NASA/TM—2005-213444

Advanced Environmental Barrier Coatings Development for Si-Based Ceramics

Dongming Zhu
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Sung R. Choi
Ohio Aerospace Institute, Brook Park, Ohio

Raymond C. Robinson
QSS Group, Inc., Cleveland, Ohio

Kang N. Lee
Cleveland State University, Cleveland, Ohio

Ramakrishna T. Bhatt
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Robert A. Miller
Glenn Research Center, Cleveland, Ohio

March 2005

The NASA STI Program Office . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the Lead Center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized databases, organizing and publishing research results . . . even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI Program Home Page at ***http://www.sti.nasa.gov***

- E-mail your question via the Internet to help@sti.nasa.gov

- Fax your question to the NASA Access Help Desk at 301–621–0134

- Telephone the NASA Access Help Desk at 301–621–0390

- Write to:
  NASA Access Help Desk
  NASA Center for AeroSpace Information
  7121 Standard Drive
  Hanover, MD 21076

NASA/TM—2005-213444

Advanced Environmental Barrier Coatings Development for Si-Based Ceramics

Dongming Zhu
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Sung R. Choi
Ohio Aerospace Institute, Brook Park, Ohio

Raymond C. Robinson
QSS Group, Inc., Cleveland, Ohio

Kang N. Lee
Cleveland State University, Cleveland, Ohio

Ramakrishna T. Bhatt
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Robert A. Miller
Glenn Research Center, Cleveland, Ohio

Prepared for the
Third Annual Workshop on Environmental Barrier Coatings and Enhanced Silicon Nitride for Gas Turbines
cosponsored by the Department of Energy's Office of Distributed Energy, Air Force Research Laboratory, and National Aeronautics and Space Administration
Nashville, Tennessee, November 17–18, 2004

National Aeronautics and
Space Administration

Glenn Research Center

March 2005

Available from

NASA Center for Aerospace Information
7121 Standard Drive
Hanover, MD 21076

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22100

Available electronically at http://gltrs.grc.nasa.gov

Advanced Environmental Barrier Coatings Development for Si-Based Ceramics

Dongming Zhu
U.S. Army Research Laboratory
Glenn Research Center
Cleveland, Ohio 44135

Sung R. Choi
Ohio Aerospace Institute
Brook Park, Ohio 44142

Raymond C. Robinson
QSS Group, Inc.
Cleveland, Ohio 44135

Kang N. Lee
Cleveland State University
Cleveland, Ohio 44115

Ramakrishna T. Bhatt
U.S. Army Research Laboratory
Glenn Research Center
Cleveland, Ohio 44135

Robert A. Miller
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Abstract

Advanced environmental barrier coating concepts based on multi-component HfO_2 (ZrO_2) and modified mullite systems are developed for monolithic Si_3N_4 and SiC/SiC ceramic matrix composite (CMC) applications. Comprehensive testing approaches were established using the water vapor cyclic furnace, high pressure burner rig, and laser heat flux steam rig to evaluate the coating water vapor stability, cyclic durability, radiation, and erosion resistance under simulated engine environments. Test results demonstrated the feasibility and durability of the environmental barrier coating systems for 2700-3000 °F monolithic Si_3N_4 and SiC/SiC CMC component applications. The high-temperature-capable environmental barrier coating systems are being further developed and optimized in collaboration with engine companies for advanced turbine engine applications.

Advanced Environmental Barrier Coatings Development for Si-Based Ceramics

Dongming Zhu [1*], Sung R. Choi [3], Raymond C. Robinson[1], Kang N. Lee [1], Ram Bhatt [2*], Robert A. Miller [1]

[1]Durability and Protective Coatings Branch, [2]Ceramics Branch, Materials Division
[3]Life Prediction Branch, Structures Division
*ARL Vehicle Technology Directorate
NASA Glenn Research Center, Cleveland, Ohio

This work was supported by NASA Ultra-Efficient Engine Technology (UEET) and DoD Integrated High Performance Turbine Engine Technology (IHPTET) Programs

Third Environmental Barrier Coatings Workshop, Nashville, Tennessee
November 17-18, 2004

Objectives

— **Advanced EBC concept and material systems for monolithic Si_3N_4 and SiC/SiC ceramic matrix composite (CMC) applications**

— **Testing facilities and approaches**

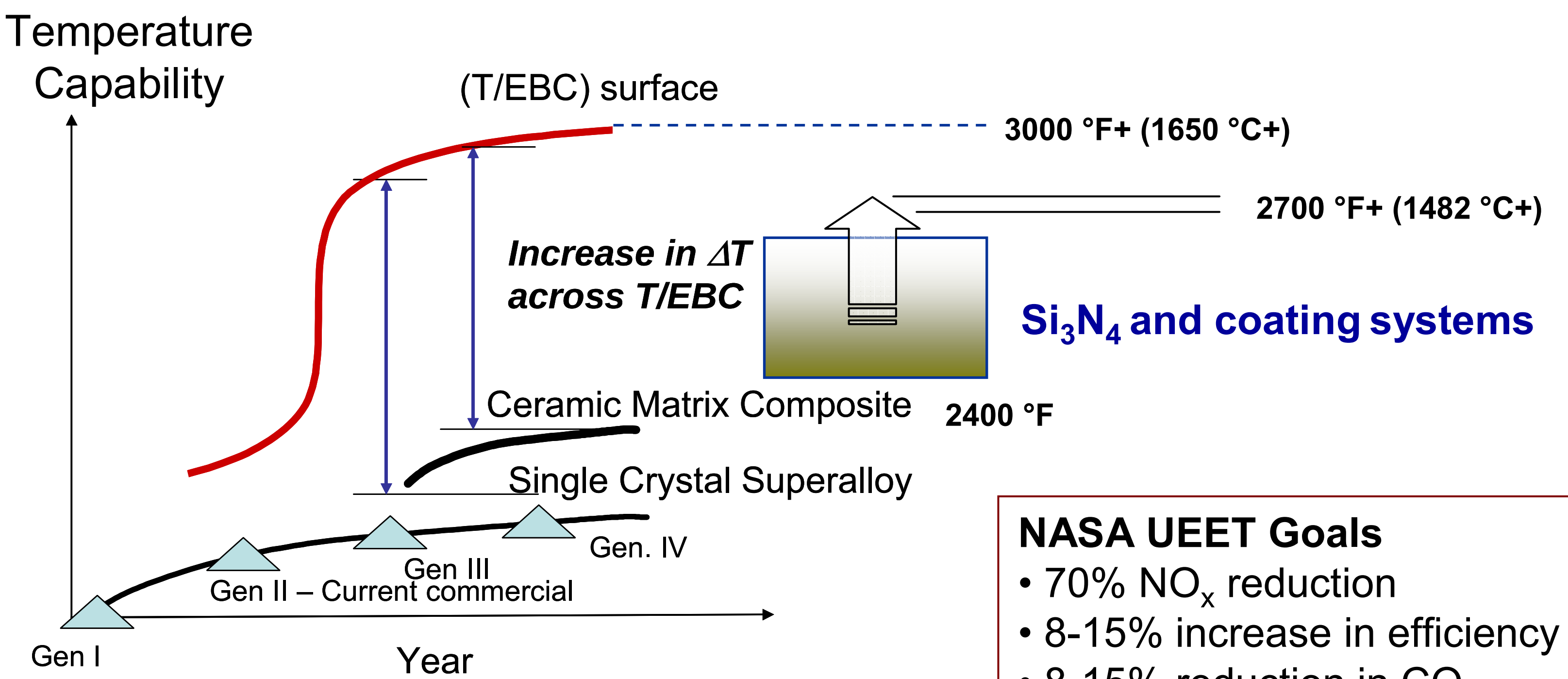
— **Current coating development status**

- Current coating limitations
- Materials concept/system selections
- Advanced coating feasibility demonstrations
- Temperature capability and cyclic durability
- Thermal radiation and erosion resistance

— **Summary and conclusions**

Motivation

— **Advanced ceramic coating technology will significantly increase ceramic component temperature capability and usability**



NASA UEET Goals
- 70% NO_x reduction
- 8-15% increase in efficiency
- 8-15% reduction in CO_2

Introduction

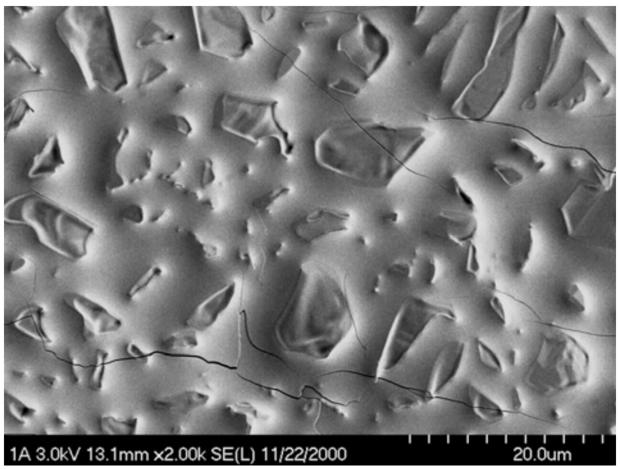
- **Ceramic environmental barrier coatings are critical for Si-based ceramics**

