Thermomechanical and Environmental Durability of Environmental Barrier Coated Ceramic Matrix Composites Under Thermal Gradients

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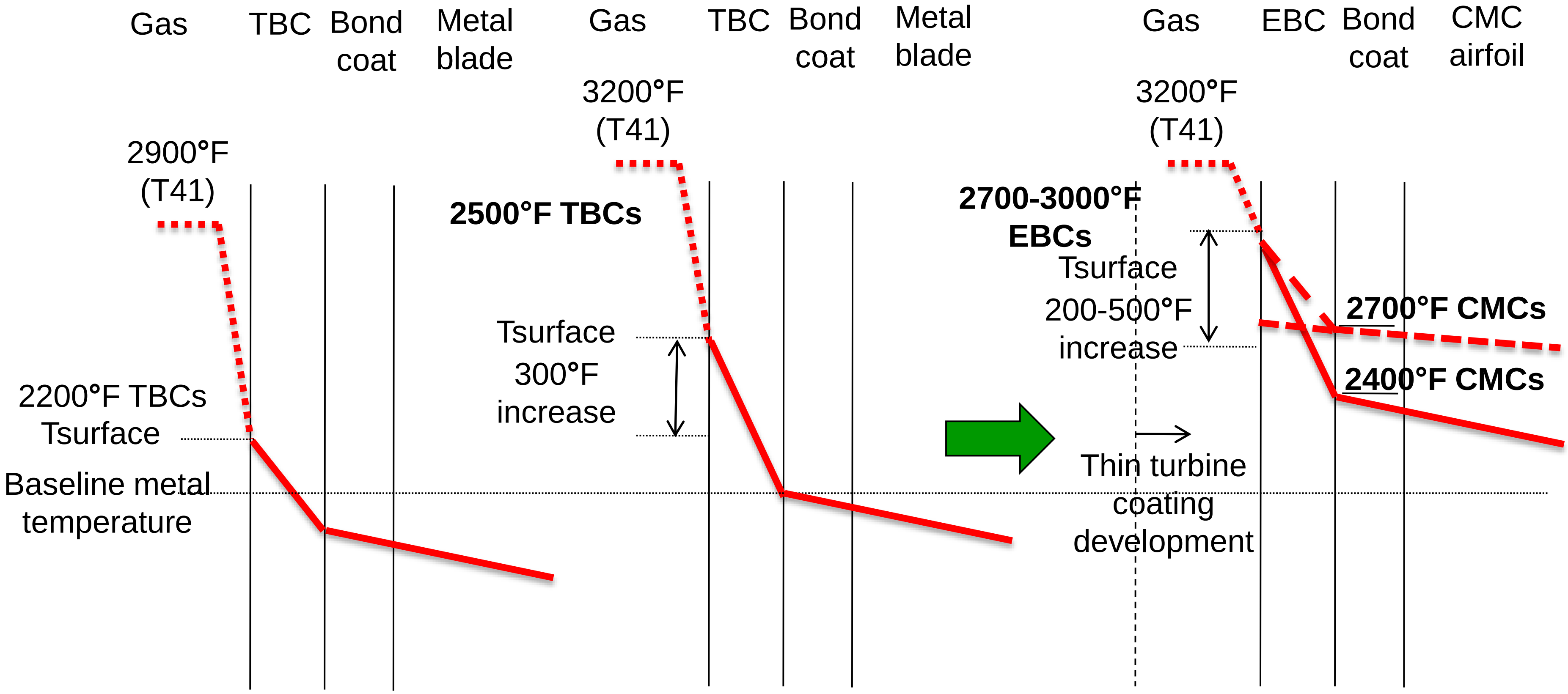
NASA Turbine Environmental Barrier Coatings for CMC-EBC Systems

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



Gas
TBC
Bond coat
Metal blade
2900°F (T41)
2200°F TBCs Tsurface
Baseline metal temperature

Gas
TBC
Bond coat
Metal blade
3200°F (T41)
2500°F TBCs
Tsurface 300°F increase

Gas
EBC
Bond coat
CMC airfoil
3200°F (T41)
2700-3000°F EBCs
Tsurface 200-500°F increase
2700°F CMCs
2400°F CMCs
Thin turbine coating development

Current metal turbine airfoil system

State of the art metal turbine airfoil system 2500°F TBCs

2700-3000°F CMC turbine airfoil systems

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 CO_2 laser rig development
  - Thermal conductivity
  - Cyclic durability and monitoring degradations of EBCs and CMCs

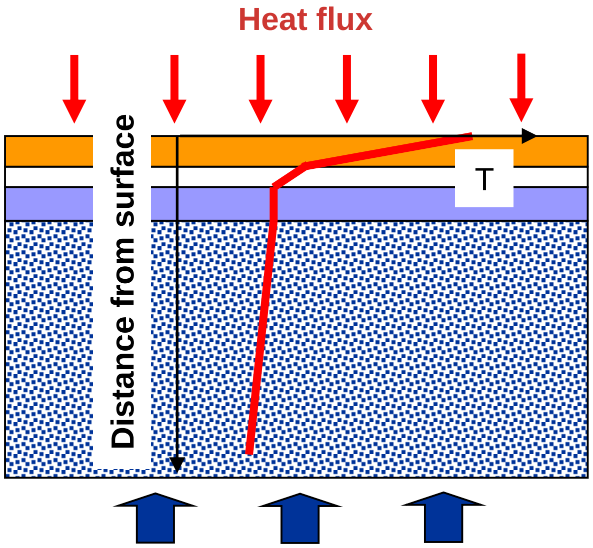
- **Laser high heat flux and mechanical tests**
  - Combined high heat flux - mechanical tests
  - High heat flux biaxial creep/fatigue test rigs
  - Sub-element testing

