Advanced Environmental Barrier Coating Development for SiC-SiC Ceramic Matrix Composite Components

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Durable Thermal and Environmental Barrier Coating Systems for Ceramic Matrix Composites (CMCs):
Enabling Technology for 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
National Aeronautics and Space Administration

NASA Environmental Barrier Coating (EBC) - Ceramic Matrix Composite (CMC) Development Needs

- **NASA Aeronautics Programs**: Next generation high pressure turbine airfoil environmental barrier coatings with advanced CMCs
  - N+3 generation (2020-2025) with advanced 2700°F CMCs/2700-3000°F EBCs (uncooled/cooled)

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

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**Diagram Description**

- **Fuel Staging**
- **CMC Liners**
- **Instability Control**
- **Multipoint Injection**
- **Low emission combustor**
- **High Pressure Turbine CMC vane and blade**
**NASA Environmental Barrier Coatings (EBCs) and Ceramic Matrix Composite (CMC) System Development**

- **Emphasize material temperature capability, performance and long-term durability:**
  - Highly loaded EBC-CMCs with temperature capability of 2700°F (1482°C)
  - 2700-3000°F (1482-1650°C) turbine and CMC combustor coatings
  - 2700°F (1482°C) EBC bond coat technology for supporting next generation
    - Recession: <5 mg/cm² per 1000 h
    - Coating and component strength requirements: 15-30 ksi, or 100-207 Mpa
    - Resistance to Calcium Magnesium Alumino-Silicate (CMAS)

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### Temperature Capability

- **2800°F** combustor TBC
- **2500°F** Turbine TBC

**Step increase in the material’s temperature capability**

- **2700°F (1482°C)**
- **3000°F+ (1650°C+)**

**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), PtAl and NiAl bond coats**

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**Ceramic Matrix Composite**

**Single Crystal Superalloy**

**Gen II – Current commercial**

**Gen III**

**Gen. IV**
Outline

— **SiC/SiC ceramic matrix composite environmental barrier coating system development**
  • Environmental barrier coatings combustors – compositions, processing scaleup, and rig tests
    – HfO$_2$-Si based 2700°F bond coats
    – Rare Earth and Hafnium-rare earth-silicate EBCs
  • Advanced thermal spray and hybrid vapor - plasma processing coatings for SiC/SiC CMC components – environmental stability assessments

— The EBC system degradations and failure modes in long-term High Pressure Burner Rig liner tests

— NASA advanced 2700°F CMAS resistance coating developments
  • CMAS resistance evaluations of plasma sprayed combustor coatings

— **Summary and conclusions**
NASA Combustor EBC Development

• High stability multicomponent HfO$_2$ or ZrO$_2$, HfO$_2$-RE$_2$O$_3$-SiO$_2$/RE$_2$Si$_{2-x}$O$_{7-2x}$ / environmental barrier/environmental barrier coating systems

• Advanced 2700°F capable bond coats
  – Hafnium aluminate-silicates and rare earth aluminate silicates developed
  – HfO$_2$-Si first Gen bond coat for component tests
  – Second Gen 2700°F bond coat being developed based on rare earth -Si
  – Calcium Magnesium Alumino-Silicate (CMAS) resistance was addressed

• Develop advanced compositions for combustor EBC applications with Sulzer (Oerlikon) Metco, Praxair and others

• Develop high toughness and CMAS resistant coating systems
Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability

— Hafnium Rare Earth Silicate System and Rare Earth Silicate EBC Systems
— Multi-component EBC systems are preferred and being developed

High temperature and t’ phase region high toughness system and CMAS resistance

Rare earth monosilicate and di-silicate systems
Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability

— Multi-component oxide defect clustering approach for stability and CMAS resistance
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
  
  ![Image](Plasma-sprayed ZrO$_2$-(Y, Nd,Yb)$_2$O$_3$)
  
  ![Image](EB-PVD ZrO$_2$-(Y, Nd,Yb)$_2$O$_3$)
  
  ![Image](EELS elemental maps of EB-PVD ZrO$_2$-(Y, Gd,Yb)$_2$O$_3$)

  Primary stabilizer
  Oxide cluster dopants with distinctive ionic sizes

— The nanometer sized clusters for reduced thermal conductivity, improved stability, toughness, CMAS resistance and mechanical properties

Nano-reactive high stability Yb,Hf silicate based EBCs CMAS melt stabilizers Gd or Nd dopants

Primary stabilizer
Oxide cluster dopants with distinctive ionic sizes
Thermal Gradient Tensile Creep Rupture 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 $T_{EBC} 2700^\circ F$, $T_{CMC}$ interface $\sim 2500^\circ F$

- EBCs helped extending CMC rupture life in air tests

- The HfO$_2$-Si bond coat showed excellent durability

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Typical premature failure

Gen II CMC uncoated
Tested at 20 ksi, 2400°F

Gen II CMC with advanced EBC
Tested at 15 ksi & heat flux
$T_{surface} = 2700^\circ F$
$T_{interface} = 2500^\circ F$
$T_{CMC} \text{back} = 2320^\circ F$