  - Current EBCs are limited in their temperature capability and water vapor stability
  - Coating feasibility and durability are more of concern with higher operating temperatures

- **Advanced coating concepts and technologies required for next generation ceramic engine development**

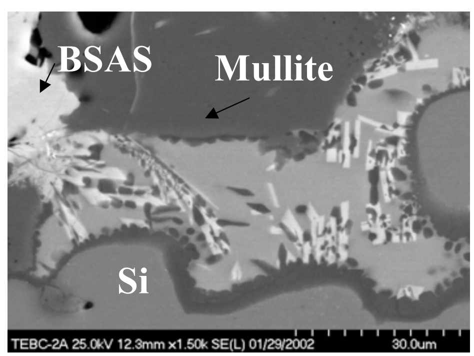
BSAS/BSAS+Mullite Based EBCs

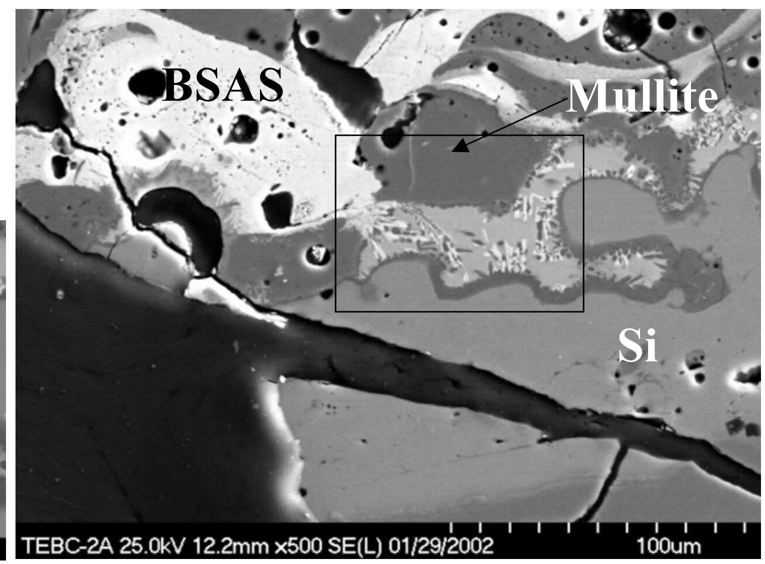
- The glass phase formation after 100 hour testing at the surface temperature of 1482 °C
- Water vapor recession for current EBC systems in higher temperature, high gas velocity, corrosive (e.g., CMAS) conditions
- The Si bond coat also greatly limited the coating temperature capability to less than 1350 °C



Top surface

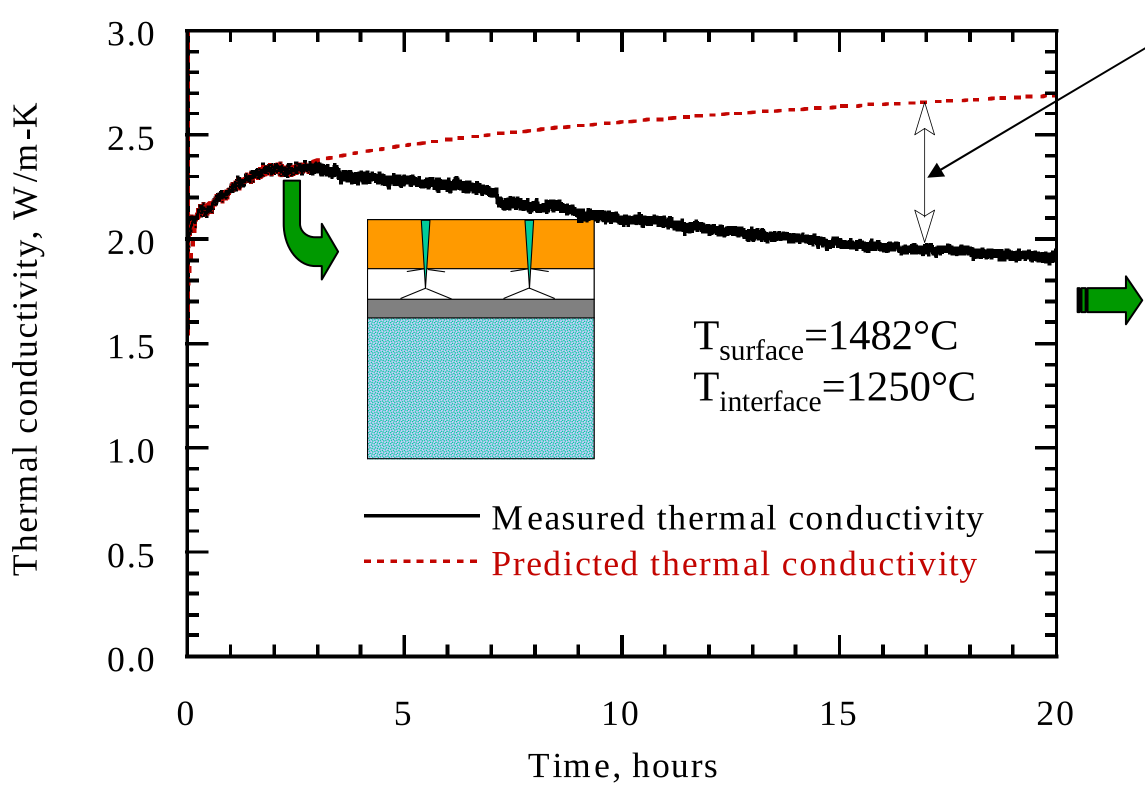
Interface





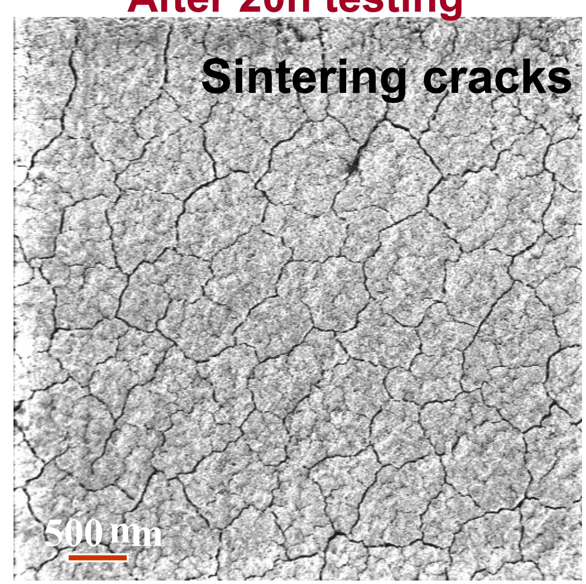
ZrO_2-8wt%Y_2O_3/Mullite+BSAS/Si System under High Temperature Thermal Gradient Testing

— Higher temperature capable ZrO_2-8wt%Y_2O_3/mullite+BSAS system tested at $T_{surface}$ 1482 °C (2700 °F) and $T_{interface}$ 1300 °C
— Coating delaminated at temperature due to sintering/creep
— CTE mismatch demonstrated also be an issue



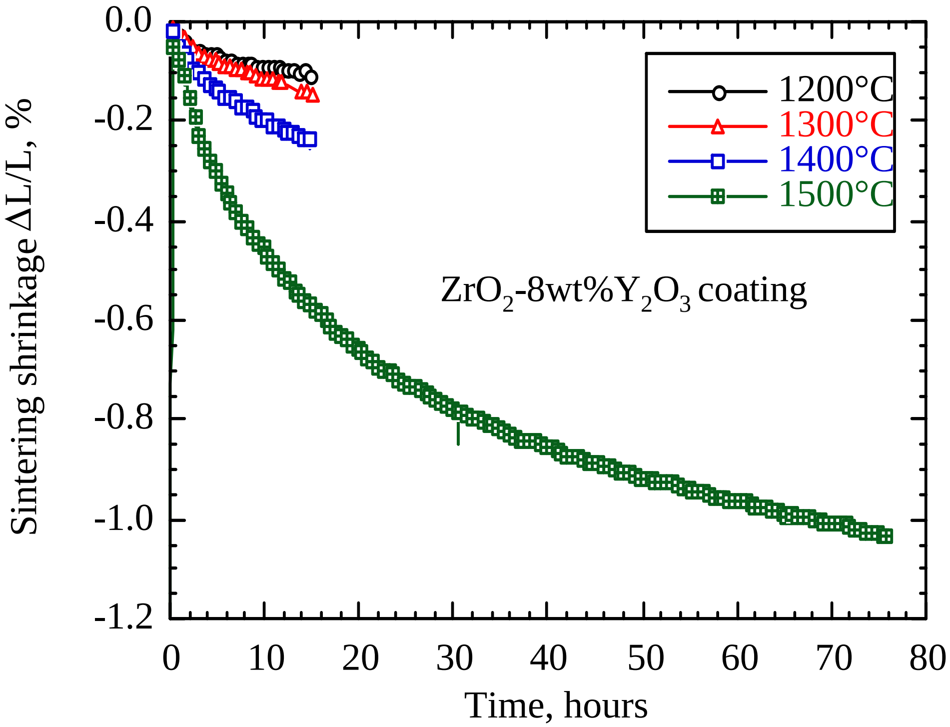
Conductivity reduction due to sintering cracking induced delamination cracking

