The Development of Environmental Barrier Coating Systems for SiC-SiC Ceramic Matrix Composites: Environment Effects on the Creep and Fatigue Resistance

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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
Outline

- Environmental barrier coating system development: needs, challenges and limitations

- 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 development, processing, testing and modeling
  - EBC and EBC bond coats: recent advances

- Design tool and life prediction of coated CMC components

- Advanced CMC-EBC rig demonstrations

- Summary and future directions
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 component system durability – working towards prime reliant coatings
  • Establish database, design tools and coating lifing methodologies
  • Improve technology readiness

Fix Wing Subsonic Aircraft  Supersonics Aircraft
NASA Environmental Barrier Coating Development Goals

- Emphasize temperature capability, performance and durability
  - Low silica activity silicate and high stability/high toughness oxide system developments
- 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 hr for subsonic aircraft and 9,000 hr for supersonics/high speed aircraft hot-time life requirements

Step increase in the material’s temperature capability

2800°F combustor TBC

2500°F Turbine TBC

Increase in ΔT across T/EBC

Ceramic Matrix Composite

Single Crystal Superalloy

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

2400°F (1316°C) Gen I and Gen II SiC/SiC CMCs

2000°F (1093°C)

* Recession: <5 mg/cm² per 1000 hr (40-50 atm, Mach 1~2)
** Component strength and toughness requirements
Evolution of NASA EBC Technology for SiC/SiC Ceramic Matrix Composites: Current State of the Art

### Advanced EBC system developments

<table>
<thead>
<tr>
<th>Engine Components:</th>
<th>Combustor</th>
<th>Combustor/ (Vane)</th>
<th>Combustor/ Vane</th>
<th>Vane/ Blade</th>
<th>- Vane/Blade EBCs - Equivalent APS combuster EBCs</th>
<th>Airfoil components</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Top Coat:</strong></td>
<td>BSAS (APS)</td>
<td>RE₂Si₂O₇ or RE₂Si₅ (APS)</td>
<td>- (Hf,Yb,Gd,Y)₂O₃ - ZrO₂/HfO₂+RE silicates - ZrO₂/HfO₂+BSAS (APS and EBPVD)</td>
<td>RE-HfO₂-Alumino silicate (APS and/or 100% EB-PVD)</td>
<td>RE-HfO₂-X advanced top coat RE-HfO₂-graded Silica (EB-PVD)</td>
<td>Advanced EBC</td>
</tr>
<tr>
<td><strong>Interlayer:</strong></td>
<td>--</td>
<td>--</td>
<td>RE-HfO₂/ZrO₂₂₆₂₆ aluminosilicate layered systems</td>
<td>Nanocomposite graded oxide/silicate</td>
<td>Gen IV interlayer not required (optional)</td>
<td></td>
</tr>
<tr>
<td><strong>EBC:</strong></td>
<td>Mullite+ BSAS</td>
<td>BSAS+Mullite</td>
<td>RE silicates or RE-Hf mullite</td>
<td>RE doped mullite-HfO₂ or RE silicates</td>
<td>Multi-component RE silicate systems</td>
<td>Multicomponent /self grown</td>
</tr>
<tr>
<td><strong>Bond Coat:</strong></td>
<td>Si</td>
<td>Si</td>
<td>Oxide+Si bond coat</td>
<td>HfO₂-Si-X, doped mullite/Si SiC nanotube</td>
<td>Optimized Gen IV HfO₂-Si-X bond coat 2700°F bond coats</td>
<td>RE-Si+X systems</td>
</tr>
<tr>
<td><strong>Thickness</strong></td>
<td>10-15 mil</td>
<td>10-15 mil</td>
<td>15-20 mil</td>
<td>10 mil</td>
<td>5 mil</td>
<td>1 - 3 mils</td>
</tr>
<tr>
<td><strong>Surface T:</strong></td>
<td>Up to 2400°F</td>
<td>2400°F</td>
<td>3000°F/2400CMC</td>
<td>2700°F/2400F CMC</td>
<td>3000°F</td>
<td></td>
</tr>
<tr>
<td><strong>Bond Coat T:</strong></td>
<td>Limited to 2462°F</td>
<td>Limit to 2642°F</td>
<td>Proven at 2600°F</td>
<td>Proven at 2600°F</td>
<td>2700°F (2011 goal)</td>
<td></td>
</tr>
</tbody>
</table>

### Challenges overcome by advancements:

- Improved phase stability, recession resistance of top coat
- Increased phase stability and toughness
- Advanced compositions & processing for thinner coatings, higher stability and increased toughness

