Microstructure Evolution and Durability of Advanced Environmental Barrier Coating Systems for SiC/SiC Ceramic Matrix Composites

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NASA EBC and CMC System Development

- Emphasize temperature capability, performance and *long-term* durability
  - Highly loaded EBC-CMCs
  - 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

Temperature Capability

2800°F combustor TBC
2500°F Turbine TBC
2700°F SiC/SiC thin turbine EBC systems for CMC airfoils
2700°F (1482°C) Gen III SiC/SiC CMCs
2000°F (1093°C), PtAl and NiAl bond coats
2400°F (1316°C) Gen I and Gen II SiC/SiC CMCs
3000°F SiC/SiC CMC airfoil and combustor technologies

Increase in ΔT across T/EBC

Ceramic Matrix Composite
Single Crystal Superalloy
Gen II – Current commercial
Gen III
Gen. IV
Year
Development Objective: Develop advanced 2700°F+ capable bond coat and EBC systems with high strength

Approaches:

- Fundamental studies of environmental barrier coating materials and coating systems, stability, temperature limits and failure mechanisms
- Focus on high performance high stability patented cluster HfO$_2$ and ZrO$_2$ -RE$_2$O$_3$-SiO$_2$/RE$_2$Si$_{2-x}$O$_{7-2x}$ environmental barrier systems
  - Controlled silica content and transition element and rare earth dopants to improve EBC stability and toughness
  - Significantly reduce diffusion, grain growth, mechanical strength and toughness with multicomponent systems
  - Develop HfO$_2$-Si based + X (dopants)
  - Develop prime-reliant Rare Earth Si alloys and composites for integrated EBC-bond coat systems
- Develop advanced NASA high toughness, compositions and processing
  - Achieving high toughness and erosion resistance
  - Achieving high stability and recession resistance
  - Improving the resistance to CMAS and Volcano ash deposits
Outline

• Advanced EBC and Rare Earth – Silicon based 2700°F+ capable bond coat developments
  - Development approaches
  - Oxidation resistance
  - Cyclic and thermomechanical durability

• Microstructural and phase composition evolution of an environmental barrier coating (EBC) system
  - Consisting of a multicomponent rare earth silicate EBC, along with YbGdSi 2700°F (1482°C) capable bond coat
  - Tested in high heat flux tensile rupture, fluxture fatigue, and furnace cyclic tests up to 500 hours at 2700°F (1482°C)
  - Examine Microstructure changes after thermomechanical and furnace cyclic testing
  - EDS - WDS Composition analysis Comparisons

• Summary and conclusion
Development Strategy for 2700°F EBC Systems

— Modify silicon with rare earth, zirconium, hafnium dopants to increase its melting point and developing slower growing protective scales and refractory silicates
  - Control oxygen content and EBC-CMC interface oxygen partial pressure for improved protection and stability

— Composite bond coat systems with refractory oxides and silicates to reinforce silicon containing bond coat matrix

— Composite bond coat systems with refractory oxide or silicate matrix, with silicon containing bond coat inter-phases

— Develop rare earth metal -, zirconium -, hafnium – silicon systems and the silicide containing systems for bond coats with engineered grain boundary phases
Advanced High Temperature and 2700°F+ Bond Coat Development

- NASA advanced Top Coat Development approach:
  - Advanced compositions ensuring high strength, high stability, high toughness
  - Bond coat systems for prime reliant EBCs; capable of self-healing

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

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

HfO₂-Si based and minor alloyed systems for improved strength and stability

Advanced 2700°F+ Rare Earth - Bond Coat systems

HfO₂-Si systems

Other systems
Furnace Cycle Test Results of Selected RESi and ZrSi + Dopant Bond Coats
- Testing in Air at 1500°C, 1 hr cycles

- Some initial multi-component systems showed excellent furnace cyclic durability at 1500°C
- FCT and steam tests also performed for RESiO-Hf systems
- Generally good correlation between FCT and oxidation resistance

An example of cross-section TGA tested specimen

Oxidation kinetics
The Environmental Barrier Coating System

- Alternating layered HfO$_2$-Rare Earth silicate EBC for fundamental stability studies
- 2700F capable Yb-YbO based bond coat
- Coated onto SiC/SiC CMC substrates using EB-PVD
Environmental Barrier Coating System

