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

* 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

<table>
<thead>
<tr>
<th>Temperature</th>
<th>TBC</th>
<th>Bond coat</th>
<th>Metal blade</th>
<th>Gas</th>
<th>TBC</th>
<th>Bond coat</th>
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Current metal turbine airfoil system

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

2700-3000°F CMC turbine airfoil systems

Thin turbine coating development

Tsurface 300°F increase

2700°F CMCs

2400°F CMCs
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

**Advanced EBCs**
- Durable CMC w/EBC developed - 2700°F turbine coating for 2400°F CMC (15 ksi 1000 h durability)
- Demo EBC for CMC subelement
- Develop creep resistant turbine EBC with advanced 2700°F bond coats
- Impingement & Film cooled recession
- Develop advanced architecture hollow CMC blade subelements
- Turbine EBC on CMC blade subcomponents
- Initial environment and fatigue interactions
- Rig Demo Optimized 2700°F EBC-3D CMC blades
- 2700°F EBC on 3D CMC vane (TTT Augmentation Project)
- 2700°F Combustor EBC on 2400°F CMC Combustor – Rig Demo 250 hr (TRL 4-5)
- Advanced CMAS Resistant Turbine Coating Rig and Engine Demos (with Army and NAVAIR)

**CMC Development Supporting Advanced EBC-CMC Development**
- 2.5D CVI CMC rig demo

**EBC/CMMC Component Development Program – NASA ERA Program**
- 2X Erosion resistant turbine TBC
- Luminance sensing TBC
- NASA-NETL Turbine TBC developments for Next-Gen high steam resistance

**Turbine TBC Erosion/CMAS (in collaboration with DOE and Air Force, Army, Navy)**
- NASA and NASA-ARFL for Embedded Sensor for TBC-EBC/CMCs
- NASA Turbine Coatings Supporting Tinker Air Force Base Turbine Engine Programs

**FY 11**
- 2700°F turbine EBC on 2400-2500°F CMC turbine vanes
- Rig Demo (TRL 4-5)

**FY 12**
- 2700°F Combustor EBC on 2400°F CMC

**FY 13**
- Advanced CMAS Resistant TBC (with ARFL Venture and IR&D programs)

**FY 14**
- Initial environment and fatigue interactions

**FY 15**
- CMAS EBC 300h durability Demo
- 2700°F EBC-Advanced architecture hollow CMC blade subelements

**FY 16-17**
- Advanced CMAS Resistant Turbine Coating Rig and Engine Demos (with Army and NAVAIR)
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.: ZrO$_2$-Y$_2$O$_3$-Nd$_2$O$_3$(Gd$_2$O$_3$,Sm$_2$O$_3$)-Yb$_2$O$_3$(Sc$_2$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 ZrO$_2$-(Y, Nd,Yb)$_2$O$_3$

EB-PVD ZrO$_2$-(Y, Nd,Yb)$_2$O$_3$

EELS elemental maps of EB-PVD ZrO$_2$-(Y, Gd,Yb)$_2$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 RE$_2$O$_3$, TiO$_2$ and Ta$_2$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

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

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Thermal conductivity, W/m-K

Total dopant concentration, mol%

Metco 204 8YSZ (Porous) k0
Metco 204 8YSZ (Porous) k20
ZrO$_2$ 312 Low k k0
ZrO$_2$ 312 Low k k20
ZComP Low k k0
ZComP Low k k20

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Plasma-sprayed coatings

Advanced ZrO$_2$-based coatings

8YSZ k0 (2500F)
8YSZ k20 (2500F)
8YSZ k0 (2600F)
8YSZ k20 (2600F)
8YSZ k0 (~2500F)
8YSZ k20 (~2500F)
8YSZ k0 (~2700F)
8YSZ k20 (~2700F)
ZrO$_2$ 312 Low k k0
ZrO$_2$ 312 Low k k20
ZComP Low k k0
ZComP Low k k20
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 $\text{ZrO}_2 - m_1 \ Y_2\text{O}_3 - m_2 \ Gd_2\text{O}_3 - m_3 \ Yb_2\text{O}_3$ System Composition Optimization
- Low thermal conductivity and low rare earth design criteria, including the commercial coating alloy 206A
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 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 - Continued

- The EB-PVD processing of low k $t'$, low k cubic phased and Gadolinium Zirconate 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.

Tested at 1165°C, 45 min hot time cycles.
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
Furnace Cyclic Behavior of ZrO$_2$-(Y,Gd,Yb)$_2$O$_3$ and with Co-doped with TiO$_2$ and Ta$_2$O$_5$

— Low to moderate concentration TiO$_2$ and Ta$_2$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
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

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

Tested at 2100°F

150 200 250 300 350 400 450 500

0.0 0.5 1.0 1.5 2.0 2.5

Spalled area, cm$^2$

Energy, J

ATES 104
ATES 105
ATES 103

Projectile velocity, m/s

103 2.0xV 40.0mm x600 SE(L)
105 2.0xV 17.2mm x1.00k SE(L)
104 2.0xV 11.9mm x1.50k SE(L)
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]
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

- 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

*Figure: NASA Low k Metco AE10389 coating specimen, after 170 hr tested in laser rig*
CMAS Reaction Studies for Advanced TEBCs: Advanced Low k and HfO₂ showed Potential Benefits