**Evolution of Gen I-VI**
- **Gen I (EPM)**: 1995-2000 R&D Award
- **Gen II (UEET)**: 2000-2004
- **Gen III (UEET)**: 2000-2005 R&D Award (2007) coating turbine development
- **Gen IV (FAP)**: 2005-2011 R&D Award (2007) coating turbine development
- **Gen V (FAP - ERA)**: 2007 – 2012 to present
- **Gen VI-VII (FAP)**: 2009 – present Patent13/923,450 PCT/US13/46946

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

**Top Coat:**
- BSAS (APS)
- RE₂Si₂O₇ or RE₂Si₅ (APS)
- - (Hf,Yb,Gd,Y)₂O₃ - ZrO₂/HfO₂+RE silicates - ZrO₂/HfO₂+BSAS (APS and EBPVD)
- RE-HfO₂-Alumino silicate (APS and/or 100% EB-PVD)
- RE-HfO₂-X advanced top coat RE-HfO₂-graded Silica (EB-PVD)

**Interlayer:**
- RE-HfO₂/ZrO₂₂₆₂₆ aluminosilicate layered systems
- Nanocomposite graded oxide/silicate
- Gen IV interlayer not required (optional)

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

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

**Thickness:**
- 10-15 mil
- 10-15 mil
- 15-20 mil
- 10 mil
- 5 mil
- 1 - 3 mils

**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 2642°F
- Proven at 2600°F +;
- 2700°F (2011 goal)
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. $V^{1/2} P_{(H_2O)}^2/(P_{total})^{1/2}$

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

Combustion gas

Cooling gas

(a) Convective

(b) Convective with film-cooling
Fundamental Recession Issues of CMCs and EBCs - Continued

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

- Early generation coatings - EBC systems

Robinson and Smialek, J. Am. Ceram Soc. 1999
SiC/SiC CMC and EBC Recession Kinetics Determined for CMCs-EBCs in High Pressure Bruner Rig and Laser Steam Rig Testing

— Determined recession under complex, and realistic simulated turbine 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
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
- Advanced EBC recession met NASA Fundamental Aeronautics Project goals (2011)

Stability of selected coatings systems

Stability and temperature capability improvements through coating composition and architecture innovations

SiC, 20m/s, 6 atm; Robinson and Smialek, J. Am. Ceram Soc. 1999;
NASA EBC Developments under NASA Programs

• Focus on improving technology readiness level (TRL), 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 seal coat, with 2600°F+ HfO$_2$-Si and 2700°F+ rare-earth silicon (silicides) bond coats
  – Calcium Magnesium Alumino-Silicate (CMAS) resistance addressed for the composition developments

• Developed and evaluated EB-PVD/plasma spray hybrid turbine airfoil coatings
  – Efforts in developing turbine EBC coatings with Directed Vapor Technologies

• Developed Triplex Pro and DVC based combustor EBC processing with Sulzer/Oerlikon Metco and Praxair
  – Efforts in developing new EBC coating composition powders with Oerlikon Metco
  – Efforts in developing EBC bond coat powders with Oerlikon Metco
  – Efforts in EBCs and DVM/DVC coatings in collaborations with Praxair

• Processing optimizations for improved plasma sprayed coating powders composition controls and coating processing
  – Plasma sprayed EBC coatings using Triplex Pro (with Mitch Dorfman)
  – Optimizing/developing commercial HfO$_2$-Si based series bond coats with Sulzer/Oerlikon (with Mitch Dorfman)
  – NASA in-house Plasma spray – Physical Vapor Depositions process optimization

• Developing 2000°F capable oxidation/fretting wear resistant coatings (Ti-Si-Cr/Ta-CN systems and NiAl/NiAl+Cr/high toughness oxide/silicate systems)
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 erosion resistant 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

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

NASA Hybrid EBC on SiC/SiC

NASA Hybrid EBC on SiC/SiC

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

- NASA PS-PVD and PS-TF coating processing using Sulzer newly developed technology
  - High flexibility coating processing – PVD - splat coating processing at lo pressure (at ~1 torr)
  - High velocity vapor, non line-of-sight coating processing for complex-shape components
  - Emphasis on fundamental process and powder composition developments for EBC depositions

