



Performance and Durability of Environmental Barrier Coatings on SiC/SiC Ceramic Matrix Composites

Dongming Zhu, Bryan Harder and Ramakrishna Bhatt
Materials and Structures Division
NASA John H. Glenn Research Center
Cleveland, Ohio 44135, USA

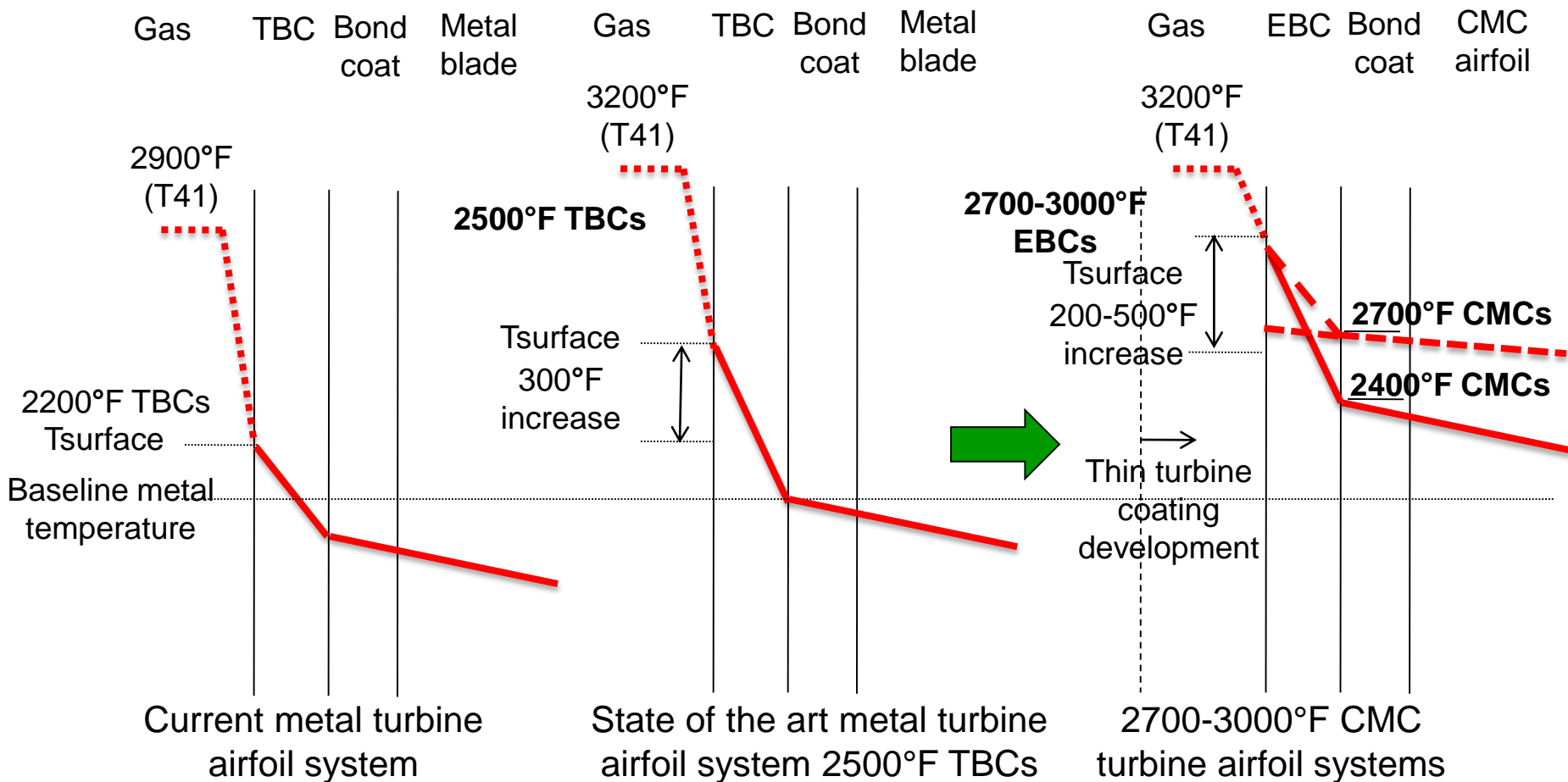


Materials Science and Technology Conference 2016
Salt Lake City, Utah
October 24-27, 2016



NASA Turbine Environmental Barrier Coatings for CMC-EBC Systems

- Emphasize temperature capability, performance and durability for next generation turbine engine systems
- Increase Technology Readiness Levels for component system demonstrations





Environmental Barrier Coating and SiC/SiC System Development: Testing Challenges

- High Temperatures: 2700 to 3000°F (1500-1650°C) along with higher interface temperatures
- Exposure to water vapor and combustion products
- High Cyclic Stresses: thermal and mechanical, creep-fatigue effect
- Combined Interactions, in-plane and through-thickness gradients
- High Velocity Gases: Mach 1 and 2
- High Pressures: ~ up to 40 to 50 atmospheres
- Long term durability: 20,000 hr design life



Outline

- **Advanced testing approaches for SiC/SiC and ceramic coating development: laser high heat flux based testing approaches**
 - NASA CO₂ laser rig development
 - Thermal conductivity
 - Cyclic durability and monitoring degradations of EBCs and CMCs

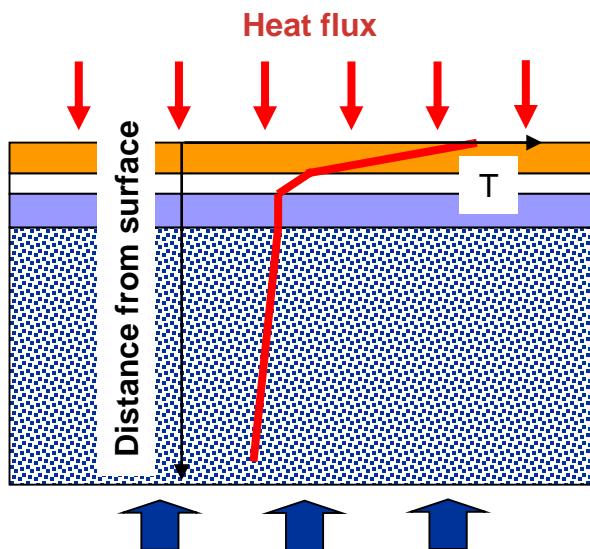
- **Laser high heat flux and mechanical tests**
 - Combined high heat flux - mechanical tests
 - High heat flux biaxial creep/fatigue test rigs
 - Sub-element testing

- **Summary and future directions**

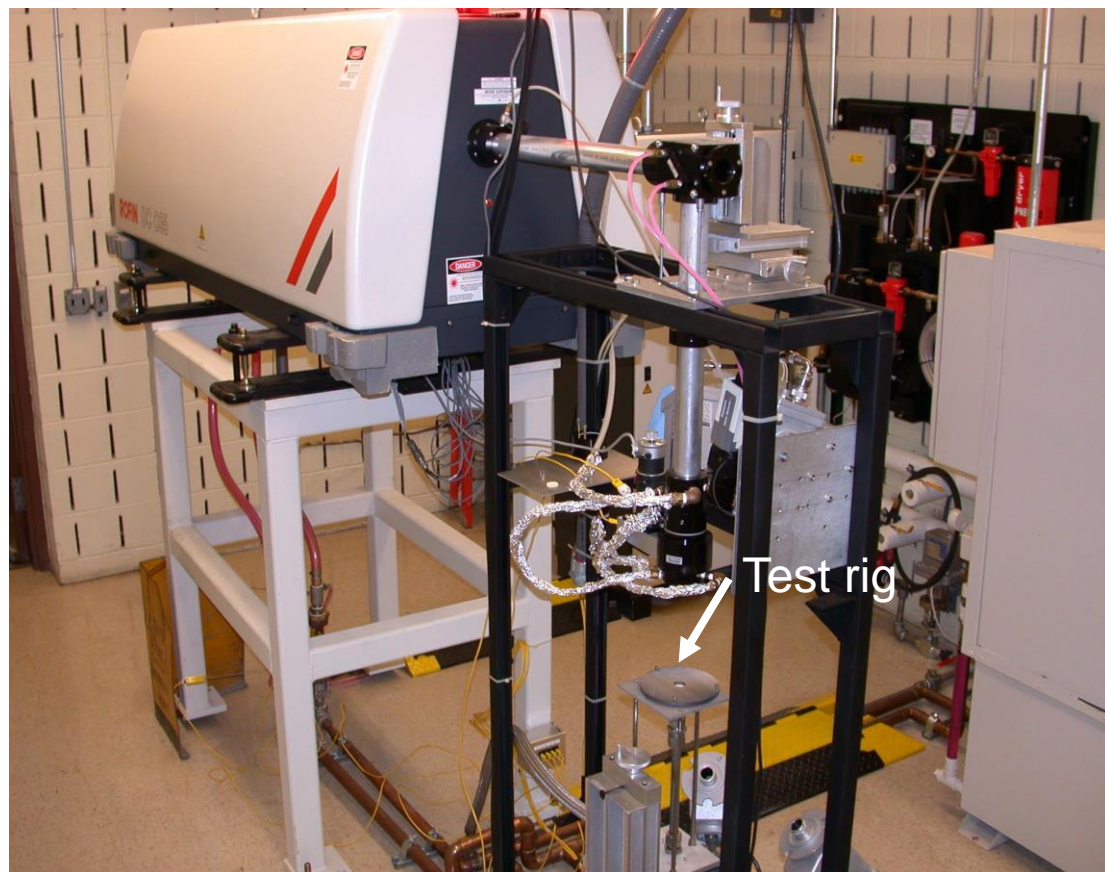
High Power CO₂ Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

- Developed in 1990's, the rig achieved turbine level high-heat-fluxes (315 W/cm²) for turbine thermal barrier coating testing
- Crucial for advanced EBC-CMC developments

