Creep Behavior of Hafnia and Ytterbium Silicate Environmental Barrier Coating Systems on SiC/SiC Ceramic Matrix Composites

Environmental barrier coatings will play a crucial role in future advanced gas turbine engines because of their ability to significantly extend the temperature capability and stability of SiC/SiC ceramic matrix composite (CMC) engine components, thus improving the engine performance. In order to develop high performance, robust coating systems for engine components, appropriate test approaches simulating operating temperature gradient and stress environments for evaluating the critical coating properties must be established. In this paper, thermal gradient mechanical testing approaches for evaluating creep and fatigue behavior of environmental barrier coated SiC/SiC CMC systems will be described. The creep and fatigue behavior of Hafnia and ytterbium silicate environmental barrier coatings on SiC/SiC CMC systems will be reported in simulated environmental exposure conditions. The coating failure mechanisms will also be discussed under the heat flux and stress conditions.
Creep Behavior of Hafnia and Ytterbium Silicate Environmental Barrier Coating Systems on SiC/SiC Ceramic Matrix Composites

Dongming Zhu*, Dennis S. Fox*, Louis J. Ghosn** and Bryan Harder*

* Durability and Protective Coatings Branch, Structures and Materials Division
** Applied Structural Mechanics Branch, Mechanical and Fluid Systems Division

NASA John H. Glenn Research Center
Cleveland, Ohio 44135, USA

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Revolutionary Ceramic Coatings and Composites Impact Turbine Engine Technology

- Advanced environmental barrier coatings and SiC/SiC CMC combustor and turbine airfoil component technologies are being developed for reduced cooling and NO\textsubscript{x} emission under NASA programs.

- Next generation environmental barrier coating-CMC development require more sophisticated laboratory testing to simulate turbine engine temperature and stress environments.

![Diagram of engine components](image)

- **Fuel Staging**
- **CMC Liners**
- **Instability Control**
- **Multipoint Injection**
- **Low emission combustor**

- Advanced core technologies – HPT first stage CMC vane with significantly reduce cooling requirements.
NASA Environmental Barrier Coating – CMC System Development: Temperature and Strength Goals

- Emphasize temperature capability, performance and durability requirements
- Help fundamental understanding, database and design tool development
- Increase the coating Technology Readiness Levels for engine applications

**Current NASA Program Goals**
- 3000°F* SiC/SiC CMC combustor and turbine vane EBC (20-30ksi**)
- 2800°F* ERA combustor EBC
- 2700°F* SiC/SiC CMC blade EBC (30-50ksi**)
- 2600°F* ERA turbine vane EBC

**Challenges:**
* Recession: <5 mg/cm² per 1000 hr (40-50 atm, Mach 1~2)
** Component strength and toughness requirements
Outline

— **Advanced creep and fatigue testing development for environmental barrier coating – CMC systems**
  - High heat flux thermal fatigue test rig – past experience
  - New heat flux mechanical test rigs
    - High heat flux tensile creep rupture rig
    - High heat flux ball-on-ring creep/fatigue test rigs
    - FEM analysis and model validation

— **Creep testing of candidate model environmental barrier coating systems for SiC/SiC CMC airfoils and combustors**
  - Ytterbium silicate
  - HfO$_2$ and HfO$_2$ Rare Earth Aluminosilicate EBCs
  - The coating degradation and delamination under thermal cycle and creep conditions

— **Preliminary failure mechanisms and modeling**

— **Summary**
Laser High Heat Flux Approach

– Turbine level high-heat-flux tests crucial for CMC coating system developments

• Existing high heat testing High power CO₂ laser high-heat-flux rig (up to 315 W/cm²)

Turbine: 450°F across 100 microns
Combustor: 1250°F across 400 microns
Real-Time Thermal Conductivity Measurements and Damage Monitoring

\[ k_{\text{ceramic}}(t) = \frac{q_{\text{thru}} \cdot l_{\text{ceramic}}}{\Delta T_{\text{ceramic}}(t)} \]