100 kW power, 1 torr operation pressure

NASA hybrid PS-PVD coater system

Processed coater system
Advanced EBC Coating Material Strength Evaluations

- EBC and bond coat constituents are designed with high strength to achieve the ultimate coating durability
  - Advanced EBC 150-200 MPa strength achieved at high temperature
  - Toughness 3-4 MPa m$^{1/2}$ achieved at room temperature
- HfO$_2$-Si based systems showed promising strength and toughness
  - 100 – 250 MPa strength
  - Toughness 2-3 MPa m$^{1/2}$ achieved (Room Temperature, compared to silicon 0.8-0.9 MPa m$^{1/2}$)
Advanced EBC Bond Coats for Turbine Airfoil and Combustor EBCs Developed

- 1500°C (2700°F) capable RESiO+X(Ta, Al, Hf, Zr …) EBC bond coat compositions and related composite coatings developed for combustor and turbine airfoil applications
- The bond coat systems demonstrated durability in the laser high heat flux rig in air and steam thermal gradient cyclic testing
- The bond coatings also tested in thermal gradient mechanical fatigue and creep rupture conditions

Selected Composition Design of Experiment Furnace Cyclic Test Series 1500°C, in air, Demonstrated 500hr durability

High heat flux cyclic rig tested Zr/Hf-RE-Si series EBC bond coats on the bond coated woven SiC/SiC CMCs at 1450°C in air and full steam environments
Advanced Bond Coats for Turbine Airfoil and Combustor
EBCs Developed - Continued

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

An oxidized bond coat after 1500°C 100 h creep testing
NASA Turbine Environmental Barrier Coating Testing Developments

- Advanced EBC top coats tested in coupons under laser heat flux cyclic rigs up 1700°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
High stability systems (Yb,Gd,Y+Hf) silicates, processed and down selected

Processing optimization also emphasized

Turbine EBCs: High pressure burner rig tested at 10 atm, 2650°F
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-sprayed 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
Understanding High Velocity Gas Flow Interactions – Columnar Structure and Toughness Considerations

- High velocity, high pressure gas impingements and shear force induced erosion in turbine engine flow condition can be of concern for low toughness coating systems
- High toughness, optimum coating density and architectures are required for durability

\[
\tau = \frac{F}{A} = C_D \left( \frac{1}{2} \rho V^2 \right)
\]

\[
F \quad \text{Shear Force} \\
A \quad \text{area} \\
C_D \quad \text{drag coefficient} \\
\rho \quad \text{density} \\
V \quad \text{velocity}
\]

For Ideal Gas:

\[
\rho = \frac{P}{R T}
\]

\[
\tau = C_D \left( \frac{1}{2} \frac{P}{R T} V^2 \right)
\]

If the coating is formed from columnar structures:

Bending stress at the base

\[
\sigma = \frac{M c}{I} = \frac{\tau \pi r^2 h}{\pi r^4} = \frac{4 \tau h}{r}
\]

\[
\sigma = \frac{4 h}{r} \left( C_D \frac{P V^2}{2 R T} \right) = \frac{2 h}{r} \left( C_D \frac{P V^2}{R T} \right)
\]

For a columnar structure with defect of size \( a \)

\[
K_I = \sigma \sqrt{\pi a f(a, r)}
\]

\[
K_I = \frac{2 h}{r} \left( C_D \frac{P V^2}{R T} \right) \sqrt{\pi a f(a, r)}
\]

Modeled parameters

<table>
<thead>
<tr>
<th>Drag Coefficient</th>
<th>( C_D )</th>
<th>0.4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>( P, \text{ psi} )</td>
<td>750</td>
</tr>
<tr>
<td>Velocity</td>
<td>( V )</td>
<td>1200 ( \text{m/sec} )</td>
</tr>
<tr>
<td>Temperature</td>
<td>( T, \text{ F} )</td>
<td>3000</td>
</tr>
<tr>
<td>Gas Constant</td>
<td>( R )</td>
<td>461.5 ( \text{J/Kg/K} )</td>
</tr>
<tr>
<td>Column Height</td>
<td>( h )</td>
<td>0.0002 ( \text{m} )</td>
</tr>
<tr>
<td>Column Radius</td>
<td>( r )</td>
<td>0.00001 ( \text{m} )</td>
</tr>
<tr>
<td>Stress</td>
<td></td>
<td>1.34E+08 ( \text{Pa} )</td>
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</table>
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

![Image of EBC coated tensile specimen](image1)

![Image of test setup](image2)
**Thermal Gradient Fatigue-Creep Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued**

- Advanced environmental barrier coatings – Prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 ksi at $T_{EBC} \approx 2700^\circ F$, $T_{CMC}$ interface \(\sim 2500^\circ F\)