Gen II CMC with advanced EBC
Tested at 15 ksi & heat flux
$T_{surface} = 2700^\circ F$
$T_{interface} = 2500^\circ F$
$T_{CMC} \text{back} = 2320^\circ F$

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Gen II CMC uncoated
Tested at 15 ksi, 2400°F

Gen II CMC with advanced EBC
Tested at 20 ksi, 2400°F

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Total strain, %

Time, hours

Gen II CMC with advanced EBC
Tested at 20 ksi & heat flux
$T_{surface} = 2700^\circ F$
$T_{interface} = 2450^\circ F$
$T_{CMC} \text{back} = 2250^\circ C$

Gen II CMC with advanced EBC
Tested at 20 ksi & heat flux
$T_{surface} = 2700^\circ F$
$T_{interface} = 2450^\circ F$
$T_{CMC} \text{back} = 2250^\circ C$

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Laser beam delivery optic system

Cooling shower head jets

High temperature extensometer

Test specimen
Thermal Gradient Tensile Creep Rupture 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 si at $T_{EBC}$ 2700°F, $T_{CMC}$ interface ~2500°F
- The HfO$_2$-Si bond coat showed tensile loading cracking resistance

EBCs on Gen II CMC after 1000 hr creep rupture testing

Hybrid EBCs on Gen II CMC after 1000 hr low cycle creep fatigue testing
**Environmental Barrier Coating Composition Development: High Temperature Capability, High Toughness and Improved Environmental Stability**

- Environmental Barrier Coatings Yb₂SiO₅/Yb₂Si₂O₇/Si on Melt Infiltrated (MI) Prepreg SiC/SiC CMC substrates
- One specimen tested in air, air testing at 1316°C
- One specimen tested in steam, steam testing at $T_{EBC}$ 1316°C, $T_{CMC}$ at ~1200°C
- Lower CMC failure strain observed in steam test environments
- Ytterbium monosilicate recession observed in the test

![Fatigue strains (amplitudes) – Time Plot](image1.png)

![Thermal conductivity – Time Plot](image2.png)
Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - Continued

- Plasma sprayed HfO$_2$-RE$_2$O$_3$ (Silicate) top coat EBCs showed good stability from 2” discs specimens
- Demonstrated high pressure environmental stability at 2600-2650°F, no measurable recession weight loss in 160-200 psi (10-16 atm) in the high pressure burner rig test

EBC ID 23_3.5.3: Three-layer system HfO$_2$-Si bond coat/Hf-Yb silicate EBC layer /Silica graded multicomponent Hf-RE silicate EBC on Prepreg

High pressure burner rig, 16 atm, 31 hr

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

High pressure burner rig tested new ND series Hybrid EBC systems coated on 2” diameter Gen II Prepreg SiC/SiC CMCs
Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - Continued

- Multicomponent rare earth silicate \((Yb,Gd,Y)_{2}Si_{2-x}O_{7-2x}\) EBC Composition showed excellent stability in laser heat flux steam tests at 1500°C

- High strengths and also showed improved CMAS resistance

Air, 50h

Laser Rig Steam, 200h at 1500°C

- CMAS resistance tested, 1500°C,
  100h cyclic
- More advanced systems in development
Environmental Stability Testing of the Combustor Environmental Barrier Coating SiC/SiC CMCs - Continued

- NASA Rare Earth Alumino-Silicate EBC composition, a more advanced version of the EBC system, showed high toughness (~1.8 MPa m^{0.5}) and high strength (~160 MPa), also with improved creep resistance.
- Laser steam 50 h tests showed significant strength reductions.
- More studies underway to confirm the results, helping develop high performance EBCs.

EBC Flexural Strength

<table>
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<th>Material Run</th>
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<tr>
<td>Flexural Strength (MPa)</td>
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<td>Temperature (°C)</td>
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<td>697-13 tensile surface</td>
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<td>697-13 side</td>
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<tr>
<td>697-14 tensile surface</td>
<td></td>
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<tr>
<td>697-14 side</td>
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- 697
- Steam exposed 1500°C 50 hrs 10/20 span
- Steam exposed 1500°C 50hrs 20/40 span
Advanced EBC Processing - Plasma-Sprayed and EB-PVD Based Approaches

- Processed coatings had HfO$_2$, hafnon, and hafnium silicides nano phases

NASA PS-PVD processed HfO$_2$-Si EBC bond coat

Directed Vapor Processing systems

EB-PVD processed HfO$_2$-Si bond coat

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<th>At%</th>
<th>Wt%</th>
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<tr>
<td>O</td>
<td>26.08</td>
<td>6.70</td>
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<tr>
<td>Si</td>
<td>49.10</td>
<td>22.15</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>
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</table>
Advanced EBC Processing- Plasma-Sprayed and EB-PVD Based Approaches - Continued

- Processed coatings had HfO₂, hafnon, and hafnium silicid nano phases

Hf-Si-O coating systems processed using EB-PVD (US Patent Applications Serial No.: 13/923,450 and 15/582,874)