Where

\[ q_{\text{thru}} = q_{\text{delivered}} - q_{\text{reflected}} - q_{\text{radiated}} \]

and

\[ \Delta T_{\text{ceramic}}(t) = T_{\text{ceramic-surface}} - T_{\text{metal-back}} - \int_{0}^{l_{\text{bond}}} \frac{q_{\text{thru}} \cdot dl}{k_{\text{bond}}(T)} - \int_{0}^{l_{\text{substrate}}} \frac{q_{\text{thru}} \cdot dl}{k_{\text{substrate}}(T)} \]

8 µm pyrometer for \( T_{\text{ceramic-surface}} \)

Optional miniature thermocouple for additional heat-flux calibration

Two-color and 8 µm pyrometers for \( T_{\text{substrate-back}} \)
Typical Thermal Cyclic Behavior of EBC Systems

- Sintering and delaminations of coatings reflected by the apparent thermal conductivity changes

Cyclic Testing of 8YSZ/mullite/mullite+20wt%BSAS/Si on SiC/SiC:
- $T_{\text{surface}}$ 1482°C
- $T_{\text{interface}}$ 1175°C

Steady-State Testing of 8YSZ/on Rene N5 Superalloy:
- $T_{\text{surface}}$ 1371°C
- $T_{\text{interface}}$ 1163°C
Delamination in a High Heat Flux Thermal Cycling Condition

\[ \Delta T \approx 500^\circ C \]

Evans and Hutchinson model, Surface Coating Technology, 2007

\[ G = \frac{1}{6} \frac{(1+\nu_1)}{(1-\nu_1)} E_i h (\alpha_i (T_i - T_o))^2 \]

The FEM model
Experimental:
High-Heat-Flux Tensile Creep Rupture Test Rig

- Integrated with a High Power CO₂ laser system
- Allows very high temperature, high heat flux cooled thermal gradient testing under turbine blade stress conditions, and Long-term testing capability
- Accommodates conventional 6” tensile dog-bone and also other configuration specimens
- Specifically designed for CMC turbine airfoil system development
Experimental: New High-Heat-Flux Ball-on-Ring Creep and High Cycle Fatigue Test Rig

Integrated high power CO₂ laser and mechanical test rig allows
- High heat flux cooled thermal gradient testing under biaxial stress conditions
- High cycle fatigue (up to 100Hz) testing
- Accommodates 1” diameter, 2” diameter CMC disc test specimens and also various configuration subelements
- Designed for advanced CMC combustor material testing
EBC Processing Using Plasma-Spray (PS) and Plasma Spray-Physical Vapor Deposition (PS-PVD)

- Plasma spray YSZ/mullite/mullite+BSAS coated CMC system used for FEM model validation
- Plasma spray and/or plasma vapor HfO$_2$, HfO$_2$-Yb$_2$SiO$_5$/Yb$_2$Si$_2$O$_7$ environmental barrier coatings on Melt-Infiltrated (MI) CMC creep behavior studied

Plasma-spray processing of environmental barrier coatings

Hybrid PS-PVD coater system for PS-PVD coating processing
High-Heat-Flux Tensile Creep Rupture Test Rig

- Some early creep test examples under various stresses (34.5 – 103.5 MPa) at \( T_{\text{EBC surface}} \) 1320°C and \( T_{\text{CMC back surface}} \) 1200°C
- Large scatter in early creep tests due to CMC quality variability
- Environmental effects with high pressure burner rig exposures also studied
- Advanced EBCs demonstrated initial stress-strain resistance under thermal gradient stress rupture tests and helped protecting CMCs
Long-Term High Heat Flux Tensile Creep Rupture Tests
- Advanced EBC and MI CMC systems currently used for long-term creep testing
- Hafnia-ytterbium silicate based EBCs along with the hafnia-silicon bond coat on MI SiC/SiC specimen completed 600 hr creep rupture testing at 69 MPa (10 ksi) stress, and $T_{\text{coating surface}}$ 1350°C and $T_{\text{CMC back surface}}$ 1200°C
- Degradation monitored by through-thickness thermal conductivity measurements
- Creep rupture testing for 1000 hours at 69 MPa (10ksi) and 103.5 (15ksi) to meet project goals
- Thermal cycling fatigue testing will be incorporated
Long-Term High Heat Flux Tensile Creep Rupture Tests
- Temperature and system thermal conductivity monitoring
FEM Analysis of High Heat Flux Ball-on-Ring Creep and High Cycle Fatigue Test Rig