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

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EBCs on Gen II CMC after 1000 hr fatigue testing

Hybrid EBCs on Gen II CMC after 100 hr low cycle creep fatigue testing
Advanced HfO$_2$-Si Bond Coats: Effects of Compositions on Strength and Creep Rates

- The HfO$_2$-Si composite coatings have high strength, and improved creep resistance at high temperatures
- Increased HfO$_2$-HfSiO$_4$ contents improve high temperature strength and creep resistance
Thermal Gradient Fatigue-Creep Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued

- Effects of temperature, load, heat flux and environments (steam and combustion air) for coated SiC/SiC CMC are being investigated
- EBC coated CMCs showed improved durability

![Graph showing creep rates and materials performance](image)

- 15 Ksi, thermal gradient EBC coated CMC (TCMC average 1315°C)
- 20 Ksi, thermal gradient EBC coated CMC (TCMC average 1315°C)
- 20 Ksi EBC coated CMC at 1315°C
- 20 Ksi EBC coated CMC at 1315°C Fatigue maximum stress 20 Ksi
- 20 Ksi EBC un-coated CMC at 1315°C

EBC coated CMCs showed improved durability over uncoated CMCs in terms of thermal gradient and creep rates. The conclusions drawn from this study suggest that EBC coated CMCs are more durable and can withstand higher stress levels compared to uncoated CMCs.

Effects of temperature, load, heat flux and environments (steam and combustion air) for coated SiC/SiC CMC are being investigated.
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

- 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

Stress gradients in Prepreg SiC/SiC CMC substrates under thermal gradient + mechanical creep loading
EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling - Continued

- EBC surface and delamination cracking under heat flux, thermal gradient tensile creep loading ($T_{\text{surface}} = 1482^\circ \text{C}; T_{\text{back}} = 1250^\circ \text{C}$)
- Delamination failure diving forces determined

- Uniform displacement case: effect of coating thickness

Finite Element Analysis (FEA) Modeling

EBC degradation under tensile creep loading
EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling – Bond Coat Stiffness Effect

- Delamination driving forces: uniform remote applied stress case, 0.300 mm thickness coating with ~ 0.06% total strain
- Effect of bond coat elastic modulus: E=150 GPa vs. E=50 GPa
- Strong bond coats expected to have less creep damage (lower strain energy release rate G for strong bond coats)

Solid Lines-strong bond coat
E=150 GPa EBC
E=150 GPa bond coat

Dashed Lines: Soft bond coat
E=50 GPa
Advanced EBCs designed with higher high temperature strength and stiffness to improve creep, fatigue, and cyclic durability.
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 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**

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

- Some minor coating spalling at hot streak impingement.
Summary

• Durable EBCs are critical to emerging SiC/SiC CMC component technologies
  — The EBC development built on a solid foundation from past experience
  — Advanced EBC processing and testing capabilities significantly improved, helping more advanced coatings to be realized for complex turbine components
  — A new series of EBC and bond coat compositions developed for meeting SiC/SiC CMC component performance requirements and long-term durability, establishing expanded scientific research areas
  — Better understood the coating failure mechanisms, and helping developing coating property databases and life models, aiming at developing higher stability, higher strength EBC and bond coats
  — Emphasized thin coating turbine and combustor EBC coating configurations, demonstrated component EBC technologies in simulated engine environments

  — Continue the coating composition and architecture optimization and developments to achieve 1482-1650°C capability, targeting uncooled and highly loaded components
    • The component and subelement testing and modeling

  — Understand EBC-CMC degradation and life prediction under complex thermal cycling, stress rupture/creep, fatigue, and environmental integrations
Future Directions and Opportunities

• High stability turbine airfoil and combustor coating system development continues to be a high priority
  – Emphasize advanced composition development, optimization, new processing and modeling capabilities
  – Reduce recession rates, improve the temperature stability and environment resistance, such as in CMAS environments
  – Significantly improve the interface stability and reduce reactivity
  – Low thermal conductivity

• Advanced environmental barrier coatings with significantly improved thermal and mechanical load capability is required
  – Significantly improve the coating strength and toughness
  – Better understand and improve creep, fatigue, and environment interactions
  – Design and demonstrate long-term high heat flux cyclic stability

• Materials and component system integration
  – Optimize and test coatings with components and SiC/SiC substrates
  – Enhance functionality with embedded sensing and self-healing capability
  – Integrate with virtue sensors and real time life predictions

• Laboratory simulated high heat flux stress, environment testing and life prediction methodology development, validating model developments