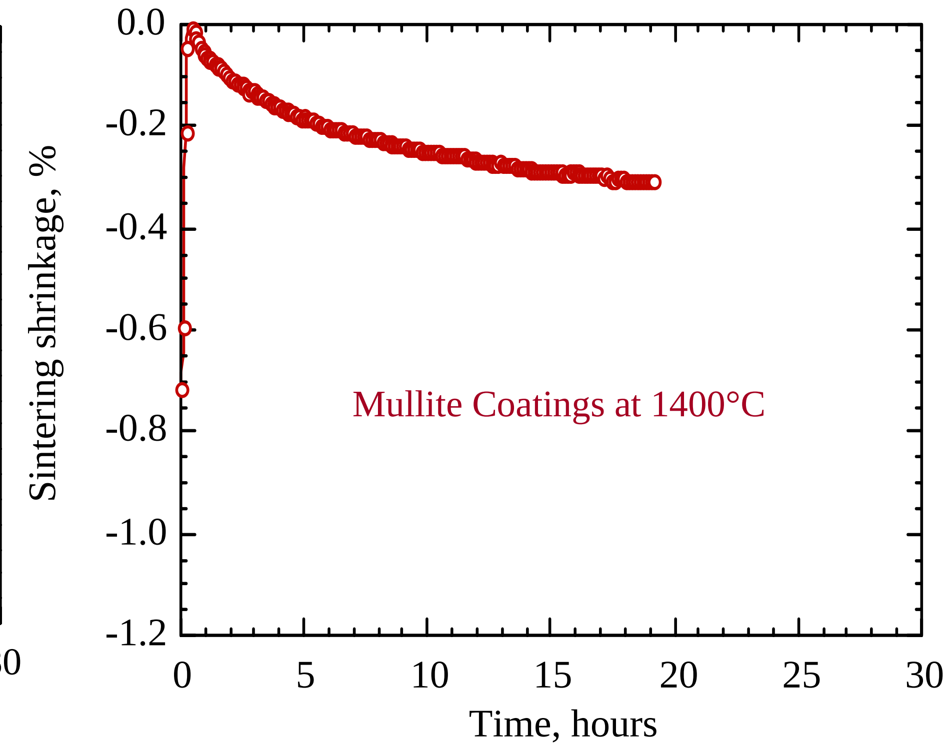
After 20h testing



Sintering Behavior and Limitations of Current ZrO_2-8wt%Y_2O_3 and Mullite Coatings

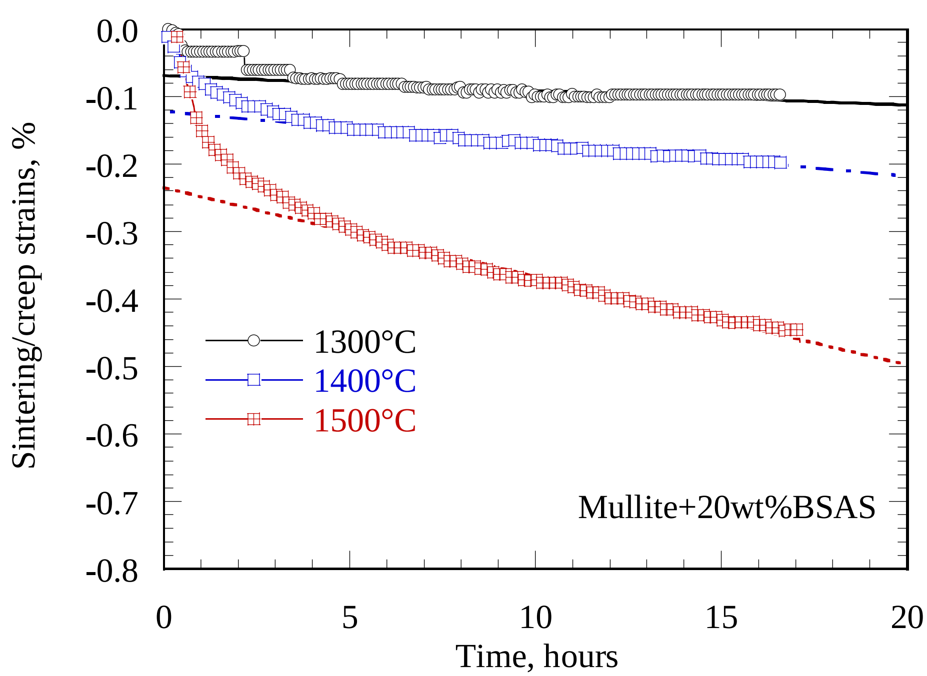
- Sintering resistance of ZrO_2-8wt%Y_2O_3 coating is of concern
- Conventional mullite still has fast sintering/creep rates, low strength, and weak bonding to the ceramic substrates

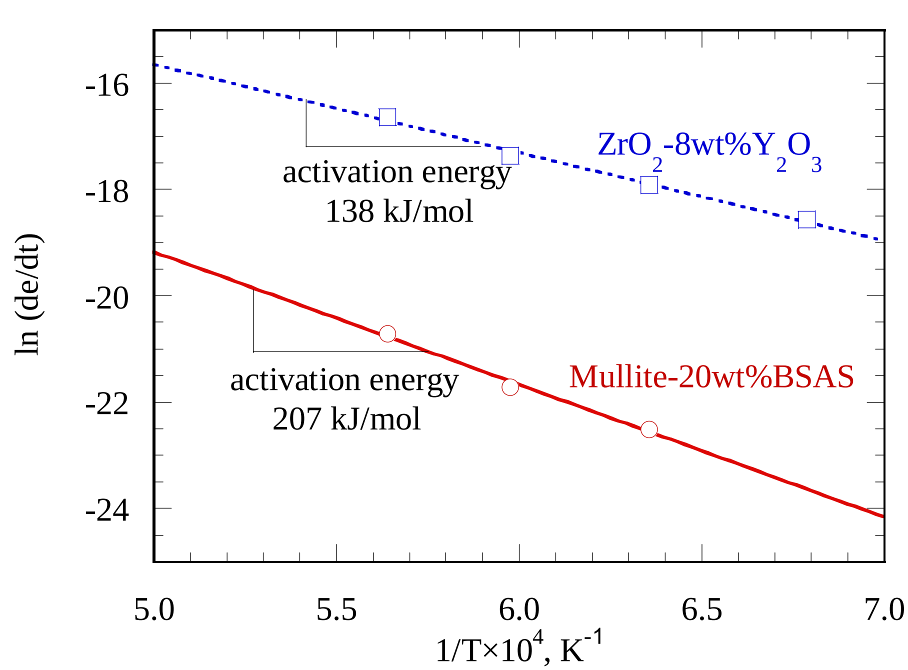




Sintering and Creep Behavior of Relatively Dense Plasma-Sprayed Mullite-20wt%BSAS

— Sintering/creep rate increases with temperature but decreases with time
— Near steady-state rates (5-20 hrs) used for temperature dependence data





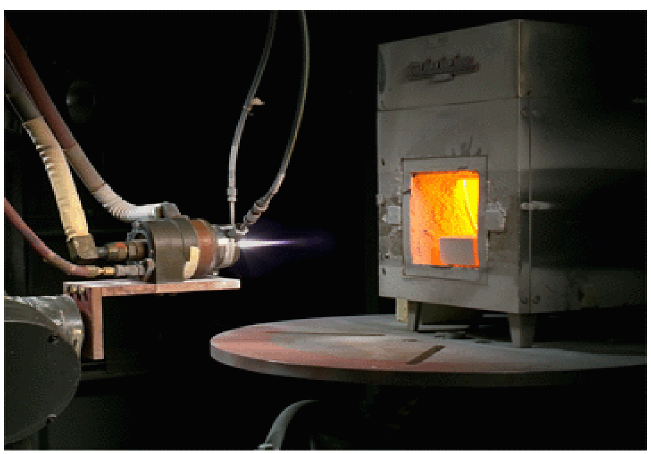
Advanced Coating Development Strategies

– **Advanced Environmental Barrier Coating System Concept**

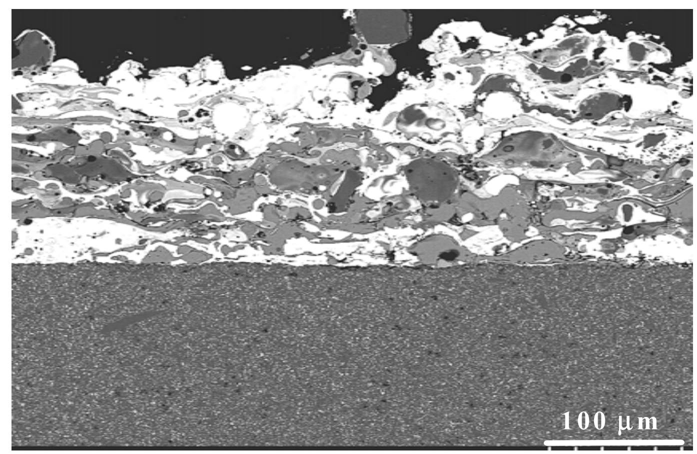
- An advanced ZrO_2 or HfO_2 top layer for high temperature stability and excellent water vapor recession and corrosion resistance
- Graded modified mullite interlayer and environmental barrier for enhanced protection
- Advanced ZrO_2/HfO_2, and/or mullite composite bond coats for excellent adhesion, strength and stability