- CMAS reactions studied for selected coating candidate materials
- Preliminary results showed 7YSHf, ZrO₂-9.6Y₂O₃-2.2Gd₂O₃-2.1Yb₂O₃, 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 HfO$_2$ and ZrO$_2$ -RE$_2$O$_3$-SiO$_2$/RE$_2$Si$_{2-x}$O$_{7-2x}$ environmental barrier systems, including processing optimizations for improved composition control and process robustness

- Advanced NASA 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 HfO$_2$-Si based + X (dopants) bond coat systems for 2700°F (1482°C) long-term applications
  - Develop *prime-reliant* 2700°F+ (1482°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

Recursion rate = const. $V^{1/2} \frac{P_{(H_2O)}^2}{(P_{total})^{1/2}}$

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

**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

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

Examples of environmental barrier coating recession in laboratory simulated turbine engine conditions
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 $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7$ 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
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 $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7/\text{Si}$ 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 tests

In Air; EBC cracking

In steam; EBC cracking and volatility
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

Temperature capability increase

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

HfO₂-Si based and minor alloyed systems for improved strength and stability, e.g., rare earth dopants

High strength, high stability reinforced composites: HfO₂-Si and a series of Oxide-Si systems

Advanced 2700°F+ Rare Earth - Si Bond Coat systems

HfO₂-Si systems

Advanced 2700°F+ Rare Earth - Si Bond Coat systems

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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Process and composition transitions

- HfO2-Si; REHfSi
- GdYSi
- GdYbSi
- GdYb-LuSi
- NdYSi

- Hf-RESilicate
  Used in ERA components as part of bond coat system

- Hf-RE-Al-Silicate
  Used also in ERA components
  Used 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

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

NASA HfO$_2$-Si bond coat on SiC/SiC

NASA Hybrid EBC on SiC/SiC

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 (Oerlikon) 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 potentially suitable for complex-shape components
  - Significant progress made in processing the advanced EBC and bond coats

![Images of nozzle section view, mid section view, and end section (sample side) view of the coating system.](Image)

![NASA PS-PVD Coater System](Image)

![Processed coating systems](Image)

100 kW power, 1 torr operation pressure

**HfO₂-Si bond coat**
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

### Relative Intensity vs. Diffraction angle, $2\theta$

<table>
<thead>
<tr>
<th>Compound</th>
<th>HfO$_2$-Si 25wt%Si-1350°C</th>
<th>HfO$_2$-50wt%Si-1350°C</th>
<th>HfO$_2$-75wt%Si-1400°C</th>
<th>EB-PVD HfO$_2$-Si 48-6</th>
</tr>
</thead>
<tbody>
<tr>
<td>At%</td>
<td>26.08</td>
<td>22.15</td>
<td>71.15</td>
<td>100.00</td>
</tr>
<tr>
<td>Wt%</td>
<td>6.70</td>
<td>22.15</td>
<td>71.15</td>
<td>100.00</td>
</tr>
<tr>
<td>Units</td>
<td>wt.%</td>
<td>wt.%</td>
<td>wt.%</td>
<td>wt.%</td>
</tr>
</tbody>
</table>

### HfO$_2$-Si (EB-PVD 48-6) Bond Coat

- **O**: 26.08 at%, 6.70 wt% (wt.%)
- **Si**: 49.10 at%, 22.15 wt% (wt.%)
- **Hf**: 24.82 at%, 71.15 wt% (wt.%)
- **Total**: 100.00 at%, 100.00 wt% (wt.%)

**Note:** The graph shows the relative intensity of different bonds at various diffraction angles, indicating the distribution and intensity of the components in the bond coat.
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

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Surface Temp. (°C)</th>
<th>Back Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>coated</td>
<td>1230</td>
<td>1070</td>
</tr>
<tr>
<td>uncoated</td>
<td>1200</td>
<td>1010</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Specimen</th>
<th>E (GPa)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>$\varepsilon_{fail}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>coated</td>
<td>241</td>
<td>266</td>
<td>238</td>
</tr>
<tr>
<td>uncoated</td>
<td>146</td>
<td>221</td>
<td>166</td>
</tr>
</tbody>
</table>
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

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)

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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

Fatigue-creep strain, %

Fatigue cycles

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 $R=0.05$

1537°C, 10ksi, 300 h fatigue (3 Hz, R=0.05) on 14C579-011001_#8 CVI-SMI SiC/SiC (with CMAS)
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

Laser MTS 810 Test rig

Laser NDE and in-plane thermal conductivity measurements

Example EBC cross-section

EBC
Bond Coat
SiC/SiC CMC

Laser rig test creep strains
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

Ahlborg & Zhu

Surface side of the CMAS melts
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)  
\[ T_{EBC \text{ surface}} = 2850-3000°F \ (1600-1650°C) \]  
\[ T_{cmc \ back} \ at \ ~2600°F \ (1426°C) \]

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

- EB-PVD (Hf,Yb,Gd,Yb)\(_2\)Si\(_2\),xO\(_7\),x EBC/GdYbSi bond coat on CVI-MI SiC/SiC (with CMAS)  
  1537°C, 10ksi, 300 h fatigue (3 Hz, R=0.05)


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
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

Swirl jet flows

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

- 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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