Turbine: 450°F across 100 microns
Combustor: 1250°F across 400 microns



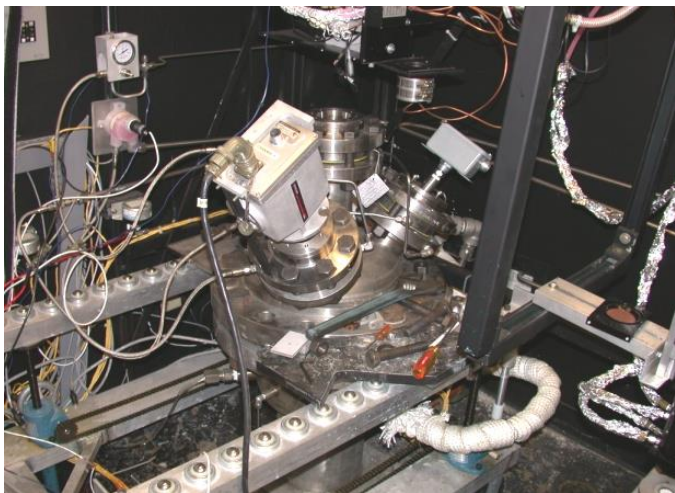
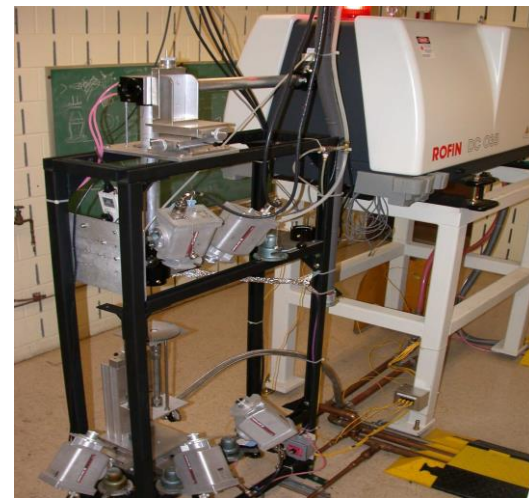
Cooling – high velocity air or air-water mist
Achieved heat transfer coefficient 0.3 W/cm²-K



High Power CO₂ Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

- Continued

- NASA high power CO₂ laser rig systems
- Various test rigs developed
- 7.9 micron single wavelength and 1 micron two color wavelength pyrometers for temperature measurements
- Thermography system for temperature distribution measurements
- Capable of programmable test mission cycles
- Capable of mechanical load cycles under high heat flux
- Environment test conditions (e.g., steam and vacuum)



Laser heat flux high temperature thermal gradient combustor subelement test rig

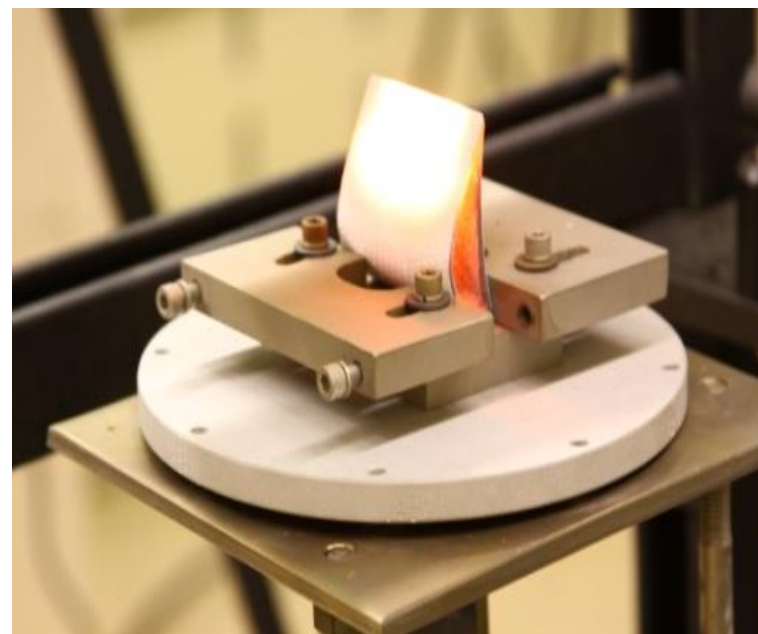
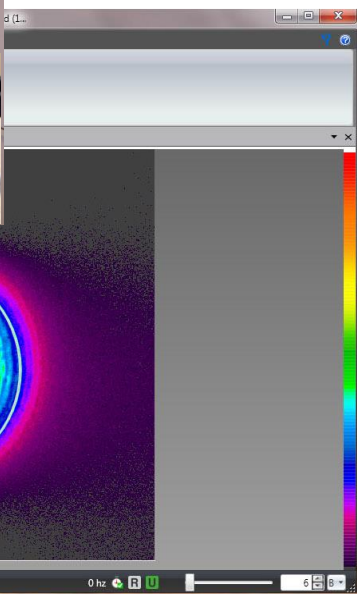
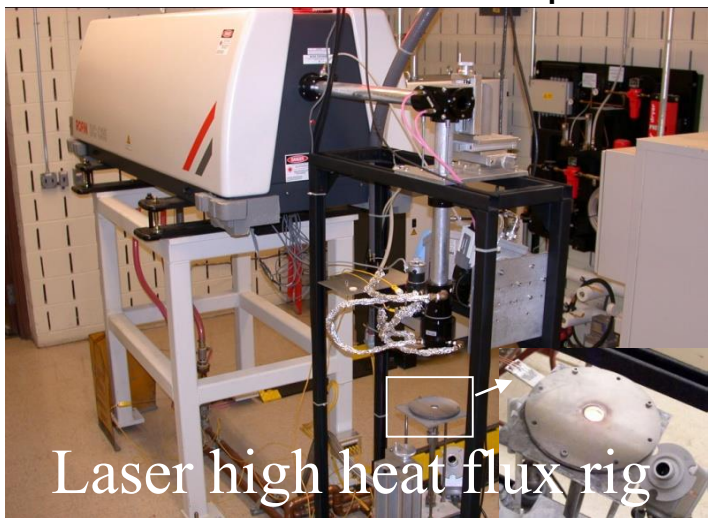


Laser high heat flux creep rupture test rig

High Power CO₂ Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

– Continued

- Controlled beam profiles, beam size and power density were major emphases, by using rotating ZnSe integrating lens with various focus lengths
- Uniform distribution up to 2-3” diameter beam size for various testing

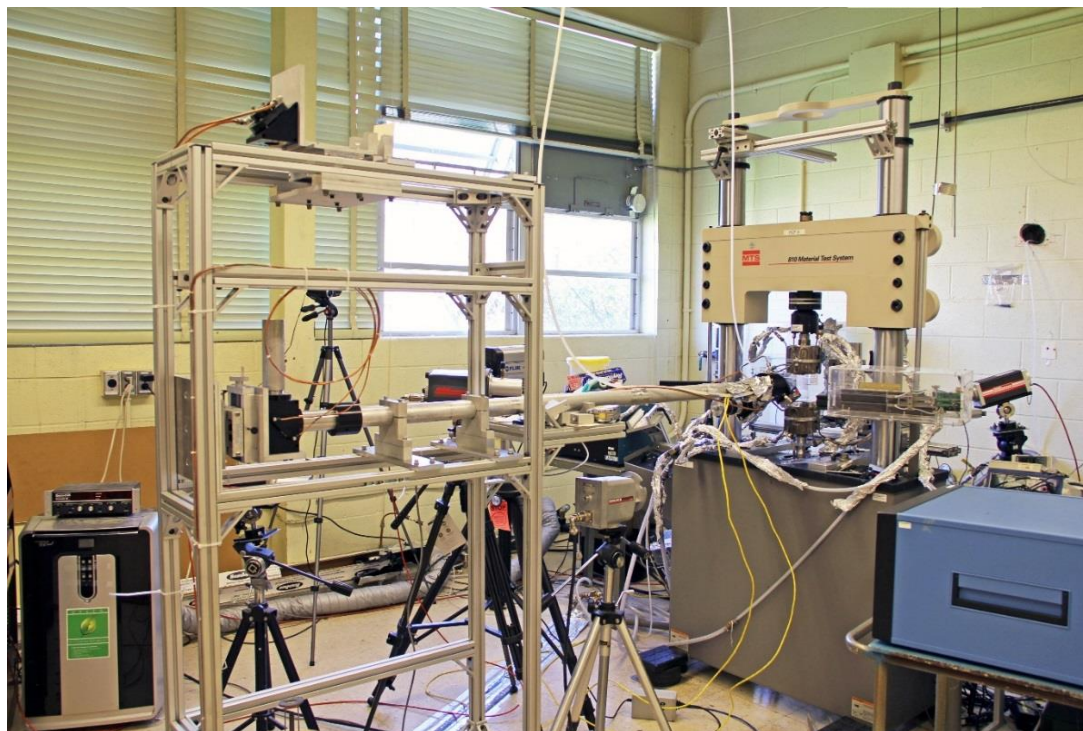


2” beam size subelement tests

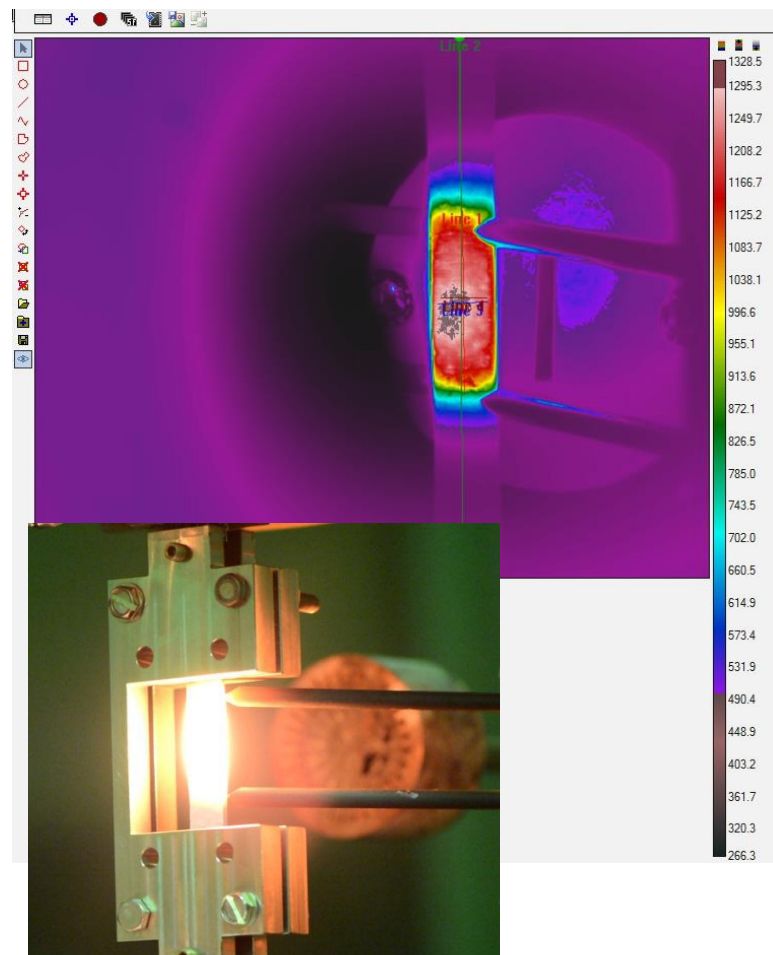
Example of 1” diameter disc specimen tests and beam profile