Advanced Environmental Barrier Coatings for Si_3N_4 Applications

- **Multi-layered, rare earth and silicon doped HfO_2/mullite 2700 °F environmental barrier coating systems developed:**
  - Advanced low expansion doped HfO_2 used for high stability top layer
  - Modified mullite as the interlayer and environmental barrier
  - Doped HfO_2 or mullite 2700 °F+ capable bond coats
- **Plasma-spray technique used for coating processing**
- **Cyclic furnace, laser steam heat flux rig and high pressure burner rig used for coating evaluation**



Plasma-spray processing of Environmental barrier coating



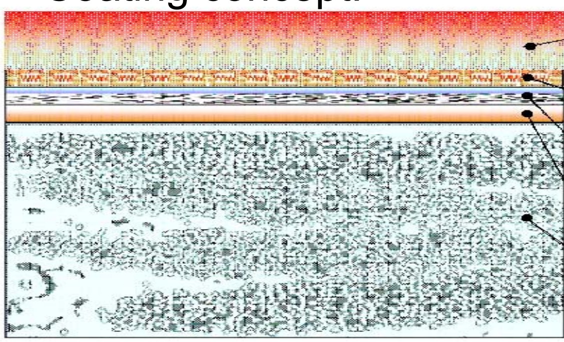
A 2700 °F capable coating system for Si_3N_4

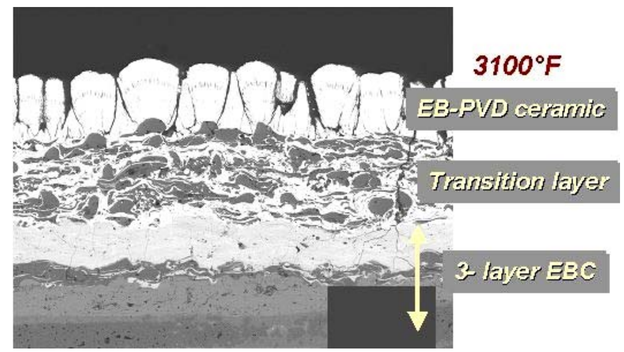
Advanced Environmental Barrier Coatings for SiC/SiC CMC Applications

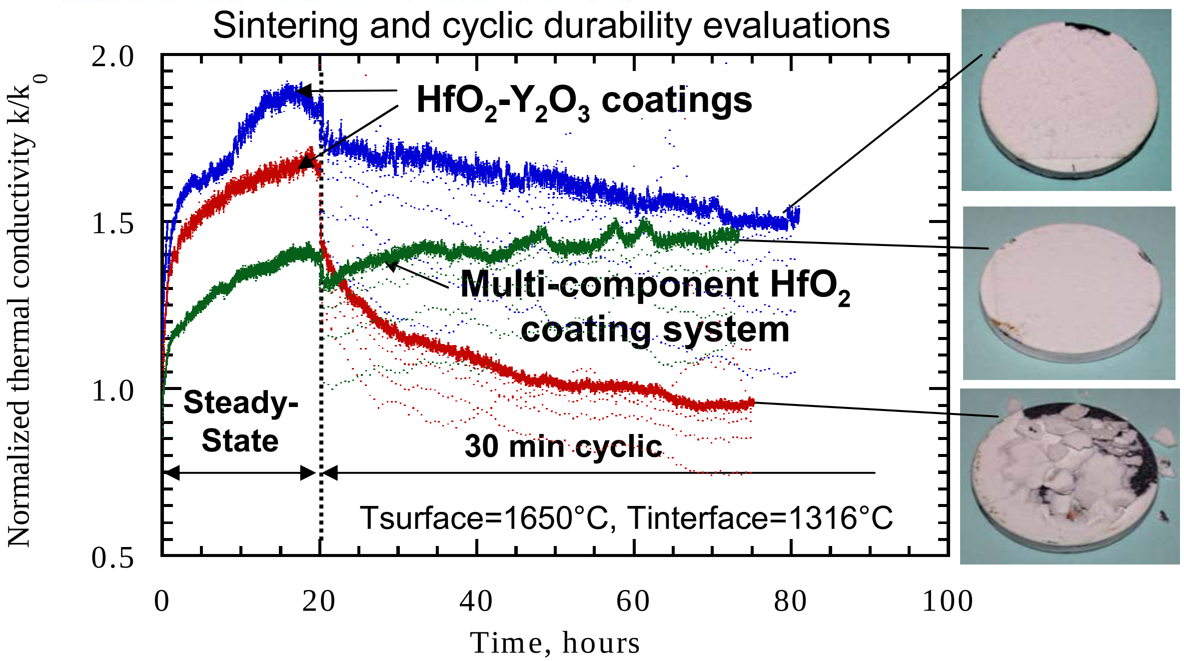
— The multicomponent hafnia(zirconia) coating/modified mullite systems demonstrated excellent cyclic durability and radiation resistance at 1650 °C (3000 °F)

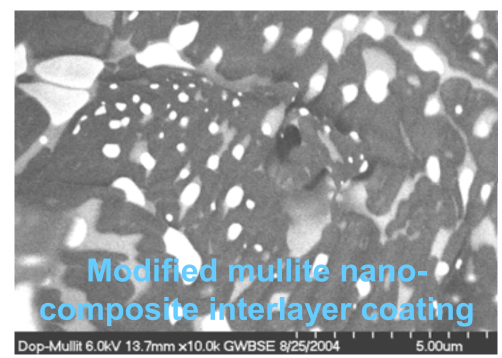
— Advanced ceramic bond coats also developed for CMCs

Coating concept:







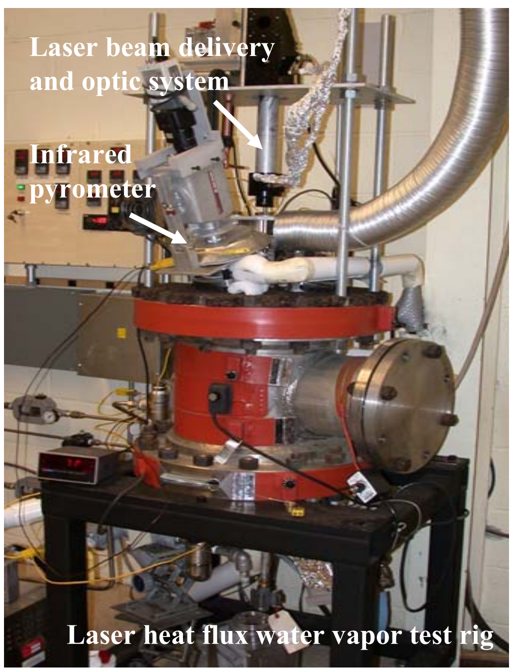


Also Alternating Composition Layered Coatings (ACLC) for 3000 °F applications

NASA Laser Heat Flux Rig Testing in Water Vapor Environments

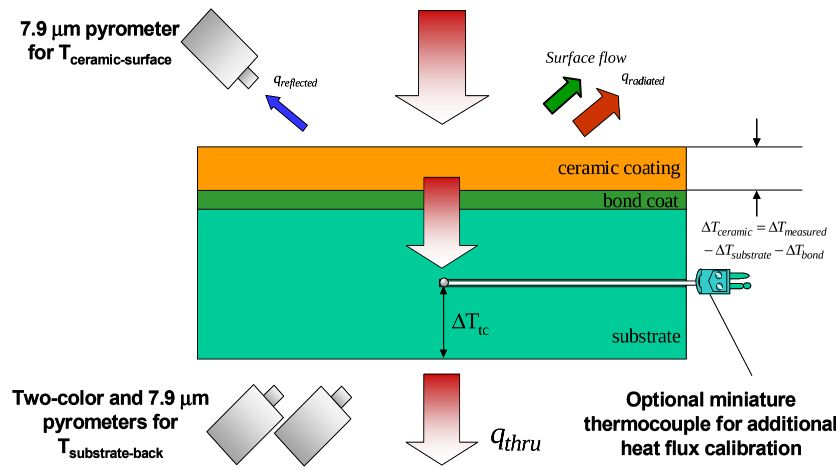
– **Laser heat flux steam rig**

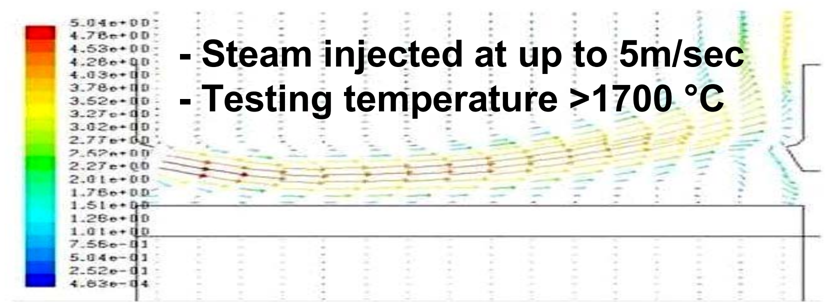
- Precise control of heat flux and temperatures of test specimen
- Automated control of chamber temperature and steam environments
- High temperature and high-heat-flux testing capabilities
- Innovative “micro-steam environment” concept allows high vapor pressure (100% water vapor), velocity (5 m/sec) and temperatures as required
- Real time specimen health monitoring capability



