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

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Materials Science and Technology Conference,
Salt Lake City, Utah
October 24-27, 2016
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
NASA Advanced Environmental Barrier Coating Technology Development

- **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

- HfO$_2$-Si systems
- Advanced 2700°F bond coat systems: RE-Si based systems
- Advanced 2700°F+ Rare Earth - Bond Coat systems

Other systems

High strength, high stability reinforced composites: HfO$_2$-Si and a series of Oxide-Si systems

HfO$_2$-Si based and minor alloyed systems for improved strength and stability
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

Oxidation kinetics
An example of cross-section TGA tested specimen
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

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

(Yb,Gd,Y)$_2$Si$_{2-x}$O$_{7-2x}$

The bond coat region
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

Examples of fatigue test EBC systems on Tyrannohex SA SiC composites (Ube Industries, Inc.)
SEM – EDS Analysis of the Tensile Rupture Tested Environmental Barrier Coatings System

- 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

EDS A

EDS B

EDS C – higher silicon content

Hf(O)-HfSi₂(O) bond coat region, 100hr
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 HfO₂ and Rare Earth Silicates Showed Compatibility and Stability

- 1500°C, 500 hr, 1 hr cycles, in air

HfO₂-(Yb,Gd,Y)₂O₃-(SiO₂)

HfO₂-(Yb,Gd,Y)₂O₃-SiO₂
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

WDS 10 points to obtain the average composition of the L region
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
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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**PROBE DATA-average 4pts**
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.
Acknowledgements

The work was supported by NASA Fundamental Aeronautics Programs, and Aeronautical Science Project.

The authors are grateful to
- Dr. Kang N. Lee for helpful Discussions.
- Ron Phillips and Ralph Pawlik for their assistance in mechanical testing;
- Don Humphrey, John Setlock and Michael Cuy for assisting Thermogravimetric analysis (TGA) and furnace oxidation tests;
- Sue Puleo and Rick Rogers for X-ray analysis