- **Summary and future directions**

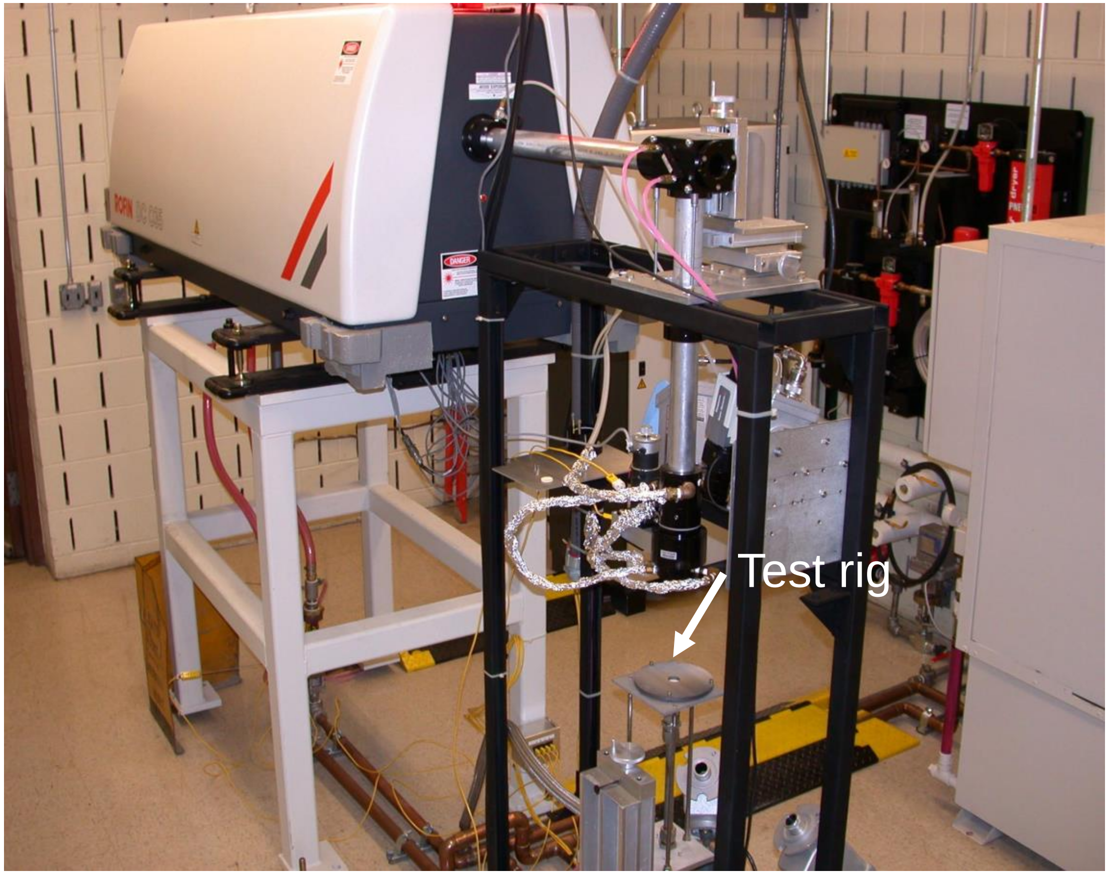
High Power CO_2 Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

- Developed in 1990's, the rig achieved turbine level high-heat-fluxes (315 W/cm^2) for turbine thermal barrier coating testing
- Crucial for advanced EBC-CMC developments

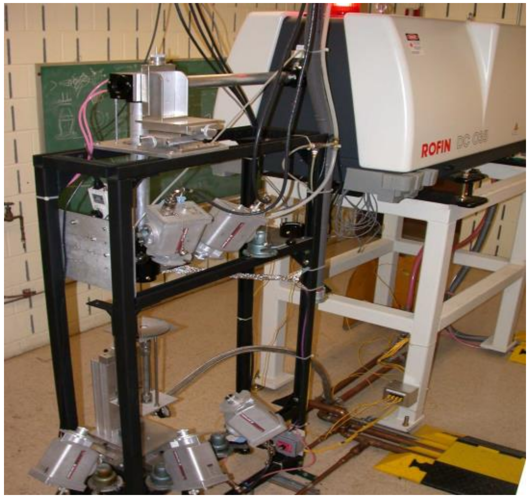
Turbine: 450°F across 100 microns
Combustor:1250°F across 400 microns



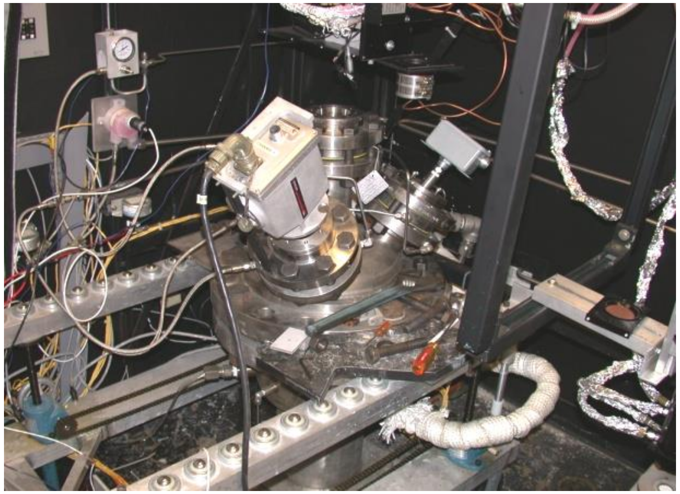
Cooling – high velocity air or air-water mist
Achieved heat transfer coefficient 0.3 W/cm^2-K



High Power CO_2 Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development - Continued



- NASA high power CO_2 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)