Specimen holder and water vapor jets

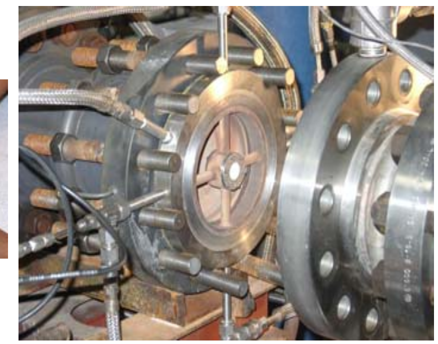

Specimen under testing





High Pressure Burner Rig (HPBR) for Ceramic Coatings Testing

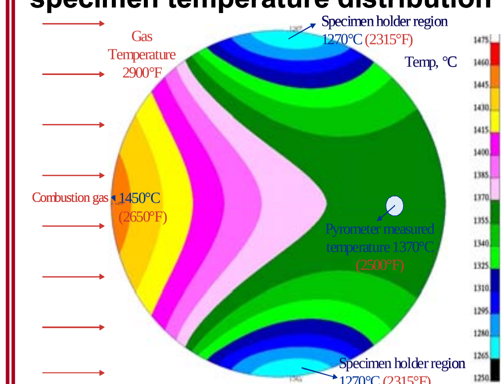
- **High pressure burner rig for advanced environmental barrier coating evaluation for Si_3N_4 applications**
  - Burns jet fuel and air with gas temperature up to 1650 °C (3000 °F)
  - Combustion gas velocity 10-30 m/s (6” ID)
  - Thermocouple and pyrometer temperature measurements
  - Variable specimen geometry



1” button specimen can also be used for testing

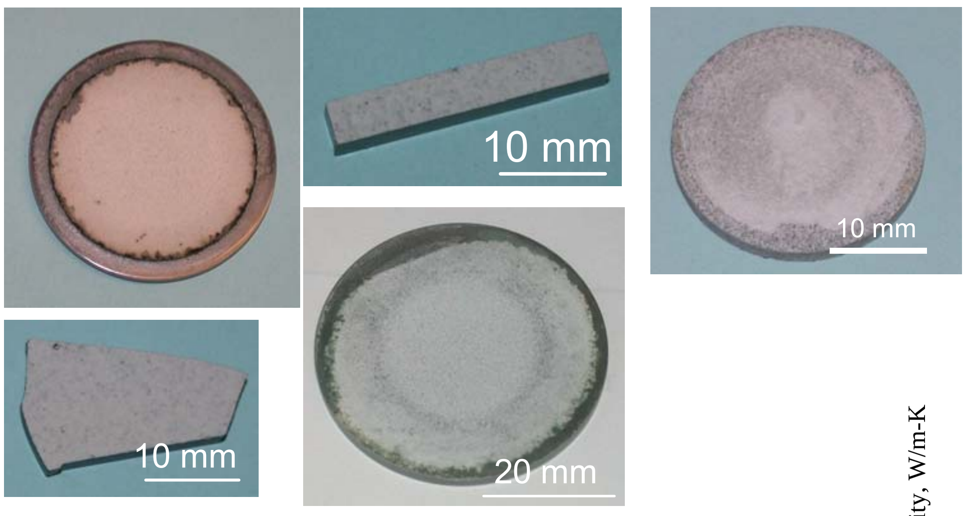
High pressure burner rig for advanced environmental barrier coating system evaluation

Finite Element Analysis modeling of specimen temperature distribution

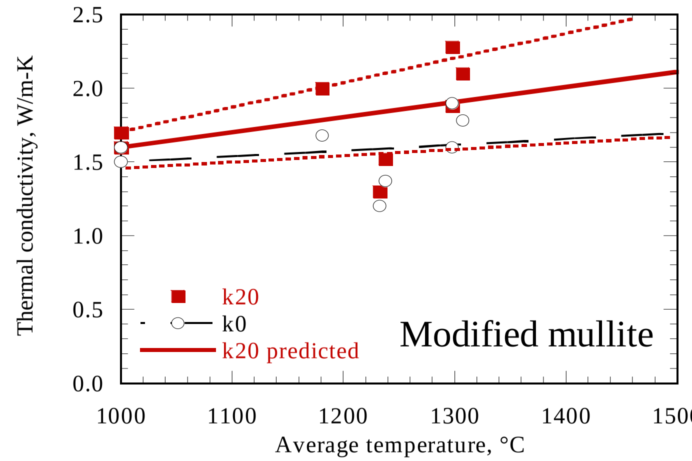


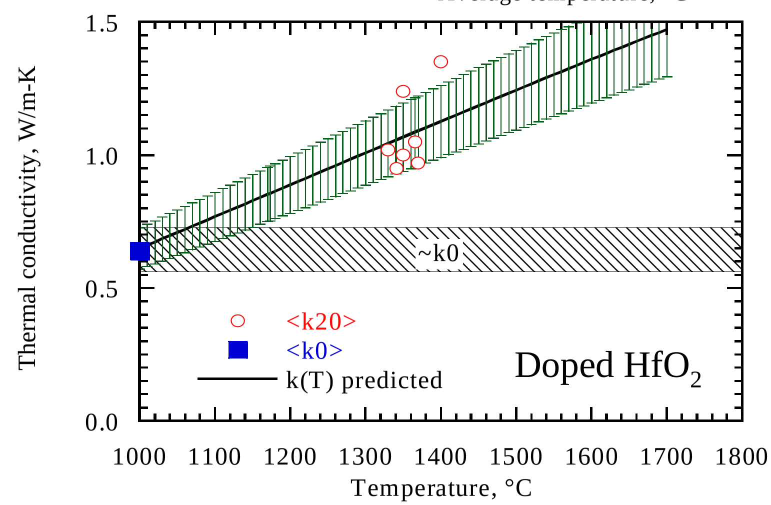
Multicomponent HfO_2-Mullite Coating Systems on Si_3N_4 Demonstrated High Temperature Durability

– The coatings tested using cyclic furnaces, laser rig and the high pressure burner rig at the temperatures up to 2650 °F (1450 °C) for at least 50 hours
– Coating temperature capability, water vapor stability and durability demonstrated
– Thermal conductivity being evaluated