High Power CO₂ Laser Based High Heat Flux Fatigue Test Rig

- Laser creep and fatigue testing capable of full tension and compression loading
- Uniform distribution up to 2-3” diameter beam size for various testing, depending on the heat flux requirements



Laser heat flux Thermal HCF/LCF Rig – Overall View



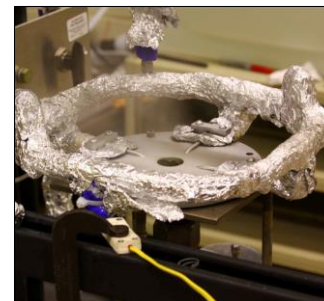
Specimen under testing in tensile-compression fatigue rig

High Heat Flux Rig Testing with Water vapor Steam Chamber – Established in Early 2000

- High temperature and high-heat-flux testing capabilities
- “Micro-steam environment” allowing high water vapor pressure (100% steam), relatively high velocity under very high temperature condition
- Used for 3000°F EBC-CMC developments



Specimen under testing

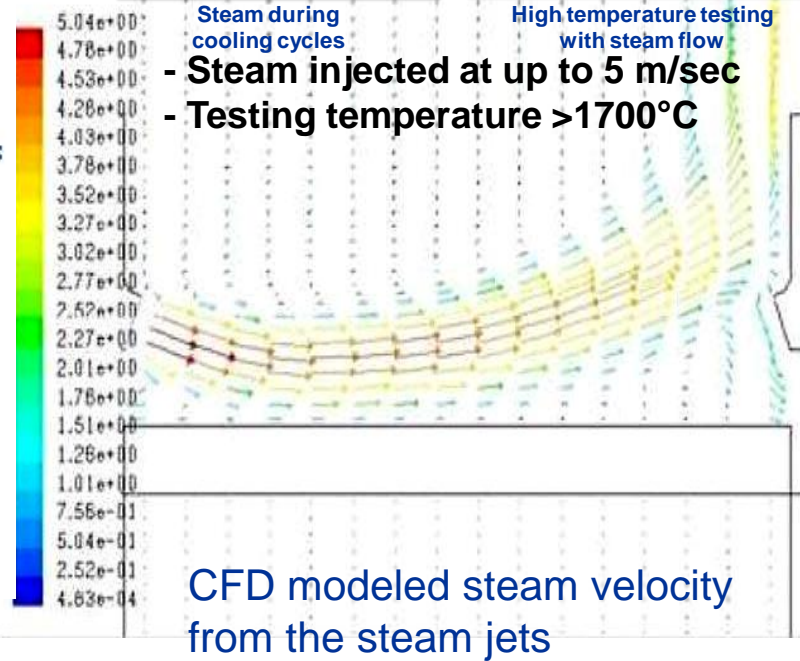


Steam during cooling cycles



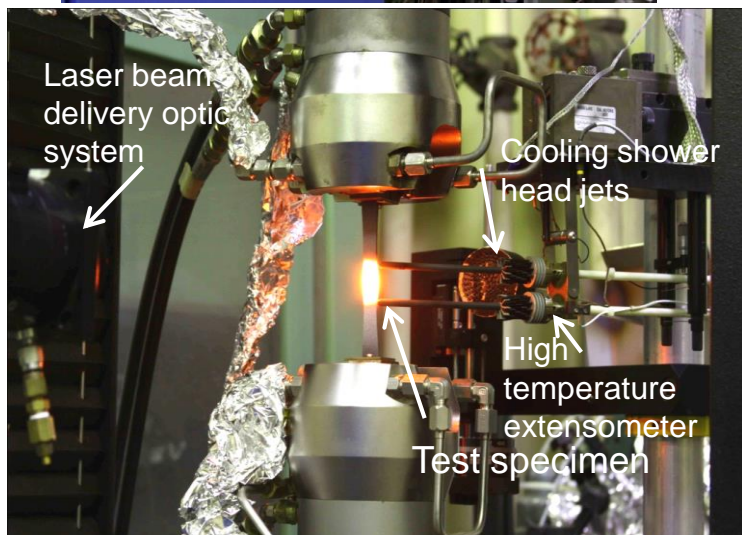
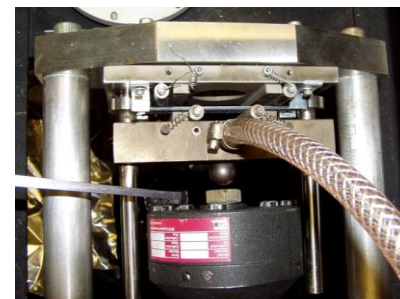
High temperature testing with steam flow

- Steam injected at up to 5 m/sec
- Testing temperature >1700°C

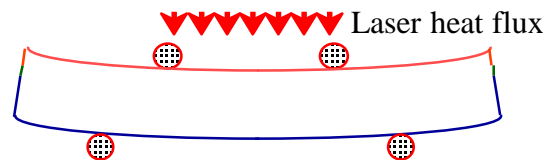


High Heat Flux Thermomechanical Testing for EBC Development

- High heat flux and combined thermal-mechanical loading capabilities established to allow SiC/SiC system performance data to be obtained under simulated operating thermo-mechanical and environmental conditions



High heat flux tensile TMF and rupture testing



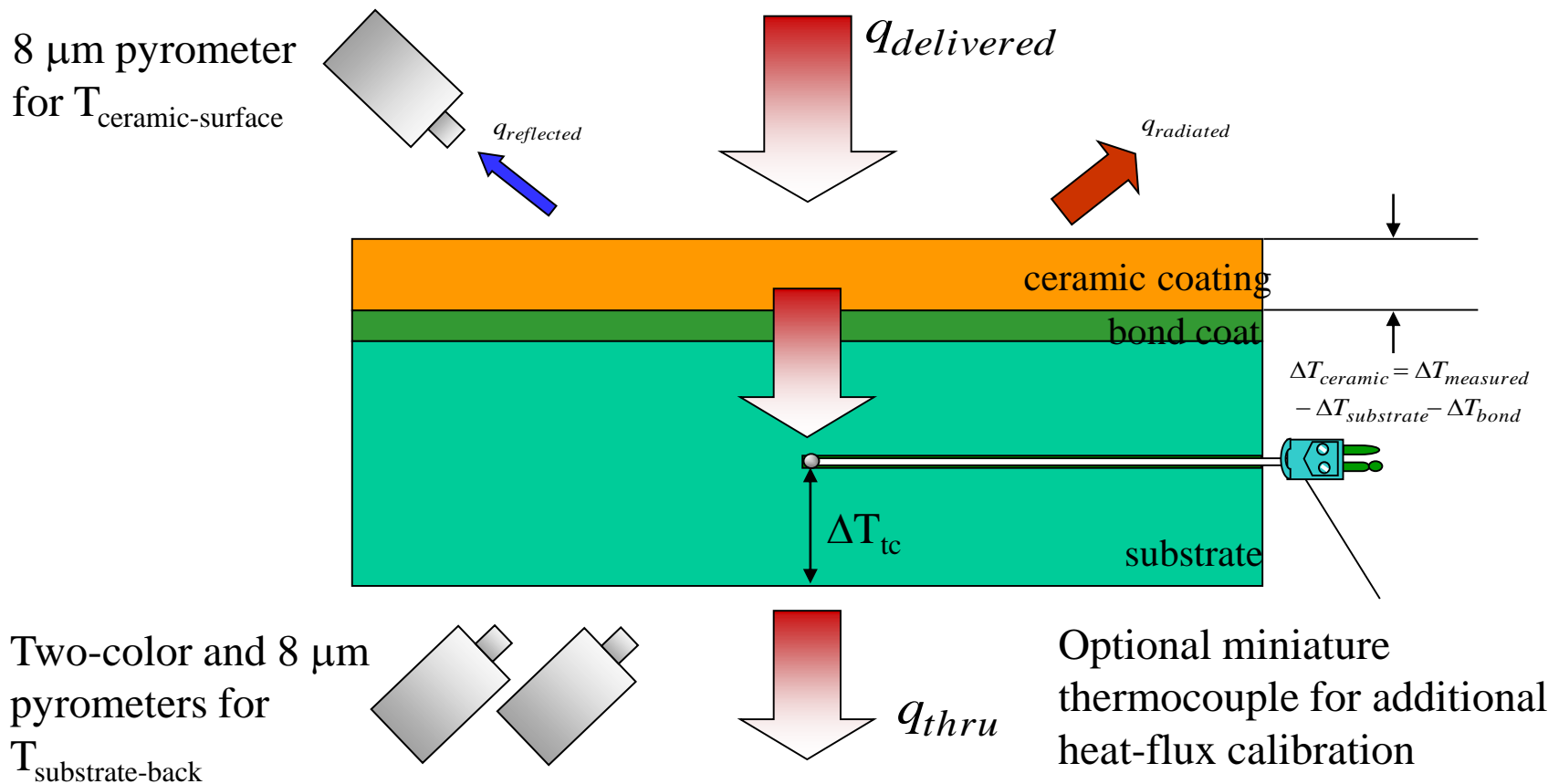
High heat flux flexural TMF testing: HCF, LCF, interlaminar and biaxial strengths