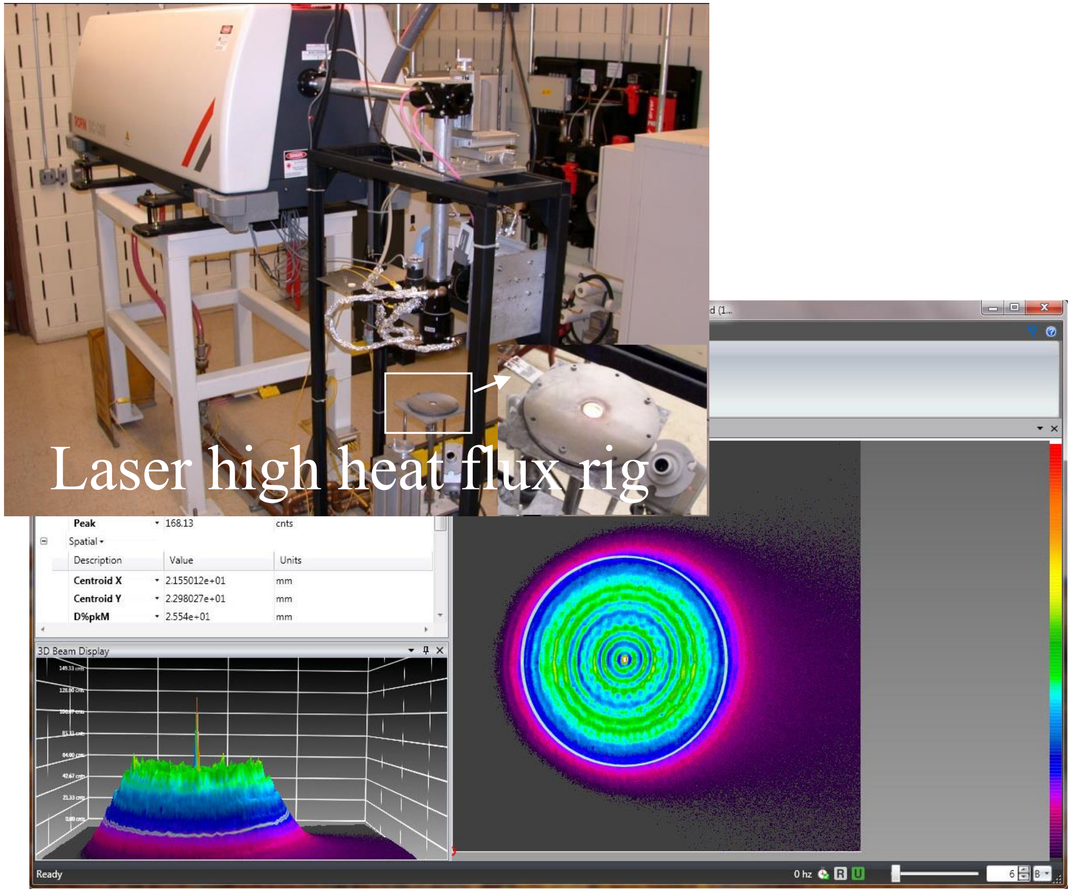


Some temperature thermal gradient cycles

High heat flux combustor rig

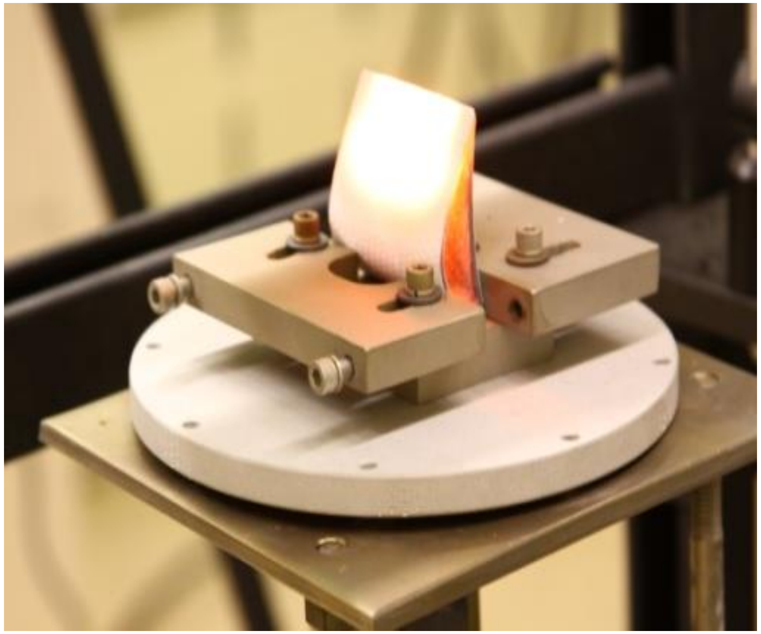
High Power CO_2 Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development – Continued

- 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



Description	Value	Units
Peak	168.13	cnts
Spatial		
Centroid X	2.155012e+01	mm
Centroid Y	2.298027e+01	mm
D%pkM	2.554e+01	mm

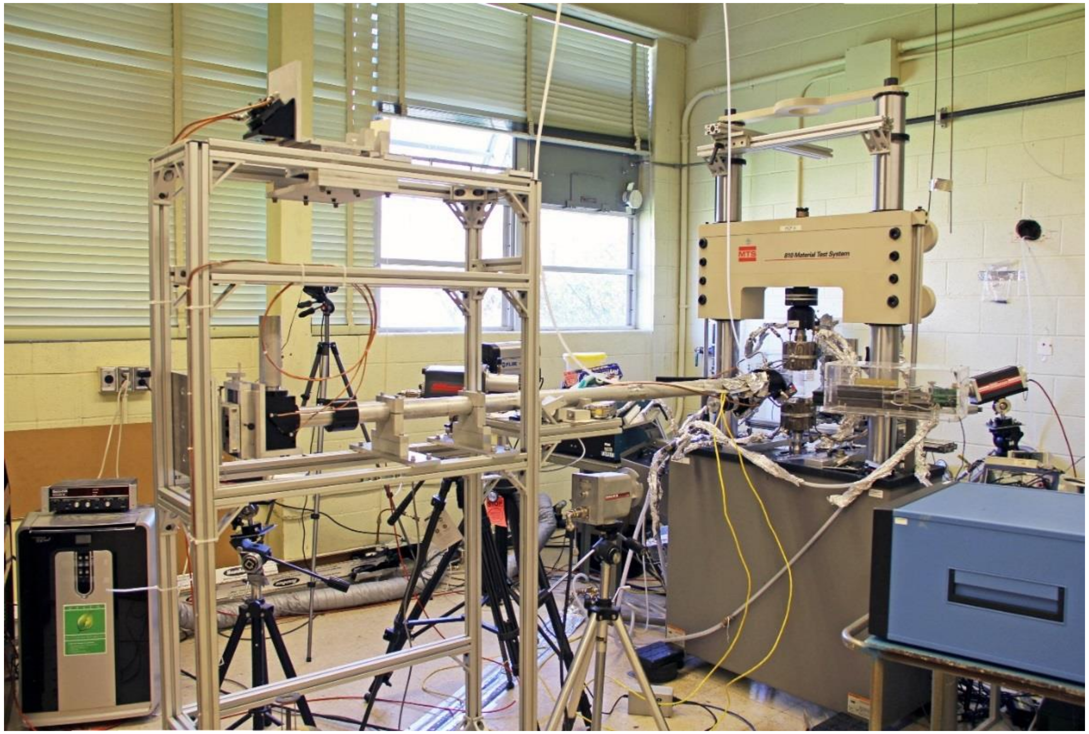
Example of 1” diameter disc specimen tests and beam profile