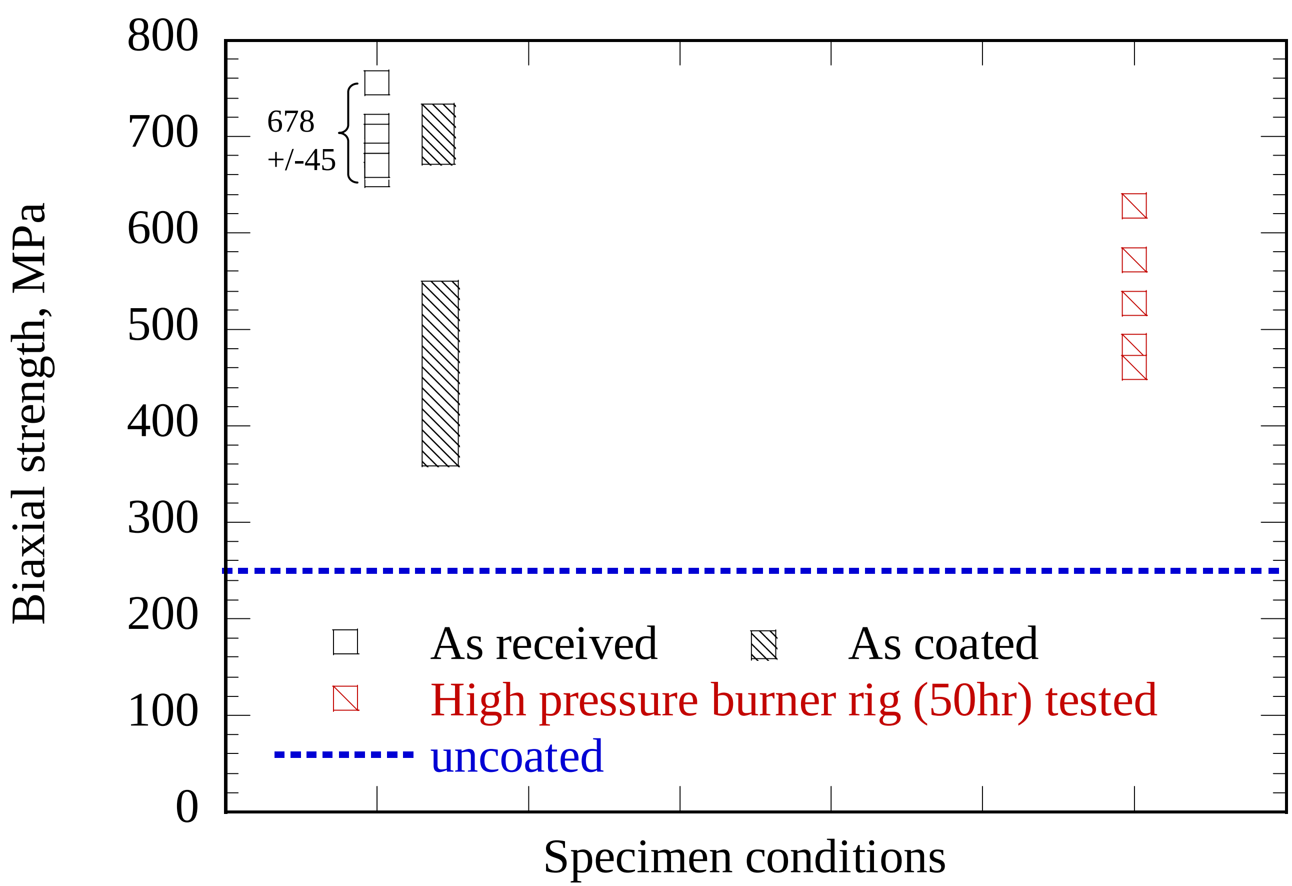
Selected test specimens after testing





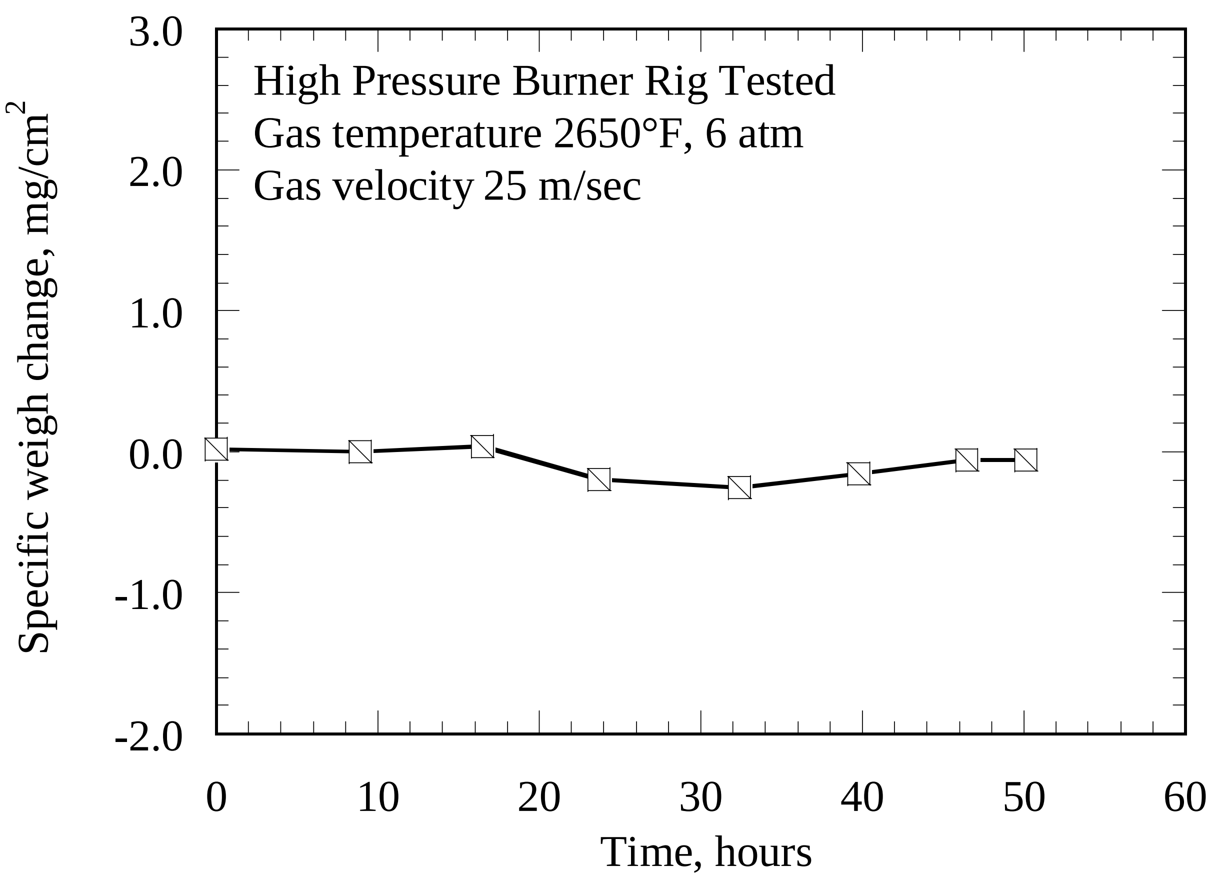
Environmental Barrier Coatings for Si_3N_4 Showed the Effectiveness in maintaining the Substrate Strength

- The coated specimens retained as-coated strength after high pressure burner rig testing
- Coated specimens also showed significant improvement in slow crack growth resistance



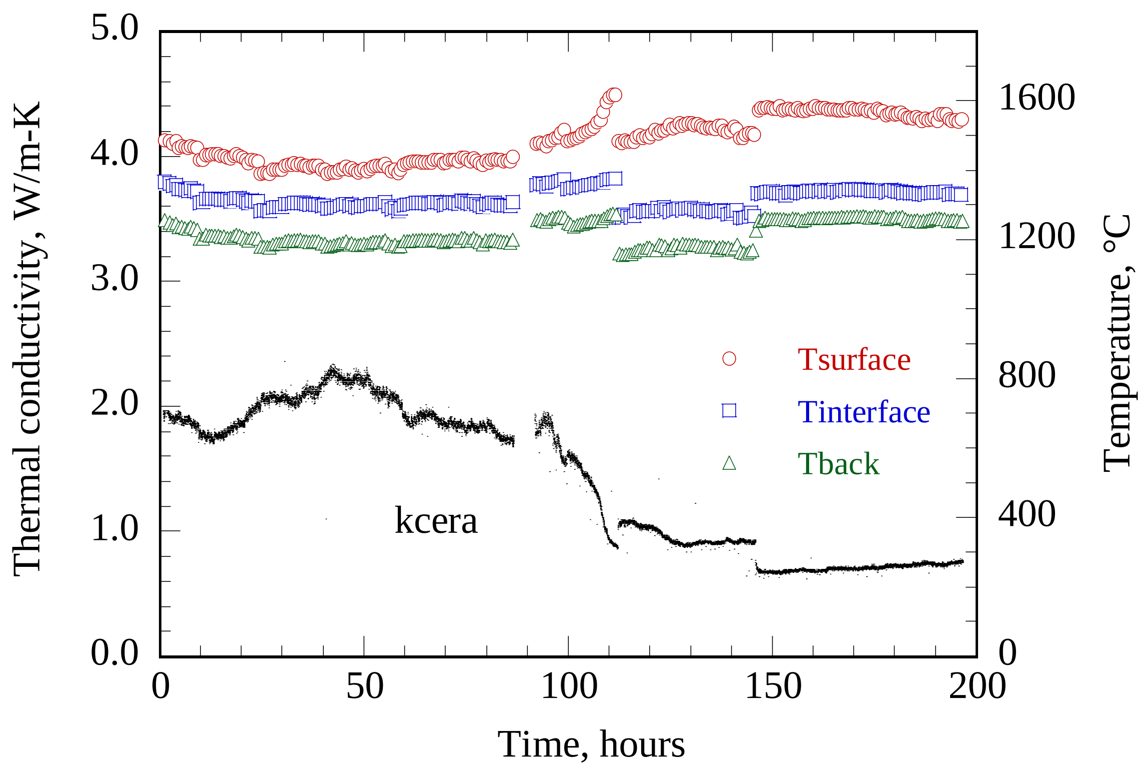
Hafnia-Based Coating Systems Showed Water Vapor Stability and Durability in High Pressure Burner Rig Testing

— HfO_2/mullite EBC system demonstrated 50 hours at 1300-1350 °C without weight loss and damage



Modified Mullite Showed Long-Term High Heat Flux Cyclic Durability on Si_3N_4 AS 800