Thermal Conductivity Measurement by a Laser High-Heat-Flux Approach

$$k_{ceramic}(t) = q_{thru} \cdot l_{ceramic} / \Delta T_{ceramic}(t)$$

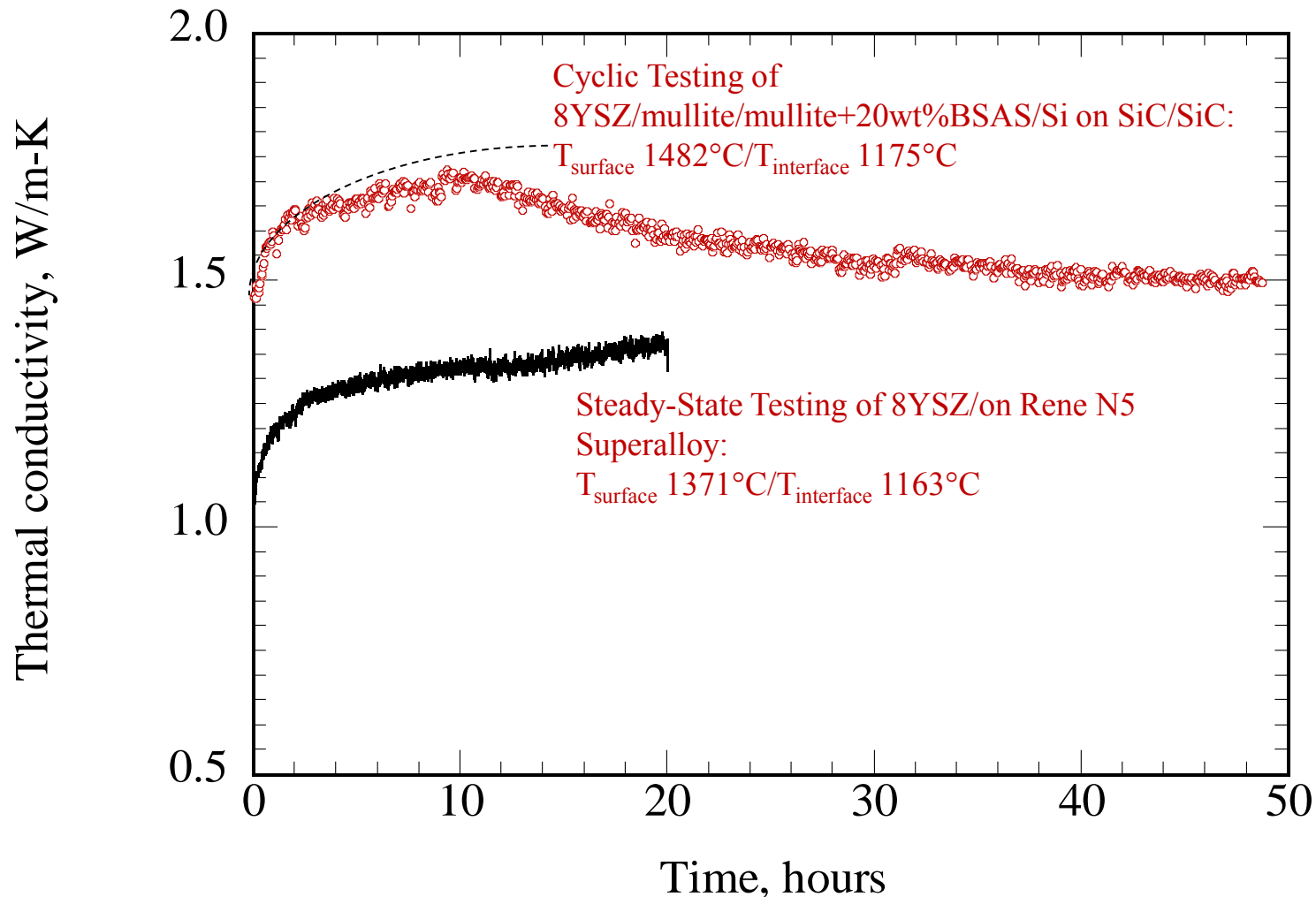
Where

$$q_{thru} = q_{delivered} - q_{reflected} - q_{radiated} \quad \text{and} \quad \Delta T_{ceramic}(t) = T_{ceramic-surface} - T_{metal-back} - \int_0^{l_{bond}} \frac{q_{thru} \cdot dl}{k_{bond}(T)} - \int_0^{l_{substrate}} \frac{q_{thru} \cdot dl}{k_{substrate}(T)}$$

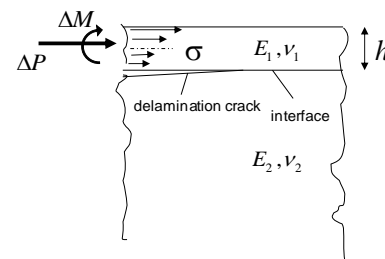
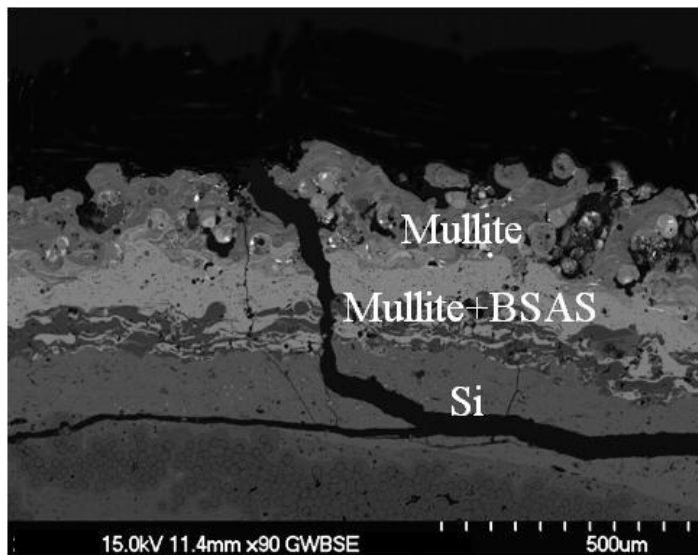


Thermal Gradient Cyclic Behavior of a Thermal Environmental Barrier Coating System

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

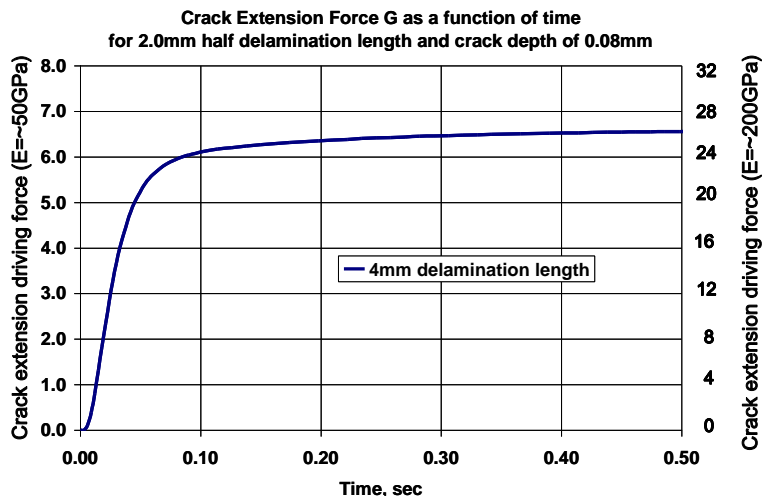


Environmental Barrier Coating and High Heat Flux Induced Delaminations

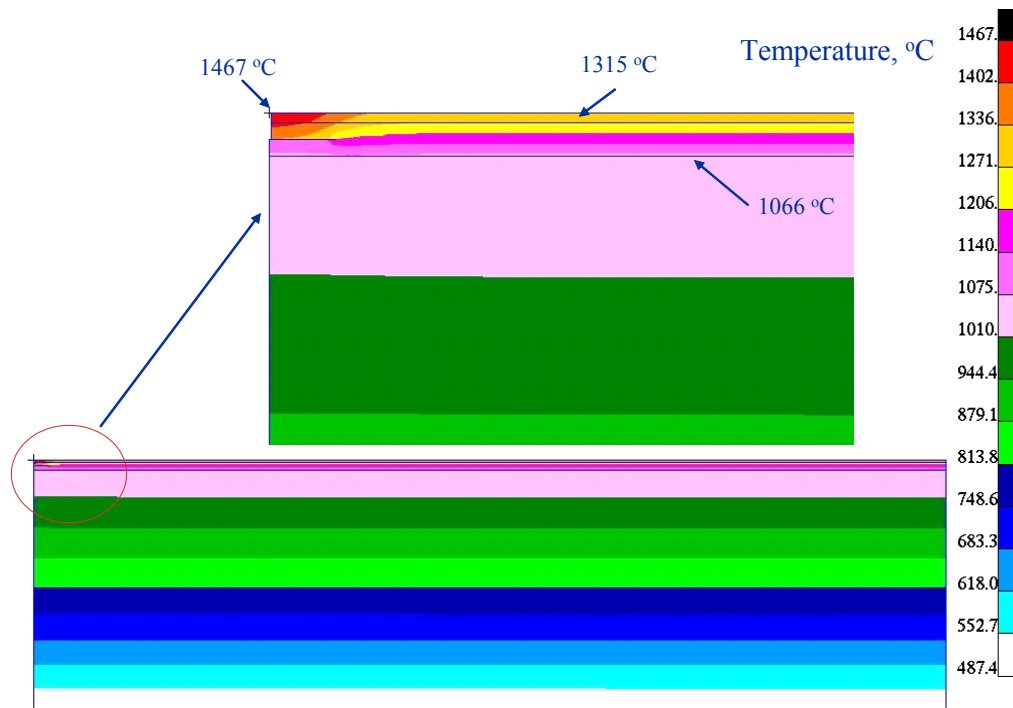


Evans and Hutchinson model, Surface Coating Technology, 2007

$$G = \frac{1}{6} \left(\frac{1+\nu_1}{1-\nu_1} \right) E_1 h (\alpha_1 (T_s - T_0))^2$$