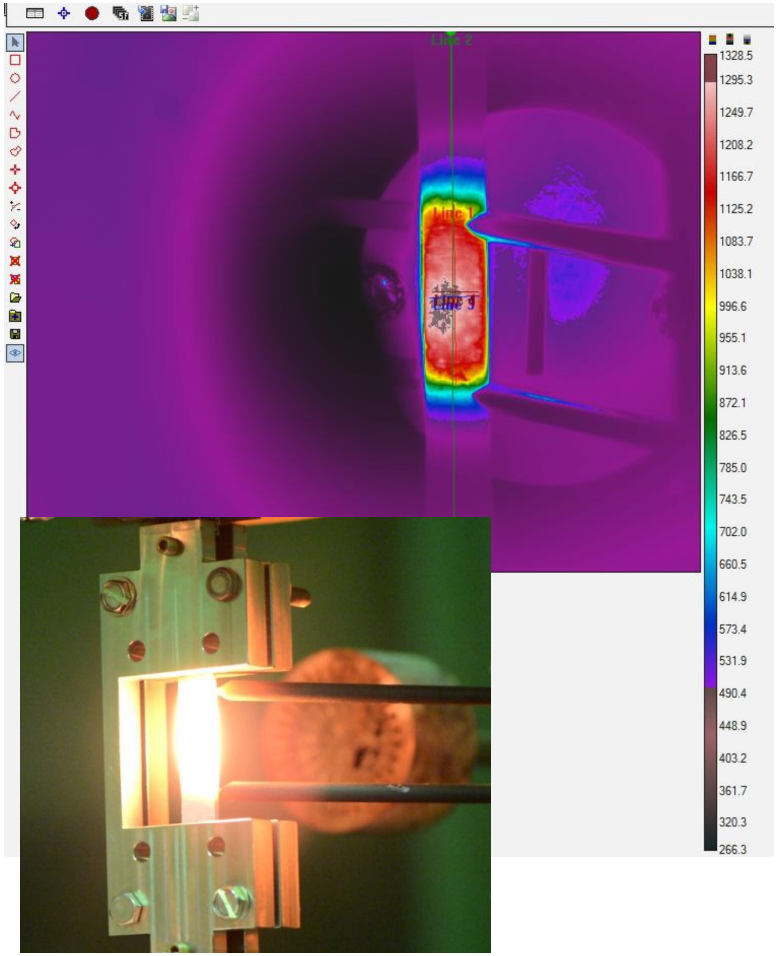
2” beam size subelement tests

High Power CO_2 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

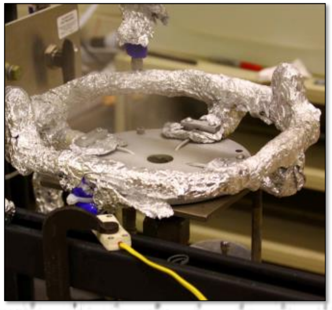
High Heat Flux Rig Testing with Water vapor Steam Chamber – Established in Early 2000

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

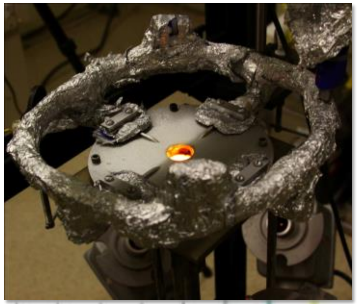
Laser heat flux water vapor test rig

Specimen holder and water vapor jets

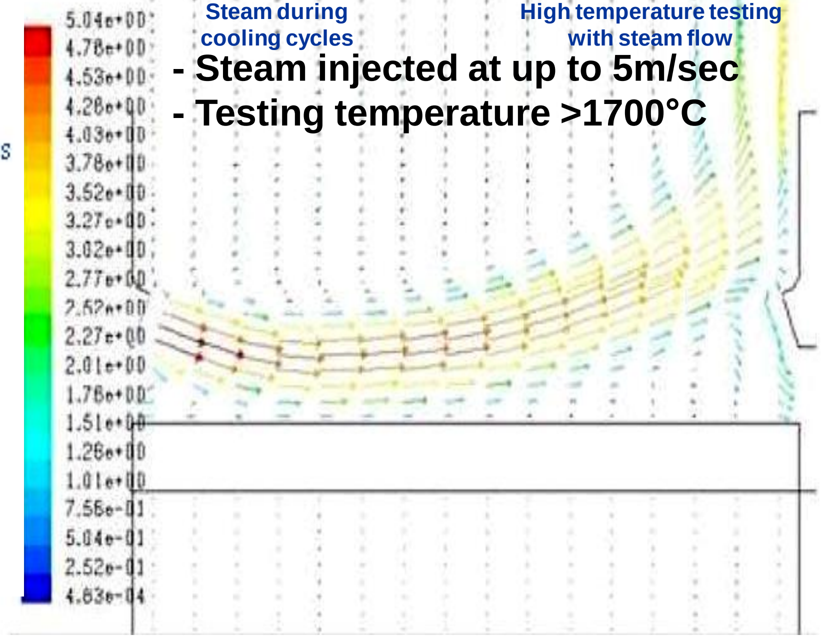
Specimen under testing



Steam during cooling cycles



High temperature testing with steam flow



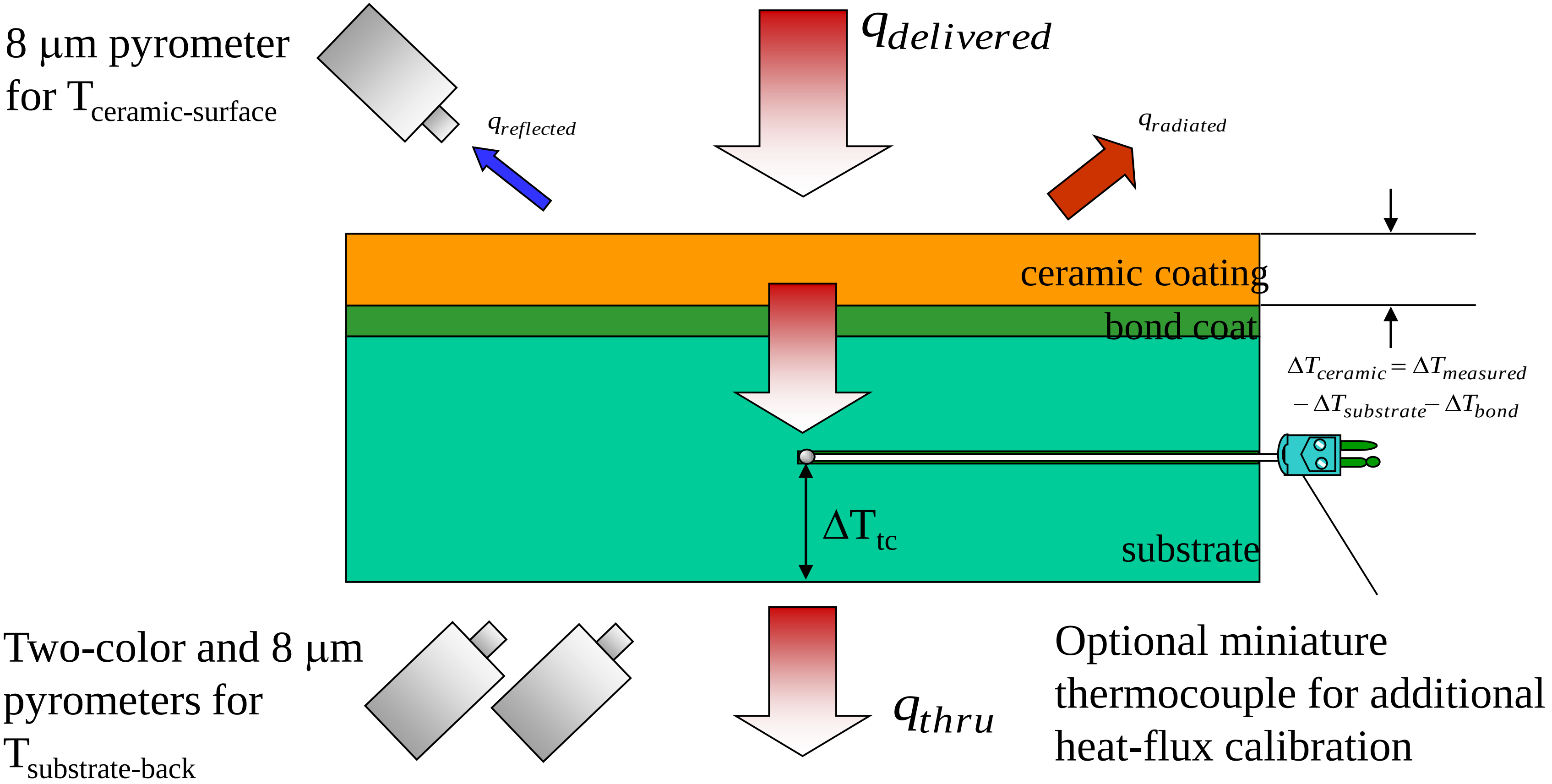
- Steam injected at up to 5m/sec
- Testing temperature >1700°C

