537°C, $T_{\text{bond coat}}$ 1480°C, $T_{\text{back CMC surface}}$ 1250°C with CMAS
- Fatigue stress amplitude 69 MPa, at frequency $f=3$Hz, stress ratio $R=0.05$
Laser Rig Testing and Development of NASA Advanced Multicomponent Yb-Gd-Y Silicate EBC/HfO$_2$-Si System on 3D Architecture SiC/SiC CMC under 2700°F+ SPLCF Conditions

- The EBC specimens tested under the laser heat flux test rig under 10 ksi (500 hr) and 15 ksi (140 hr completed) SPLCF conditions, respectively;
- One EBC specimen tested in isothermal furnace test at 2700°F, 300 hr completed for comparisons.

```
RB2014-54-4, EBC 512h/CMC 492h
RB2014-54-6, EBC 140h
RB2014-54-8, Isothermal furnace 300hr
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Laser MTS 810 Test rig

Example EBC cross-section

Laser rig test creep strains

Laser NDE and in-plane thermal conductivity measurements
Fundamental EBC Studies, and High Stability and CMAS Resistant Advanced EBC Developments: High Melting Point Coating, and Multi-Component Compositions

- Demonstrated Calcium-Magnesium-Alumino-Silicate (CMAS) resistance for NASA RESi system at 1500°C, 100 hr
- Silica-rich phase precipitation
- Still some rare earth elements leaching into the melts (low concentration ~9 mol%)

Valerie Wiesner

Interaction Region

Y₂Si₂O₇ Substrate (EBC)

Y₂Si₂O₇ Substrate Exposed to CMAS at 1200°C for 20h

200 hr, 1500°C

Valerie Wiesner

Ahlborg & Zhu

CMAS melts

Surface side of the CMAS melts

Area A

Area B

EDS E

200 hr, 1500°C

Ahlborg & Zhu
Laser Rig Thermomechanical Creep - Fatigue Tests of Advanced 2700°F+ RESi Bond Coats and EBC Systems

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

**EB-PVD Rare Earth Silicate EBC/YbGdYSi bond coat on CVI-(MI)**

\[ T_{EBC\ surface} = 2850-3000°F (1600-1650°C) \]

\[ T_{cmc\ back} \approx 2600°F (1426°C) \]

**Fatigue Tested**

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

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

NASA 2700°F(1482°C)+ EBC System 188 on SA Tyrannohex SiC Composite, 1482°C 15 ksi, 500hr

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**Creep and Fatigue Tests with CMAS**

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

**Creep rate**

\[ 7.1 \times 10^{-6} \text{/s} \]

The Advanced EBCs 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

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

Uncoated vane tested 15 hr

EBC Coated Rig Inner and outer liner testing 2500°F, 10-16 atm, completed 250 h

Vane leading edge seen from viewport in High Pressure Burner Rig Testing 16 atm, 200 m/s, up to 2650°F
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, $T_{CMC}$ interface ~2500°F

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

- Advanced thermal barrier coatings are based on rare earth co-doped, defect clustered oxide systems, aiming at low thermal conductivity, and high thermal stability, and impact/erosion CMAS resistance
- Durable EBCs are critical to emerging SiC/SiC CMC component technologies, requiring prime-reliant designs
- 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 Zr, Hf, 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 for helping prime-reliant EBC designs
  - New EBC compositions improved combustion steam and CMAS resistance, and protecting CMCs
- EBC processing and testing capabilities significantly improved
- Advanced testing and modeling being emphasized
- Focused on next generation turbine airfoil EBC developments, demonstrated component EBC technologies in simulated engine environments
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