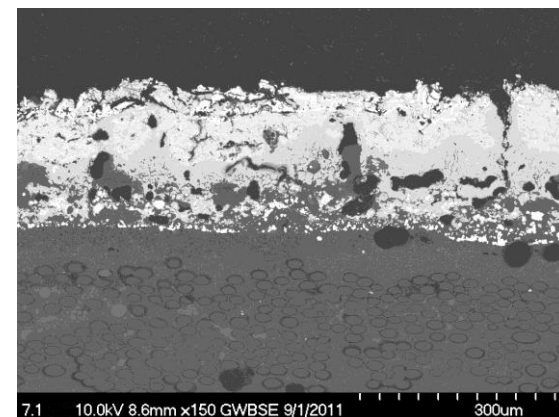
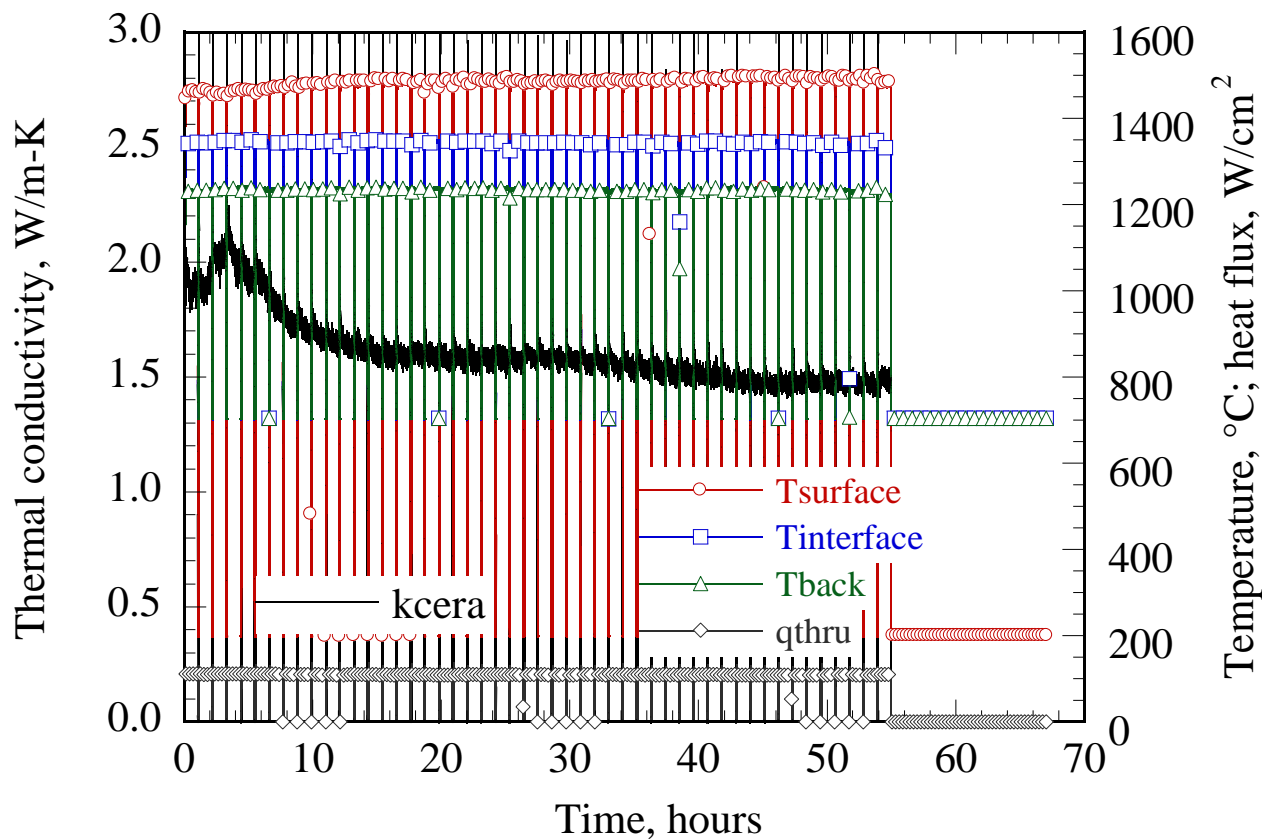


The FEM model



Thermal Gradient Cyclic Behavior of Air Plasma Sprayed Yb_2SiO_5 (with HfO_2 Composite)/ $\text{Yb}_2\text{Si}_2\text{O}_7$ / HfO_2 -Si Coatings on SiC/SiC CMCs

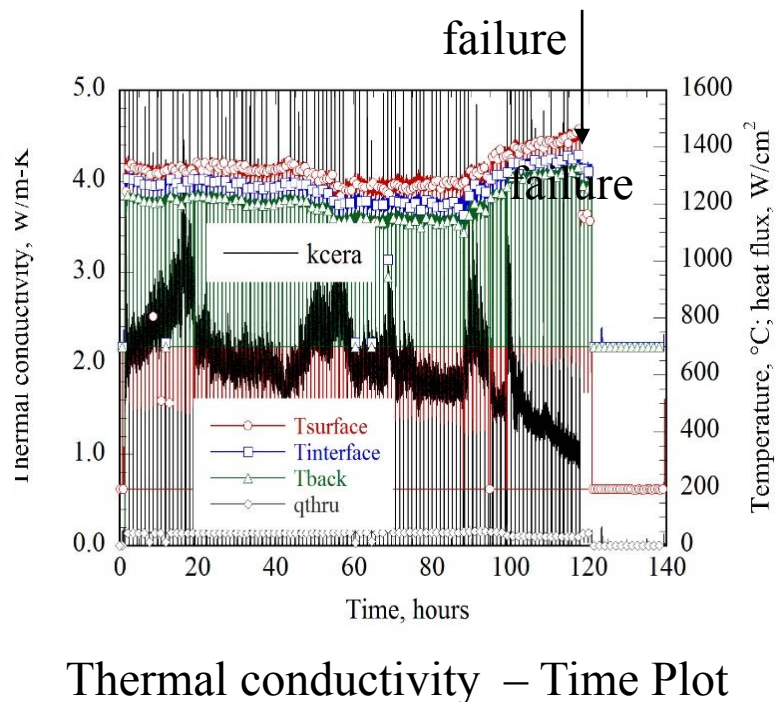
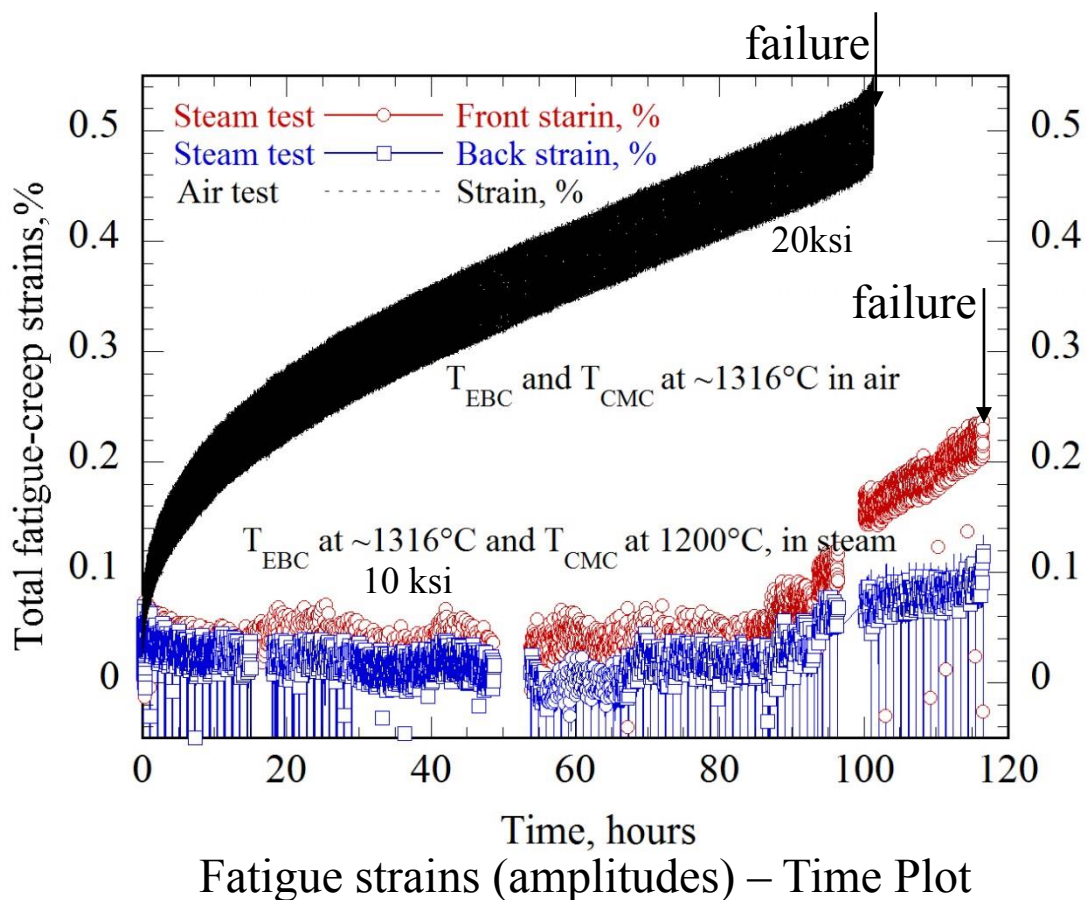
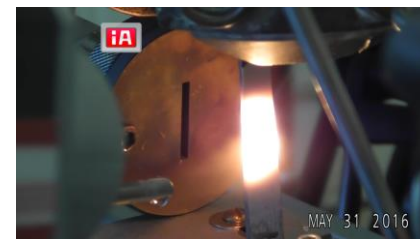
- $T_{\text{surface}} \sim 1482\text{-}1500^\circ\text{C}$, $T_{\text{interface}} 1350^\circ\text{C}$, $T_{\text{back surface}} 1225^\circ\text{C}$, heat flux 110 W/cm^2
- Localized pore formation