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

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

Where

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



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

$q_{delivered}$

$q_{reflected}$

$q_{radiated}$

ceramic coating

bond coat

substrate

$\Delta T_{ceramic} = \Delta T_{measured} - \Delta T_{substrate} - \Delta T_{bond}$

ΔT_{tc}

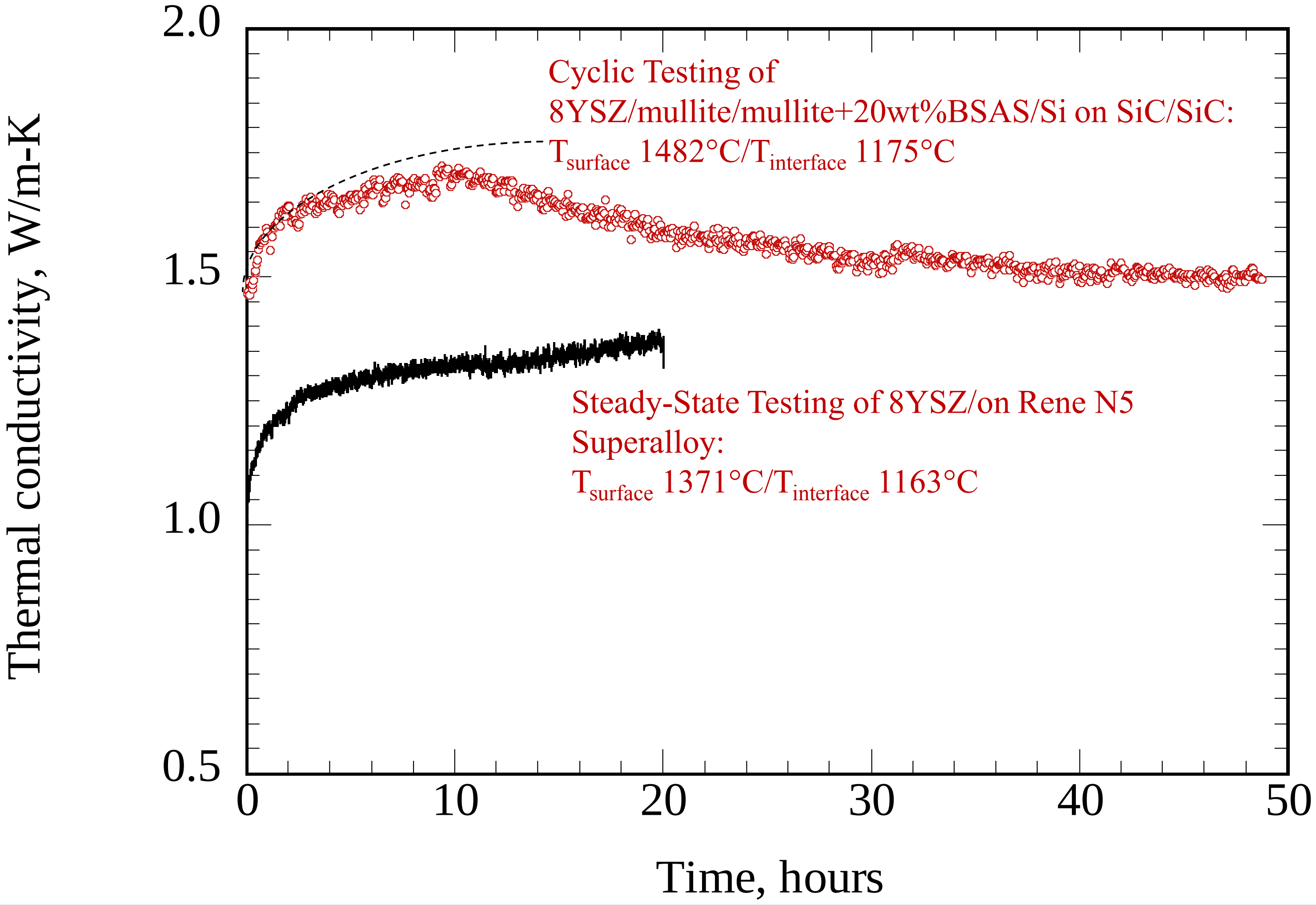
Two-color and 8 µm pyrometers for $T_{\text{substrate-back}}$

q_{thru}

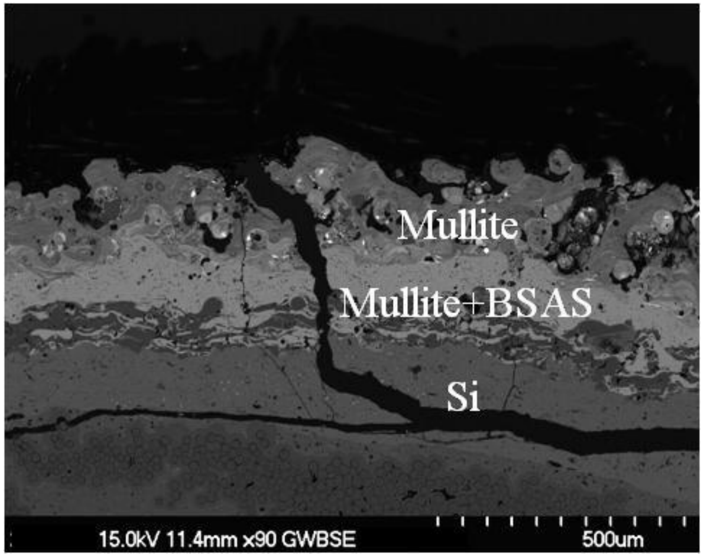
Optional miniature thermocouple for additional heat-flux calibration