- FEA models used to help understand the heat transfer and axial displacements of the 1” disc specimen under a Ball-on-Ring test
- Tensile strains induced on the EBC side

Temperature distribution

Axial total displacement under heat flux testing
Modeled CMC Test Stress Conditions in a Ball-on-Ring Test Rig

Radial Stress Profile

Hoop Stress Profile

Axial Stress Profile
Elastic Stress Distribution in a Disc Test Specimens under Ball-on-Ring Tests

The graph illustrates the distribution of radial stress and strain as a function of axial distance. The x-axis represents the axial distance in millimeters, while the y-axes show the radial stress in MPa and the radial strain in m/m. The graph includes data points for the substrate and EBC layers, indicating the stress and strain values at different axial distances.
New High Heat Flux Ball-on-Ring and Ring-on Ring Creep and High Cycle Fatigue Test Rig - Continued

- FEA models used to help understand the heat transfer and axial displacements of the 1” disc specimen under a Ball-on-Ring test

![Axial displacement vs. Radial strain](image1)

![Axial total creep displacement under heat flux testing](image2)
The YSZ/Mullite/Mullite+BSAS/Si EBC Degradation In-Situ Monitored in a Ball-on-Ring Creep Test

- Tested $T_{\text{surface}}$ 1482°C and $T_{\text{interface}}$ 1250°C
- Constant tested load 445 N
- Excellent correlations between thermal conductivity and creep strain response due to coating failure
Creep model validated for the EBC-CMC system (at 200 MPa)
Examples of a Ytterbium Silicate EBC – SiC-SiC CMC Testing

- High creep system resulted in the material system early failure
- Real time monitoring through the conductivity and creep strain changes
Examples of a Ytterbium Silicate EBC – SiC-SiC CMC
Testing - Continued

- Real time monitoring through the conductivity and creep strain changes
- Cracking-delamination rates can be determined for EBC
- Creep rates for the CMC system
Examples of HfO₂ and Hf-RE-Aluminum Silicate EBC Testing

- Real time monitoring through the conductivity and creep strain changes
- Less degradation observed for the EBC at similar substrate creep rates
Examples of HfO₂ and Hf-RE-Aluminum Silicate EBC Testing

- Real time monitoring through the conductivity and creep strain changes
- Less degradation observed for the EBC at similar substrate creep rates

![Graph showing creep displacement and load over time](graph.png)
Failure Morphologies of Ytterbium Silicate/Si EBC system
- Coating surface cracking, interface reaction and delamination after testing
Failure Morphologies of HfO$_2$ - Based EBC

- Some coating surface cracking, perhaps crack healing observed after testing
Modeling of EBC Failure Mechanisms in Tensile Creep and Ball-on-Ring Flexural Creep Tests: Creep Induced Mixed Mode Delaminations

Mode I Opening Stress

Mode II Shear Stress

Temperature

ε_ε=0.06\% \, m/m

ε_p=0.06\%-1.0\% \, m/m

1,350 \, ^\circ\!C

2W
4 \, mm

1,200 \, ^\circ\!C

0.381 \, mm

2 \, mm
Delamination under Cyclic Fatigue Load

\[ \ln(\frac{da}{dN}) \]
\[ v \]
\[ \ln(\Delta K) \]

average slope 4.6

Zhu, Choi, Miller, Surface Coating and Technology, 2004
Summary

• Advanced high heat flux tensile rupture and ball-on-ring rigs established for simulated EBC-CMC testing
  — High temperature comprehensive testing capability
  — Real time coating degradation monitoring
  — FEM models helped understand the testing

• Initial creep and fatigue behavior evaluated for EBC systems from plasma spray and plasma spray vapor deposition
  — Coating failure mechanisms identified and modeled

• Advanced EBC demonstrated initial capability to resist thermal and mechanical stresses likely to be encountered in a turbine component
  — High strength and high stability coating systems are still being developed

• Fatigue behavior of EBC-CMC systems being investigated systematically to understand time and cycle dependent fatigue behavior
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Modeling of EBC Failure Mechanisms in Tensile Creep and Ball-on-Ring Flexural Creep Tests: Creep Induced Mixed Mode Delaminations: Elastic Solution Case

- More severe damage under larger creep strains needs a better understanding coating properties and creep stress relaxation