Developing high toughness and high stability, low diffusion 2700°F (1482°C) HfSiO bond coats
EBC Scaleup, and Designs High Pressure Burner Rig SiC/SiC Liner Test Configurations – SiC/SiC Liner Test Articles Setup

— Focused on advanced composition and processing developments using state-of-the-art techniques
— Long-term durability testing in rig environments
High Pressure Burner Rig SiC/SiC Liner Test Configurations

- High Pressure Burner Rig modified for realistic cooled liner subelement and liner component testing
  - Film-cooled durability and recession tests
  - EBC coated SiC/SiC CMC liner tests

- 3000°F combustor gas temperature
- 16 atm pressure
- Heated cooling air
- Up to 300 m/s gas velocity used

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Heated cooling air
Specimen test section
Combustor specimen test section
Burner nozzle (2" dia) and duct
CMC test specimen

Pyrometer surface temperature measurements through viewports

2" diameter disc

High pressure burner rig
High Pressure Burner Rig SiC/SiC Liner Test Configurations – SiC/SiC Liner Test Articles Setup

Combustion outer chamber modified to accommodate increased dimension and tapered configuration

First set SiC/SiC liner Thermocouple (TC) arrangement configurations (total 24 TCs, 1/16” size), film cooled liner planned for second and third set testing
The Prepreg SiC-SiC CMC Combustor Liners Successfully Tested for 50h Durability in NASA High Pressure Burner Rig up to 3600°F

- Tested pressures at 500 psi external for outliner, and 220 psi inner liners in the combustion chamber (16 atm)
- Average gas temperatures at 3000°F (1650°C), the liner EBCs tested at 2500-2600°F with heat fluxes 20-35 W/cm², and the CMC liner component at 1800-2100°F
- Hot gas streaks may have had temperatures over 3632°F (2000°C), with higher transfer coefficients
- SiC/SiC CMC liners and EBCs survived 255 h

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

- Average gas temperature 1650°C, heat transfer coefficient 0.05-0.10 W/cm²-K
- Swirl jet flows
- Hot streaks with possible gas temperature over 2000°C, with minimum back cooling

Some minor coating spalling at hot streak impingement during testing
The Prepreg SiC-SiC CMC Combustor Liners Successfully Tested for 50h Durability in NASA High Pressure Burner Rig up to 3600°F - Continued

Hafnium-rich phase melting in the hottest liner section confirms the locally very high temperatures
Some Observed Degradations after 250 h Tests

- Observed EBC delamination, possibly under combined thermal and mechanical loading in one of the most severe condition tested sections
- Plasma sprayed HfO$_2$-Si bond coat showed better adhesion and durability

RE silicates and Cubic phase EBC may not have sufficient strength and toughness
Some Observed Degradations after 250 hr Tests - Continued

- Plasma sprayed HfO$_2$-Si bond coat showed good adhesion and durability
- Some silica formation at the bond coat/CMC interface after 250hr tests
Some Observed Degradations after 250 hr Tests - Continued

- Plasma sprayed HfO$_2$-Si bond coat showed good adhesion and durability
- Maintained low oxygen content at the bond coat – CMC interface even at very high temperature regions
- Some fiber degradations observed

Multicomponent silicate EBC

HfO$_2$-Si bond coat
Some Observed Degradations after 250 hr Tests - Continued

- Observed degraded bond coat region and with more extensive C containing SiO$_2$ scale formation
- More severe fiber, fiber coating and CMC degradations

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<tr>
<th>Element Symbol</th>
<th>Atomic Conc.</th>
<th>Weight Conc.</th>
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<td>O</td>
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<tr>
<td>Si</td>
<td>19.62</td>
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<tr>
<td>C</td>
<td>25.13</td>
<td>17.38</td>
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CMAS Resistance of the Advanced EBCs: Reacted with CMAS
1500°C, 100h

- The Advanced EBC generally developed showed low Rare Earth (RE) dissolution and thus better CMAS resistance

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<th>Component</th>
<th>Type</th>
<th>Mole Conc.</th>
<th>Mole Conc.</th>
<th>Conc.</th>
<th>Conc.</th>
<th>wt.%</th>
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<td>SiO$_2$</td>
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<td>17.999</td>
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<td>wt.%</td>
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<td>CaO</td>
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<td>HfO$_2$</td>
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<td>48.256</td>
<td>wt.%</td>
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100.000 100.000 wt.%
Summary

- Advanced EBCs developed, evaluated for 2700-3000°F CMC combustor liner applications in NASA high pressure burner rig
  - Valuable test data obtained on EBCs and CMCs
  - Several new compositions evaluated and developed

- Multicomponent EBC showed promise for high temperature capability, steam and combustion environment stability, and initial CMAS resistance
  - Bond coat composition optimization being optimized and also for commercial applications
  - Rare earth –Si bond coats also developed

- The EBC – SiC/SiC liner component demonstrated initial durability in very harsh test conditions, improved the Technical Readiness Levels under the NASA programs

- Property data and EBC Failure modes also studied
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