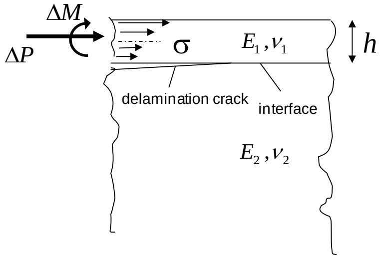
Thermal Gradient Cyclic Behavior of a Thermal Environmental Barrier Coating System

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



Environmental Barrier Coating and High Heat Flux Induced Delaminations

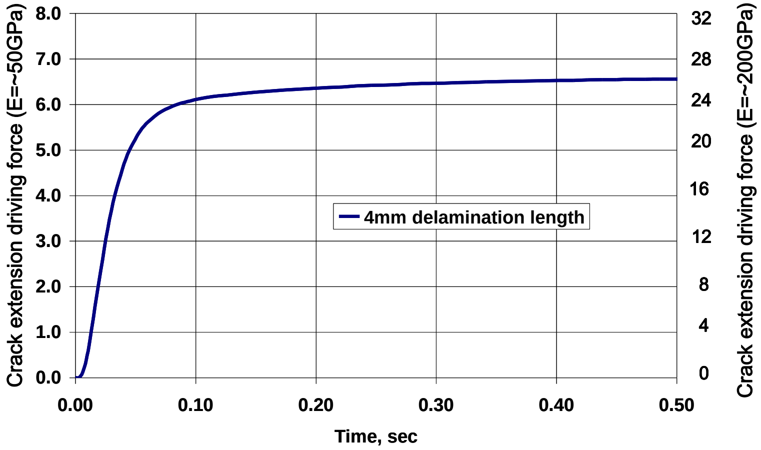




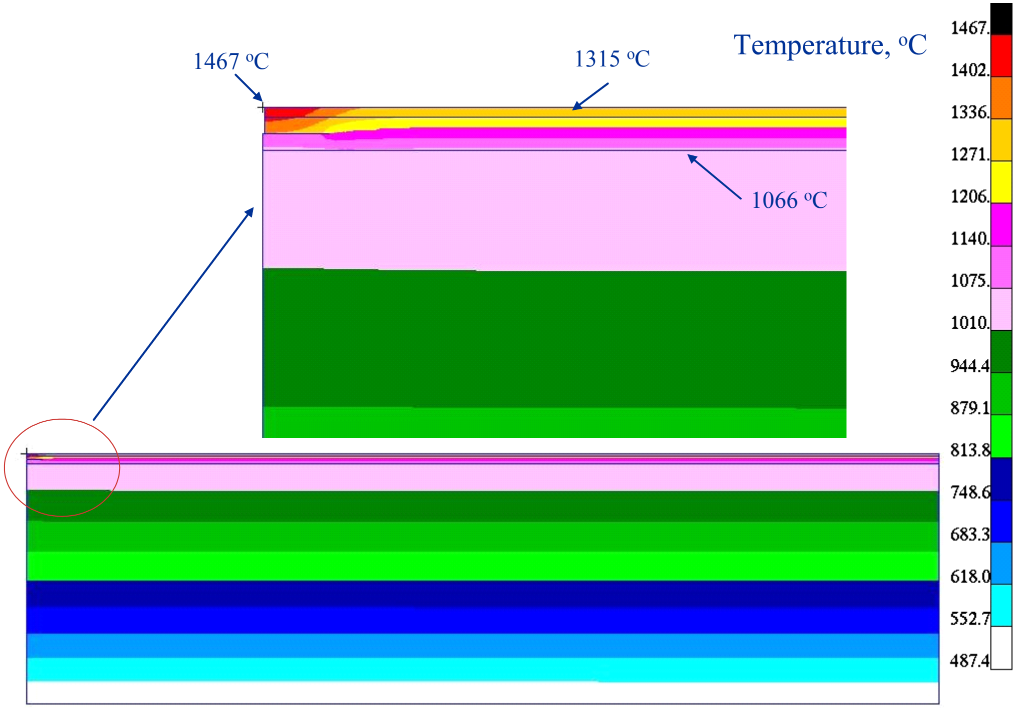
$$G = \frac{1}{6}\left(\frac{1+\nu_1}{1-\nu_1}\right)E_1 h\left(\alpha_1\left(T_S - T_0\right)\right)^2$$

Evans and Hutchinson model, Surface Coating Technology, 2007

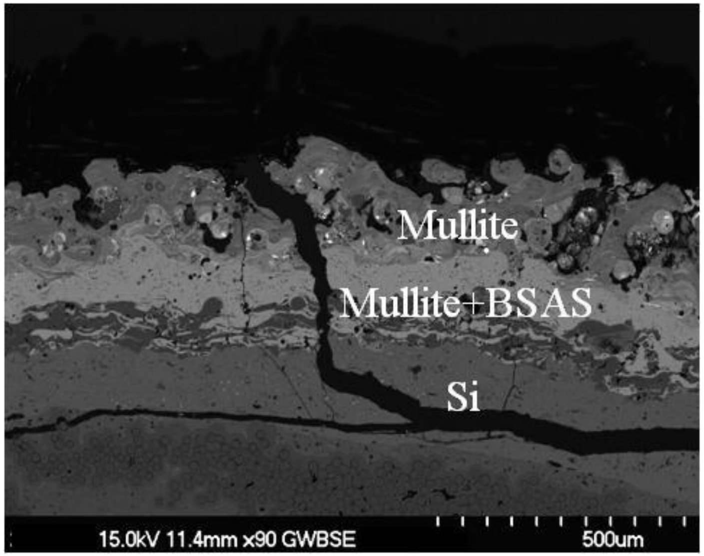
Crack Extension Force G as a function of time for 2.0mm half delamination length and crack depth of 0.08mm