- YbGdSi(O) (+Hf) Bond Coat + multi-component EBC Top Coat on woven SiC/SiC CVI-MI SiC/SiC CMC
- Creep testing conducted with 15 ksi load and laser thermal gradient at 0.15% total creep strain, bond coat at up to 2700°F (1482°C)

The bond coat remains mostly intact after 100 hr creep testing (CMC fracture) with 2700°F coating surface temperature, except some debond near the top coat/bond coat region due to high Si segregation or processing defective regions.
The Flexural Fatigue Tested Environmental Barrier Coating Systems

- Strength and Fatigue cycles in laser heat flux rigs in tension, compression and bending
- Fatigue tests at 3 Hz, 2600-2700°F, stress ratio 0.05, surface tension-tension cycles

- Flexural fatigue tests with 15 Ksi (138 MPa) stress amplitude loading

Creep-fatigue durability tests

Achieved long-term fatigue lives (near 500 hr) with EBC at 2700°F

Examples of fatigue test EBC systems on Tyrannohex SA SiC composites (Ube Industries, Inc.)
- YbGdSi(O) (+Hf) Bond Coat region

100 hr test Creep Rupture Test

EDS A
EDS B
EDS C
EDS D
EDS E
EDS F

Oxygen content increases
SEM – EDS Analysis of the Tensile Ruptured Tested Environmental Barrier Coating System - Continued

Hf(O)-HfSi₂(O) bond coat region, 100hr

EDS A

EDS B

EDS C – higher silicon content
The Flexural Fatigue Tested Environmental Barrier Coating System

- Ytterbium containing bond coat help self-healing the composite fatigue cracking

460hr, 2600-2700°F fatigue tested, bond coat only

EDS A

EDS B

EDS C
The Flexural Fatigue Tested Environmental Barrier Coating System - Continued

- Ytterbium containing bond coat help self-healing the composite fatigue cracking

460hr, 2600-2700F fatigue tested
The Long-Term Furnace Cyclic Tested Environmental Barrier Coating System: Rare Earth doped HfO$_2$ and Rare Earth Silicates Showed Compatibility and Stability

- 1500°C, 500 hr, 1 hr cycles, in air

HfO$_2$-(Yb,Gd,Y)$_2$O$_3$-(SiO$_2$)

HfO$_2$-(Yb,Gd,Y)$_2$O$_3$-SiO$_2$
The Long-Term Furnace Cyclic Tested Environmental Barrier Coating System: Rare Earth doped HfO$_2$ and Rare Earth Silicates Showed Compatibility and Stability - Continued

- 1500°C, 500 hr, 1 hr cycles, in air
EDS (Si Drift Detector) and WDS Comparisons Showed Good Agreements in the Composition Analysis

- 1500°C, 500 hr, 1 hr cycles, in air

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**WDS 10 points to obtain the average composition of the L region**

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EDS (Si Drifting Detector) and WDS Comparisons Showed Good Agreements in the Composition Analysis - Continued
- 1500°C, 500 hr, 1 hr cycles, in air

WDS 10 points to obtain the average composition of the N region

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EDS (Si Drifting Detector) and WDS Comparisons Showed Good Agreements in the Composition Analysis - Continued

- 1500°C, 500 hr, 1 hr cycles, in air

WDS 4 points to obtain the average composition of the N region

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Summary and Conclusions

- Environmental barrier coatings with YbGd-HfSi(O) based bond coat, and HfO$_2$ – multicomponent Rare Earth silicate top coat were tested on SiC/SiC ceramic matrix composites for initial durability studies.
- The coatings showed excellent oxidation resistance in O$_2$ and air testing environments, adhesion and good protection for SiC/SiC CMCs.
- The initial silicon content range of the Rare Earth-Silicon coatings was down-selected, multicomponent systems designed and demonstrated for further improved stability.
- The rare earth – silicon based coatings showed 2700°F or 1500°C operating temperature viability and durability on SiC/SiC ceramic matrix composites, the EBC-CMC system microstructure and phase changes were investigated.
- The rare earths, hafnium and silica showed wide range solubility, and composition ranges of EBC materials are being optimized for coating stability and performance.
- The extensive studies of the EDS and WDS composition analyses of the EBC system showed good agreements:
  - WDS may be more sensitive to light elements;
  - Field emission gun SEM Silicon drift detector EDS has spatial resolution advantages.
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