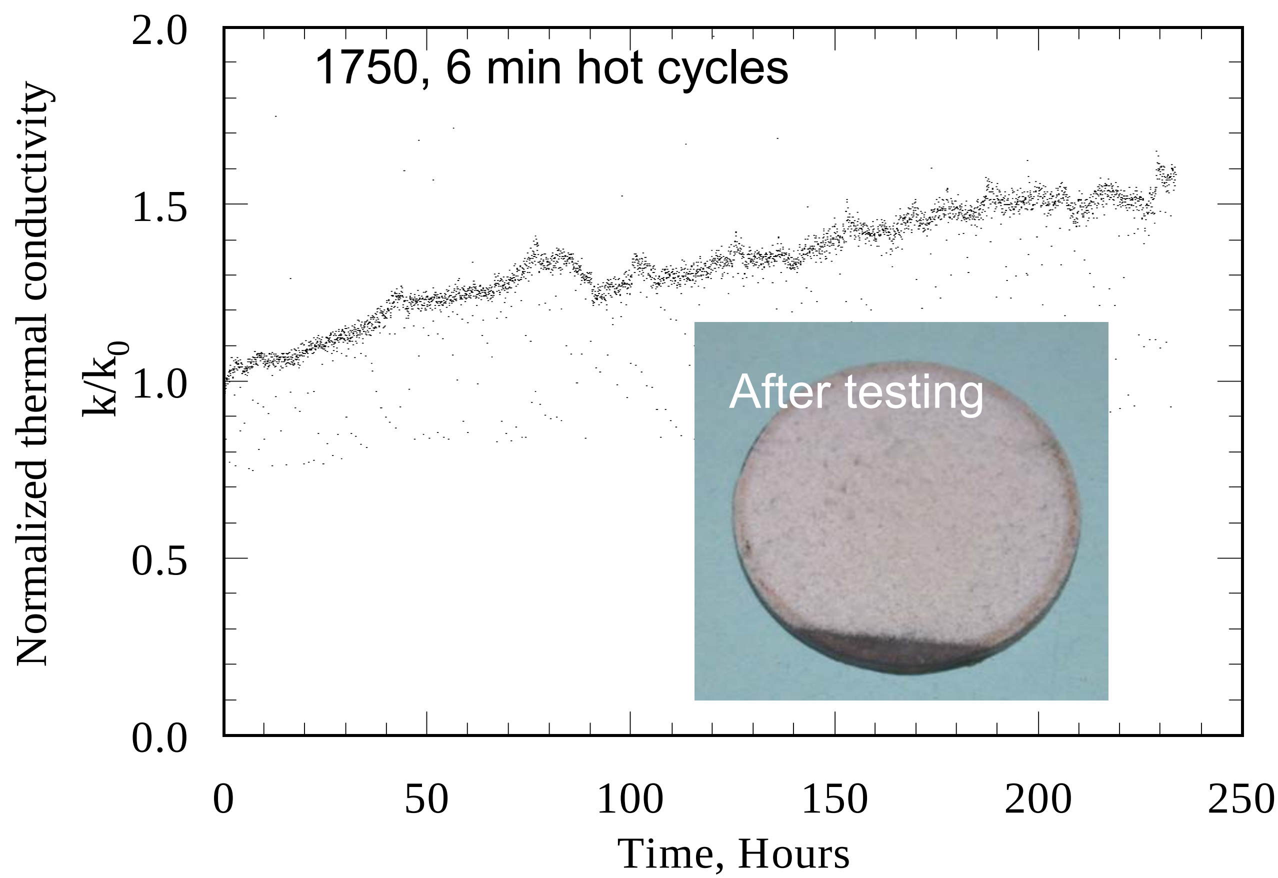
- The single layer doped mullite EBC on AS 800 Si_3N_4, tested at the surface temperature of 2700-2800 °F, Interface temperature of mostly 2500-2600 °F
- No visual damage observed after 380, 30 min hot cycles (1.5 min cool)



After 380 cycle testing

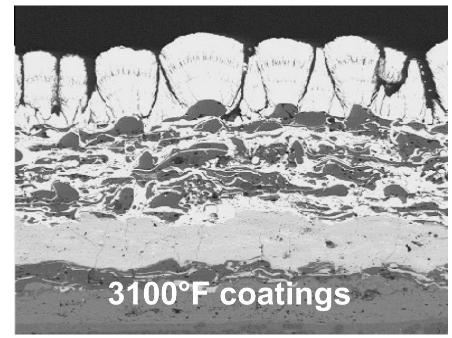
Modified Mullite Showed Long-Term High Heat Flux Cyclic Durability on SiC/SiC Ceramic Matrix Composites

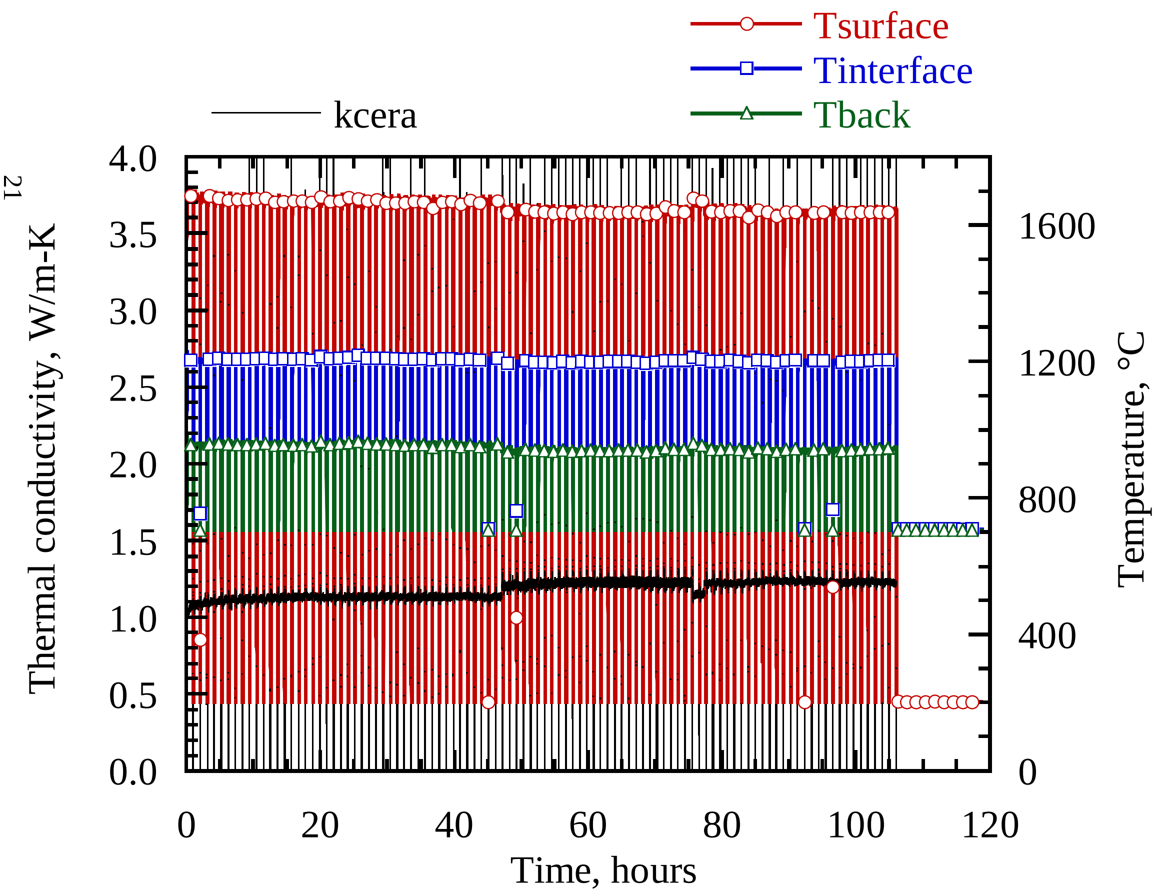
- The layer graded coating for thermal expansion adjustments and bond strength enhancements
- The interlayer coating tested at the surface temperature of 2650-2700 °F, Interface temperature of 2350 °F
- No damage observed after 1750, 6 min hot cycles (2 min cool)



NASA-GEAE 3100 °F CMC Turbine Vane and Combustor Coating Collaborative Efforts

— **The specimen successfully completed the total 100, 1 hr cycle laser heat flux test (60 min hot, 3 min cool) at 3100 °F**