After 50hr Cyclic Testing

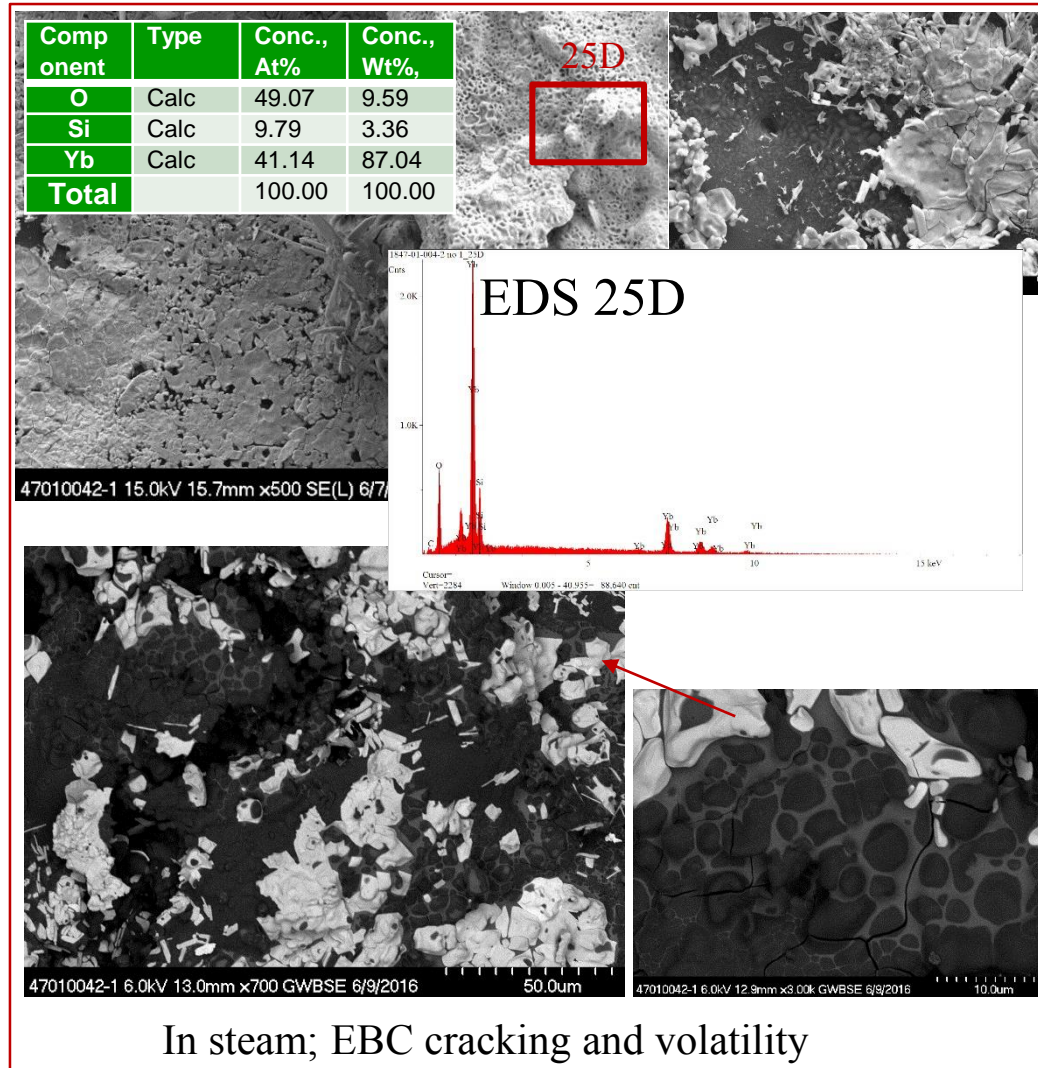
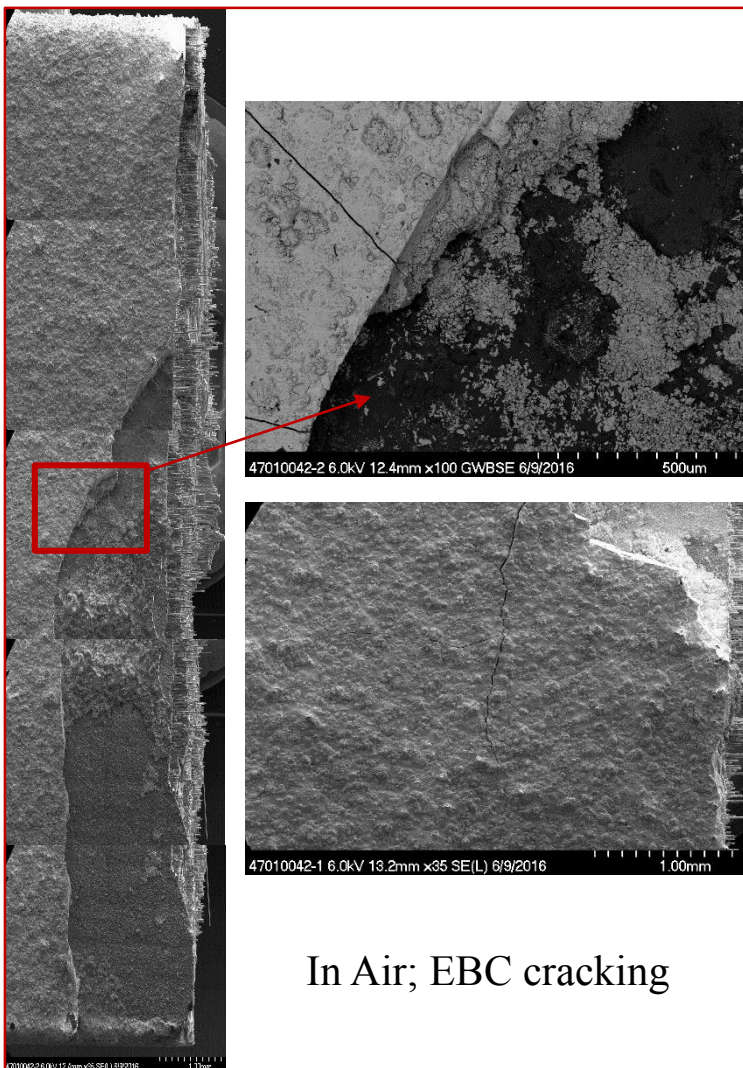
Fatigue Testing using a Laser High-Heat-Flux Approach for Environmental Barrier Coated Prepreg SiC/SiC CMCs

- Environmental Barrier Coatings $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7/\text{Si}$ on MI Prepreg SiC/SiC CMC substrates
- One specimen tested in air, air testing at 1316°C
- One specimen tested in steam, steam testing at $T_{\text{EBC}} 1316^\circ\text{C}$, T_{CMC} at $\sim 1200^\circ\text{C}$
- Lower CMC failure strain observed in steam test environments



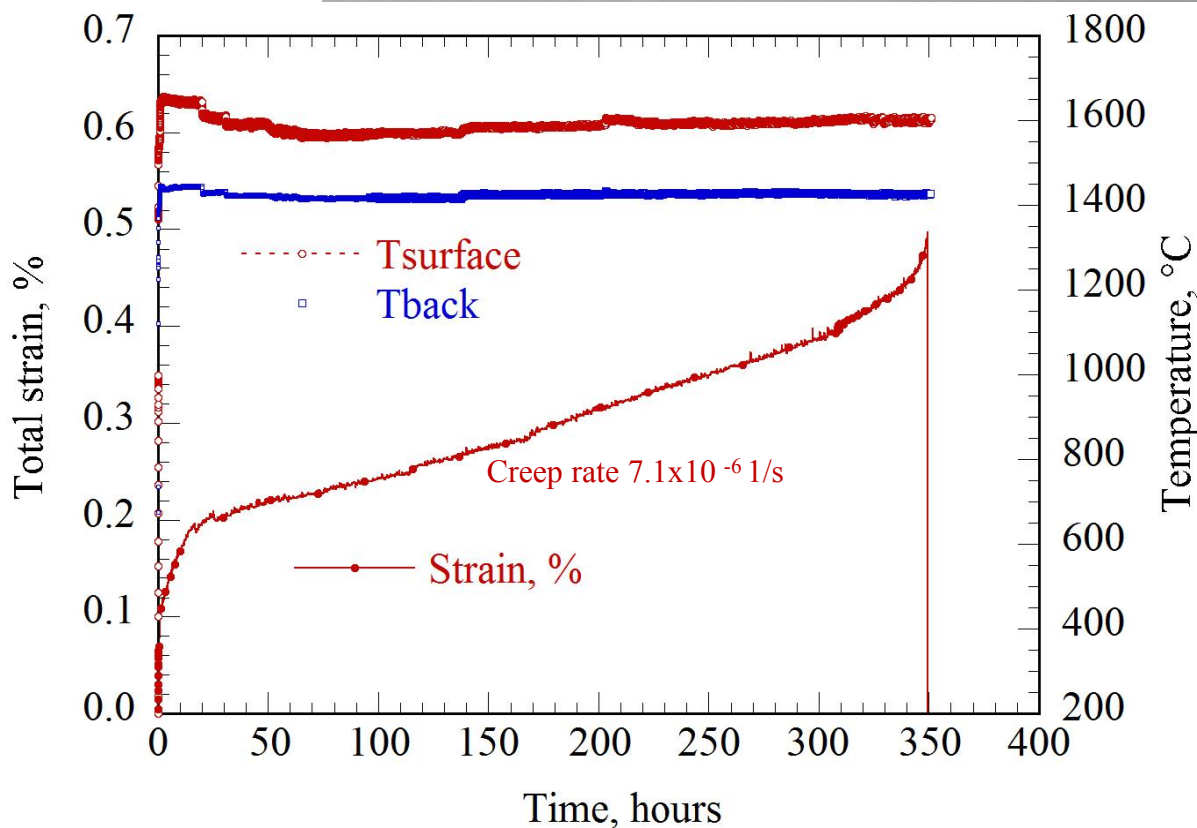
Fatigue Testing using a Laser High-Heat-Flux Approach for EBC Coated Prepreg SiC/SiC CMCs - Continued

- Crack and recession failure in air and steam tests



EBC Coated CMC 2650°F (1454°C) Creep Rupture Durability Test

- SiC/SiC CMC SiC/SiC CVI-MI CMC specimen
- Coated with RESi and Rare Earth EBC
- Test temperatures: $T_{\text{EBC surface}}$ at 2850-3000°F (1600-1650°C), and $T_{\text{cmc back}}$ at ~2600°F (1426°C)