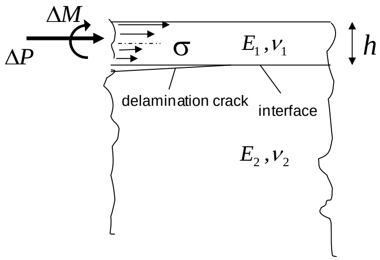


The FEM model



Environmental Barrier Coating and High Heat Flux Induced Delaminations





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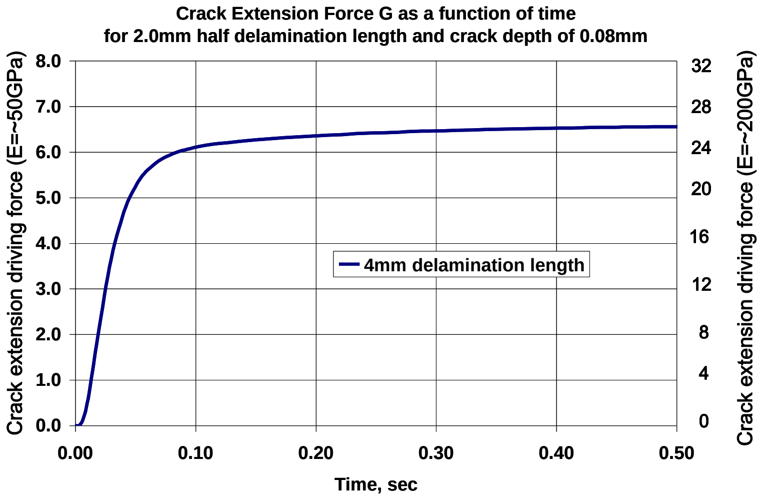
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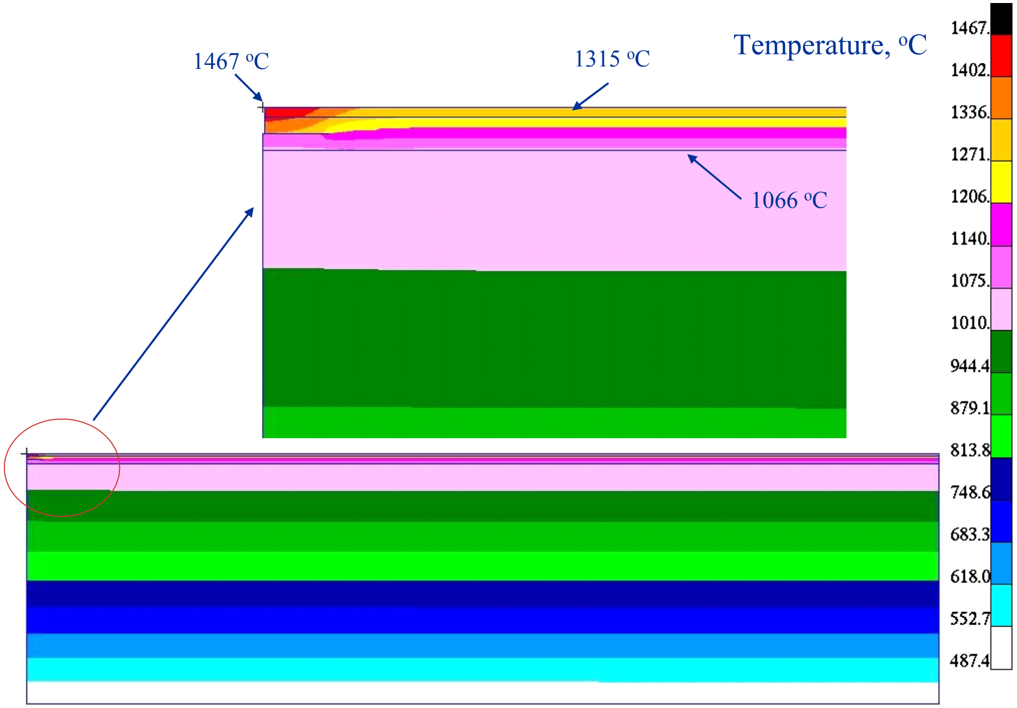
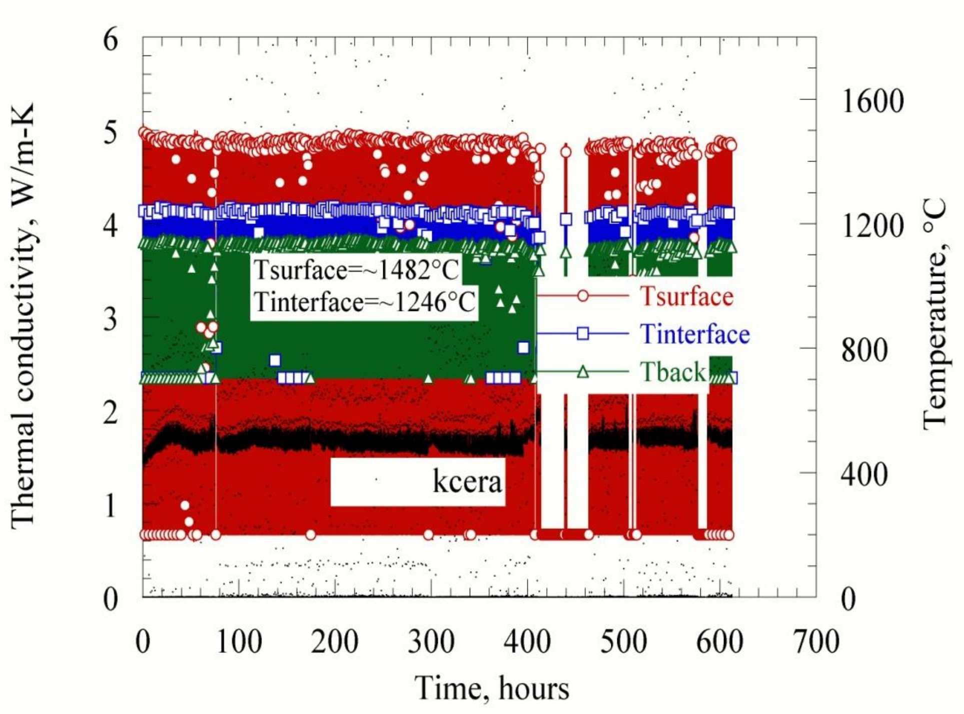


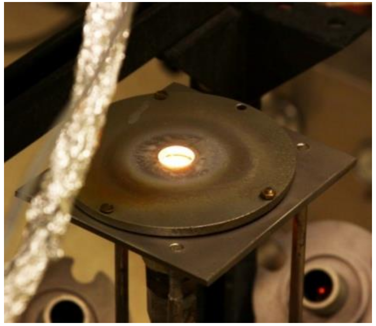
The Long-Term Durable CMC Coating System Testing under High Heat Flux Conditions