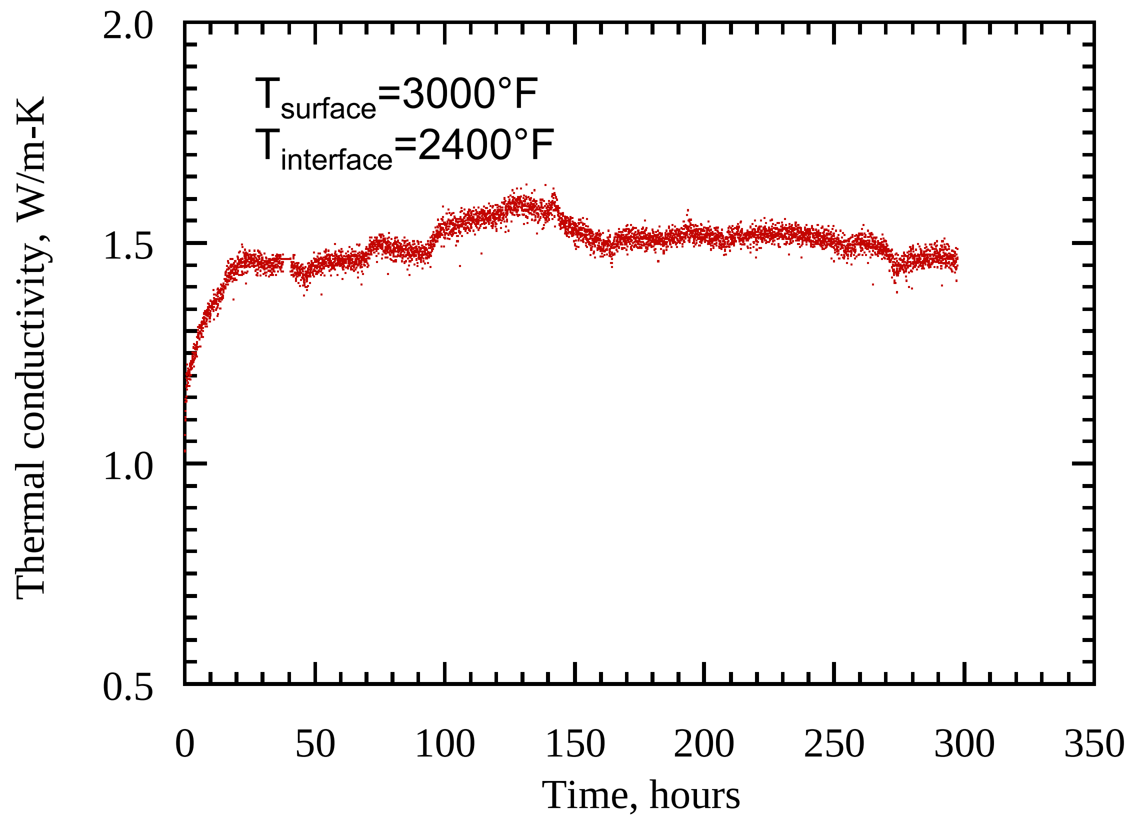




After testing

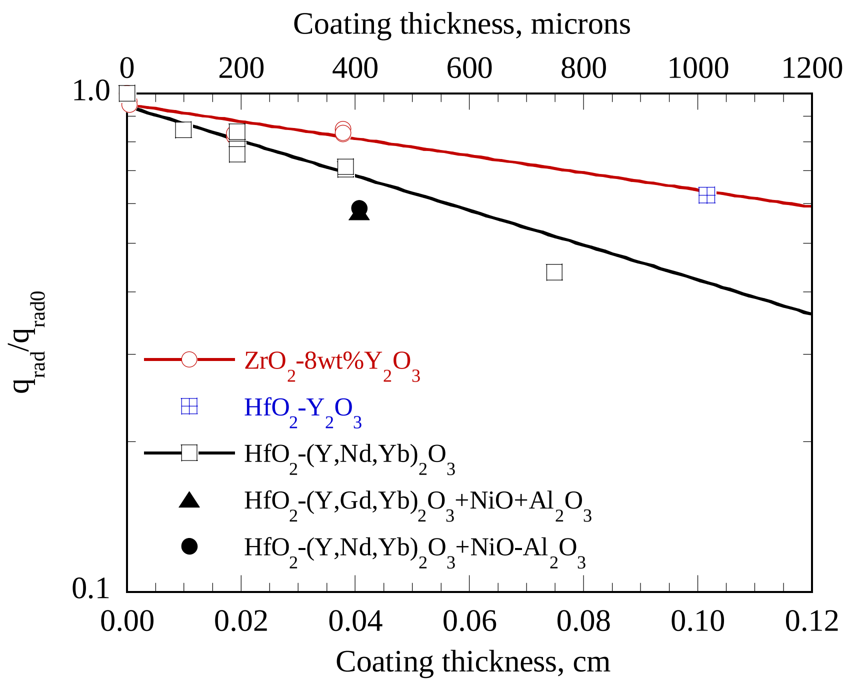
Multicomponent HfO_2-Mullite Coating Systems Demonstrated 3000 °F Sintering and Cyclic Durability

– **Multicomponent HfO_2-mullite based combustor coating systems demonstrated long-term 3000 °F sintering and cyclic durability on SiC/SiC CMCs under thermal gradient cyclic testing**

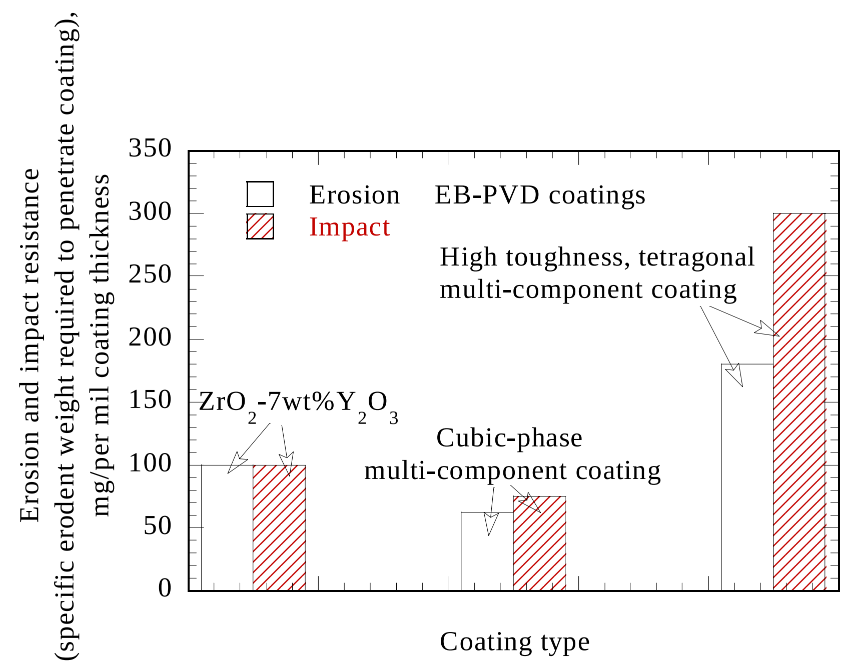


Radiation and Erosion Resistance of Advanced Coating Systems

- Oxide defect cluster and scattering center concepts established to reduce coating radiation flux and thus radiation conductivity
- Multi-component ZrO_2 and HfO_2 coatings showed promise for improving the coating radiation and erosion resistance



The ratio of the pass-through radiation heat-flux q_{rad} to the imposed radiation flux q_{rad0} for plasma-sprayed coatings, as a function of coating thickness, determined by a laser heated black-body emitting source-flux technique. The radiation resistance increased for the doped HfO_2 coatings.



Erosion and impact resistance, measured as the erodent Al_2O_3 weight required to penetrate unit thickness coating

Summary and Conclusions

- Advanced environmental barrier coating systems developed for SiC/SiC CMC and Si_3N_4 ceramics
  – Multi-component HfO_2 (ZrO_2) – mullite coating material systems
  – Multifunctionally graded coating systems and layer graded coating systems
  – Advanced high temperature capable ceramic bond coats demonstrated

- High temperature water vapor stability and cyclic durability demonstrated
  – 2700 °F Si_3N_4 coatings demonstrated 200 hr durability
  – 3000 °F combustor SiC/SiC coating demonstrated 300 hr durability
  – 3100 °F turbine vane coatings demonstrated 100 hr cyclic durability

- Thermal radiation and erosion resistance also being improved
  – Defect cluster and high toughness coatings

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)

2. REPORT DATE
March 2005

3. REPORT TYPE AND DATES COVERED
Technical Memorandum

4. TITLE AND SUBTITLE
Advanced Environmental Barrier Coatings Development for Si-Based Ceramics

5. FUNDING NUMBERS
WBS–22–714–20–09

6. AUTHOR(S)
Dongming Zhu, Sung R. Choi, Raymond C. Robinson, Kang N. Lee, Ramakrishna T. Bhatt, and Robert A. Miller

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
Cleveland, Ohio 44135–3191

8. PERFORMING ORGANIZATION REPORT NUMBER
E–14980

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM—2005-213444

11. SUPPLEMENTARY NOTES
Viewgraphs prepared for the Third Annual Workshop on Environmental Barrier Coatings and Enhanced Silicon Nitride for Gas Turbines cosponsored by the Department of Energy's Office of Distributed Energy, Air Force Research Laboratory, and National Aeronautics and Space Administration, Nashville, Tennessee, November 17–18, 2004. Dongming Zhu, U.S. Army Research Laboratory, NASA Glenn Research Center; Sung R. Choi, Ohio Aerospace Institute, 22800 Cedar Point Road, Brook Park, Ohio 44142; Raymond C. Robinson; QSS Group, Inc., 21000 Brookpark Road, Cleveland, Ohio 44135; Kang N. Lee, Cleveland State University, 1983 E. 24th Street, Cleveland, Ohio 44115; Ramakrishna T. Bhatt, U.S. Army Research Laboratory, NASA Glenn Research Center; and Robert A. Miller, NASA Glenn Research Center. Responsible person, Dongming Zhu, organization code RMD, 216–433–5422.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified - Unlimited
Subject Categories: 23, 24, and 27 Distribution: Nonstandard
Available electronically at http://gltrs.grc.nasa.gov
This publication is available from the NASA Center for AeroSpace Information, 301–621–0390.

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

Advanced environmental barrier coating concepts based on multi-component HfO_2 (ZrO_2) and modified mullite systems are developed for monolithic Si_3N_4 and SiC/SiC ceramic matrix composite (CMC) applications. Comprehensive testing approaches were established using the water vapor cyclic furnace, high pressure burner rig and laser heat flux steam rig to evaluate the coating water vapor stability, cyclic durability, radiation and erosion resistance under simulated engine environments. Test results demonstrated the feasibility and durability of the environmental barrier coating systems for 2700 to 3000 °F monolithic Si_3N_4 and SiC/SiC CMC component applications. The high-temperature-capable environmental barrier coating systems are being further developed and optimized in collaboration with engine companies for advanced turbine engine applications.

14. SUBJECT TERMS
Environmental barrier coating; Advanced heat flux testing; Plasma-spray; Thermal conductivity; Sintering; Creep

15. NUMBER OF PAGES
30

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	