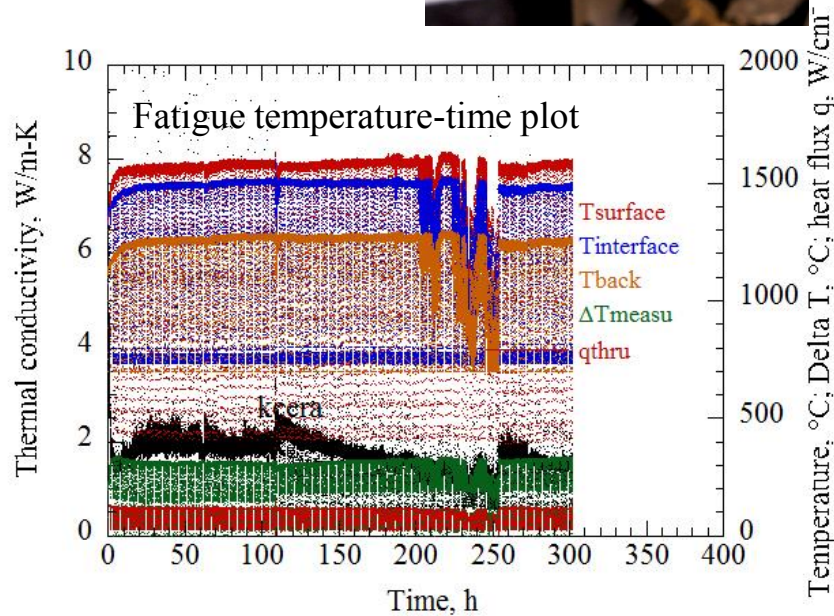
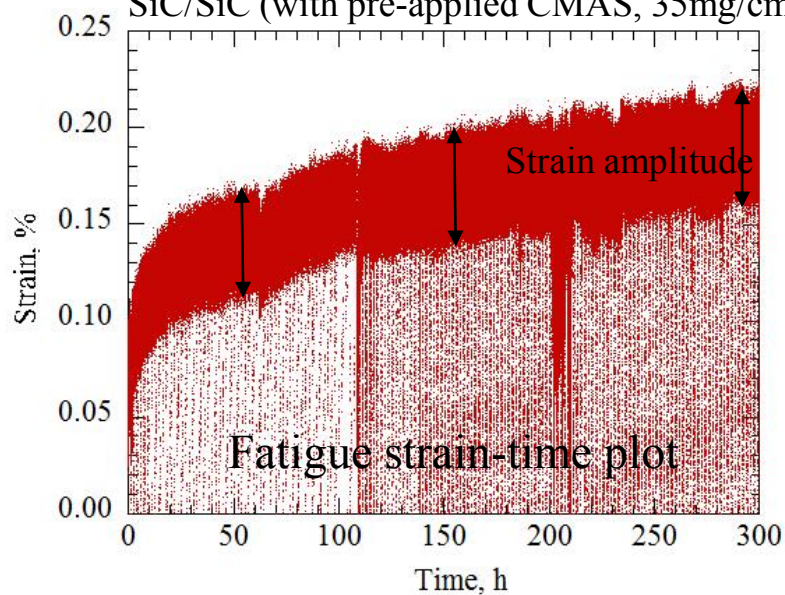


Advanced EBC-CMC Fatigue Test with CMAS: Tested 300 h Durability in High Heat Flux Fatigue Test Conditions

- A turbine EB-PVD turbine airfoil EBC system with advanced HfO₂-rare earth silicate and GdYbSi (controlled oxygen activity) bond coat tested at $T_{\text{EBC-surface}} 1537^{\circ}\text{C}+$, $T_{\text{bond coat}} 1480\text{-}1500^{\circ}\text{C}$, $T_{\text{back CMC surface}} 1250\text{-}1300^{\circ}\text{C}$
- Fatigue Stress amplitude 69 MPa, at mechanical fatigue frequency $f=3\text{Hz}$, stress ratio $R=0.05$
- Low cycle thermal gradient fatigue 60min hot, 3min cooling

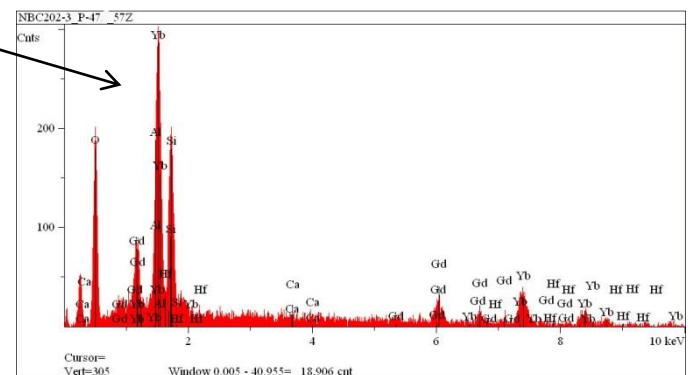
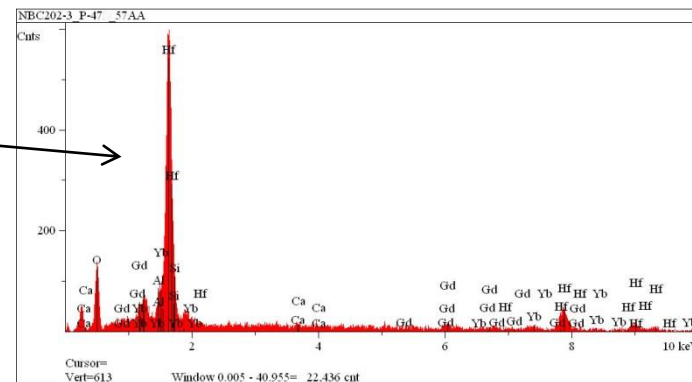
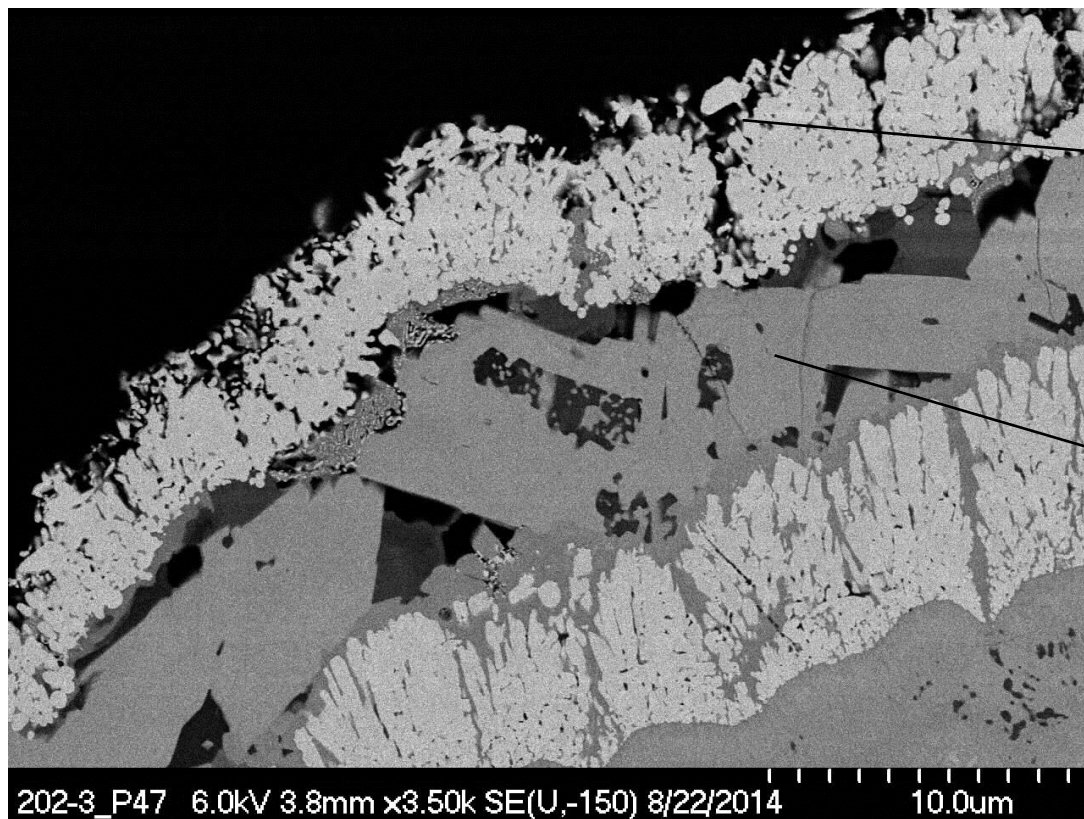


1537°C, 69MPa (10ksi), 300 h fatigue (3 Hz, R=0.05) CVI-MI SiC/SiC (with pre-applied CMAS, 35mg/cm²)



Generally Observed EBC Test Failure with CMAS

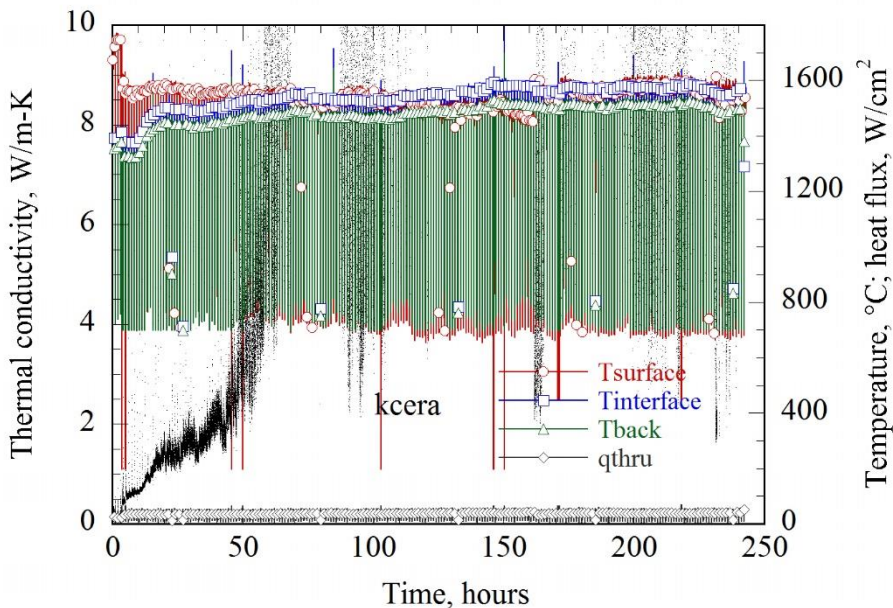
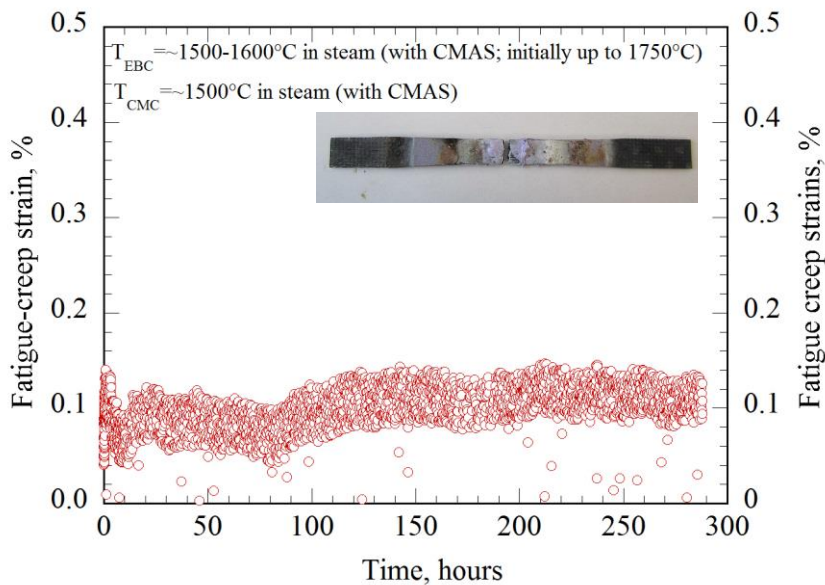
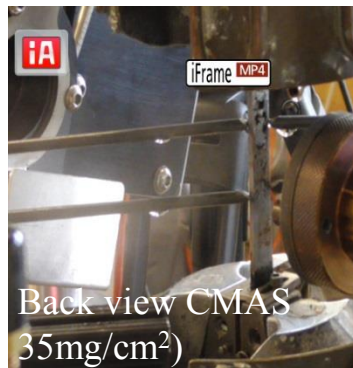
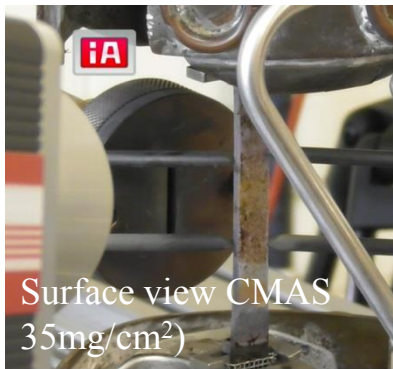
- An alternating HfO_2 -and RE-silicate coatings (EB-PVD processing) – HfO_2 - layer infiltration and rare earth silicate layer melting



EB-PVD Processed EBCs: alternating HfO_2 -rich and ytterbium silicate layer systems for CMAS and impact resistance

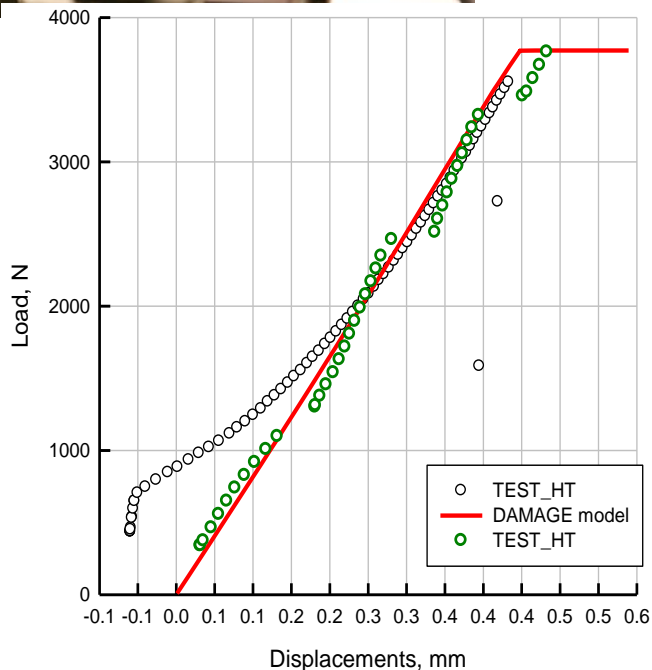
Advanced EBC-CMC Fatigue Test with CMAS and in Steam Jet: Tested 300 h Durability in High Heat Flux Fatigue Test Conditions

- Advanced Hf-NdYb silicate-NdYbSi bond coat EBC coatings on 3D architecture
CVI-PIP SiC-SiC CMC (EB-PVD processing)

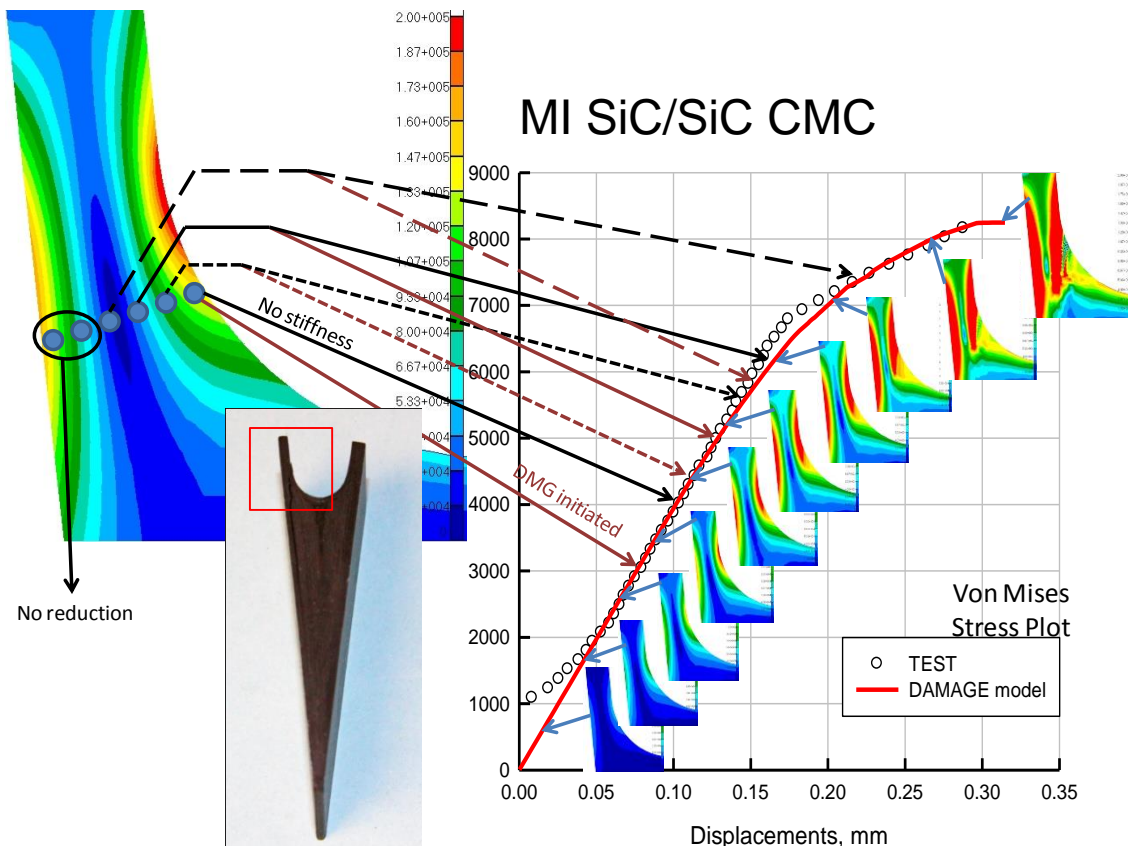


SiC/SiC Turbine Airfoil Trailing Edge Tests

- Subelement wedge testing, by applying trailing-edge element opening stresses for simulating high pressure turbine airfoil stress conditions, aiming at understanding the CMC and EBC degradation



Subelement Load-Displacement curve – CVI CMC trailing edge



Subelement Load-Displacement curve – Prepreg MI CMC trailing edge



Summary and Future Plans

- **Advanced high heat flux creep rupture, fatigue rigs established for simulated turbine engine EBC-CMC testing**
 - High temperature comprehensive environment testing capability including heat flux, steam and CMAS, at very high temperature
 - Real time coating degradation monitoring and fatigue-creep stain monitoring
 - Testing capabilities incorporated into the advanced EBC-CMC developments
- **Long term creep rupture and fatigue behavior evaluated for Hafnium Rare Earth silicate and Rare Earth-Silicon based EBCs-CMCs at 1482°C+ (2700°F+)**
 - Crucial for advanced EBC-CMC development and validations
 - Advanced EBC coated 3D architecture SiC/SiC CMCs tested at 1500°C in steam and CMAS environments
 - Compared to baseline materials
- **The heat flux thermomechanical testing of subelements for the EBC-CMC subelement**
 - Important for durability and life modeling

Future plans

- HCF high heat flux rig with additional environmental testing capabilities (steam-air mixture environments and controlled steam or vacuum capabilities)
 - EBC erosion-impact capabilities also planned in combination of laser high heat flux, creep-fatigue, high velocity steam, and CMAS integrated tests
 - Additional full field strain measurement experiments, in particular at high temperatures
 - Planned a multi-axial testing rig for CMC and EBC testing
-



Acknowledgements

- The work was supported by NASA Fundamental Aeronautics Program (FAP) and Transformational Tools and Technologies Projects

NASA colleagues include:

Dr. Kang N. Lee, for helpful discussions

Sue Puleo and Rick Rogers – X-ray

Terry McCue, Serene Farmer, Francisco Solá, SEM and TEM

Valerie Wiesner and Narottam Bansal, Gustavo Costa: Fundamental CMAS behavior