- HfO_2/Hf-Gd-Yb-Y-aluminosilicate/$Yb_2Si_2O_7$-BSAS/Si environmental barrier coating on SiC/SiC successfully demonstrated 500 hr high-heat-flux durability at 2700° F

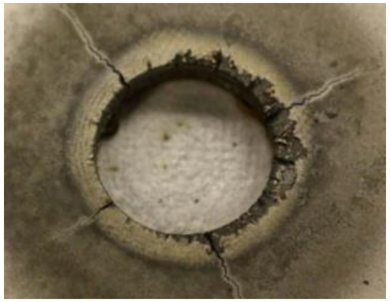


Laser high-heat-flux testing for the environmental barrier coating: surface temperature ~2700° F (1482° C), ceramic coating/CMC interface temperature ~2300° F (1260° C), CMC back ~2100° F (1150° C), with 1 hr hot time cycles; coating thermal conductivity met initial design goal

Laser high-heat-flux test rig for advanced environmental barrier coating development



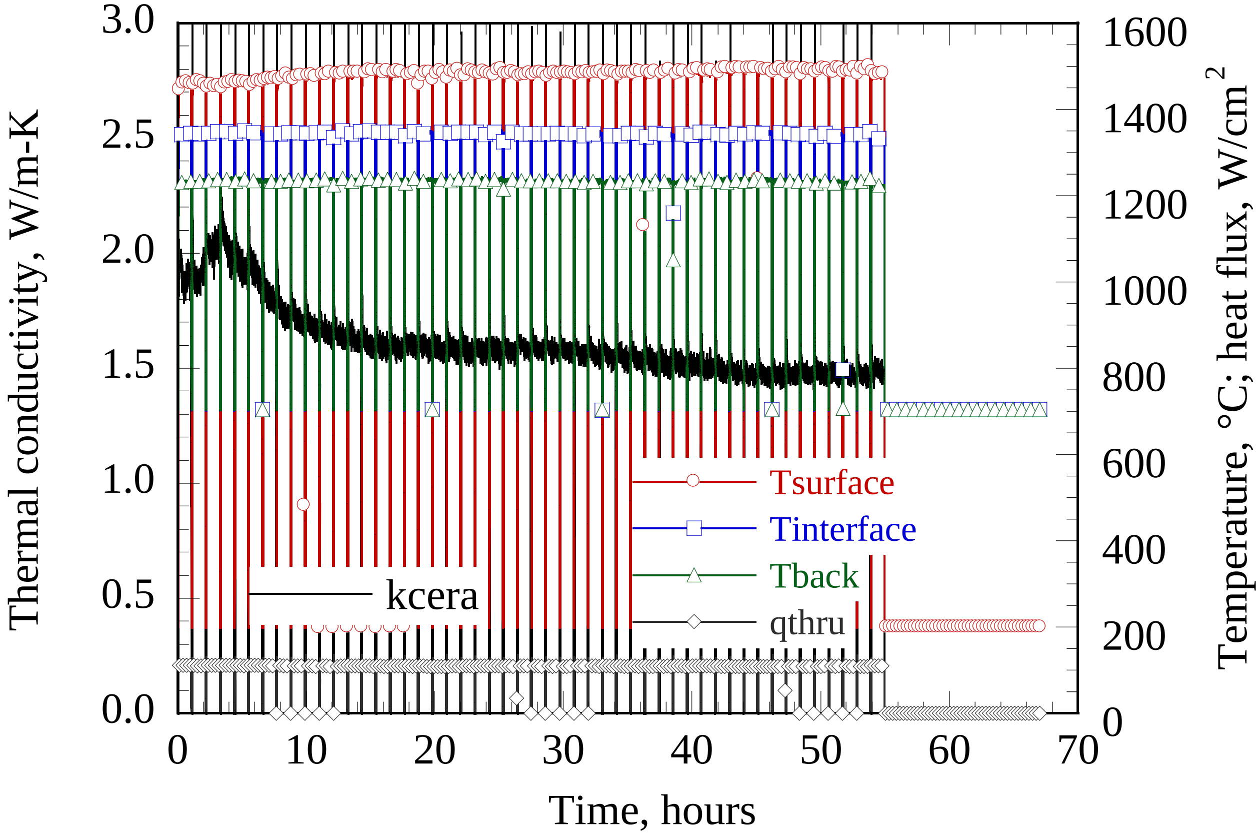
The EBC-CMC specimen under testing

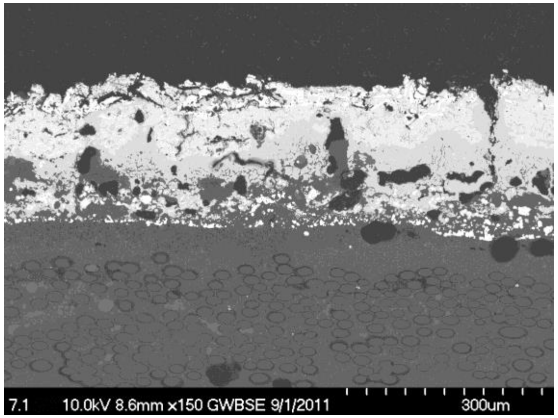


Tested EBC-CMC specimen tested for 500 hr durability

Thermal Gradient Cyclic Behavior of Air Plasma Sprayed Yb_2SiO_5 (with HfO_2 Composite)/$Yb_2Si_2O_7$/HfO_2-Si Coatings on SiC/SiC CMCs

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

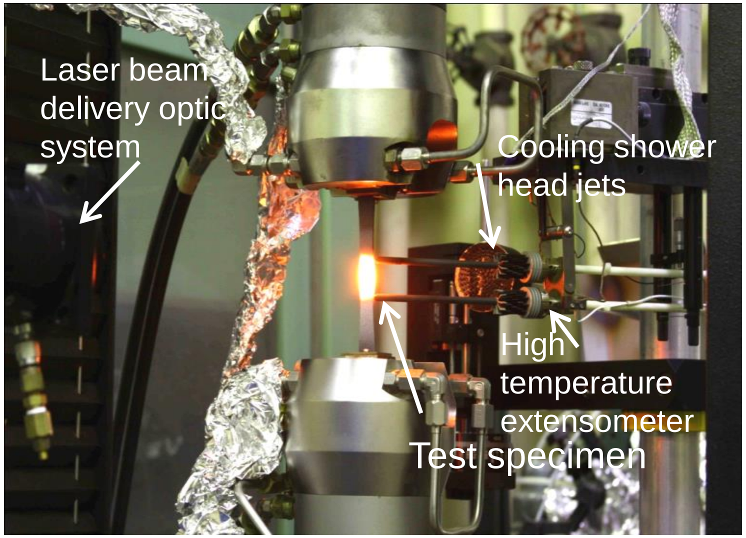




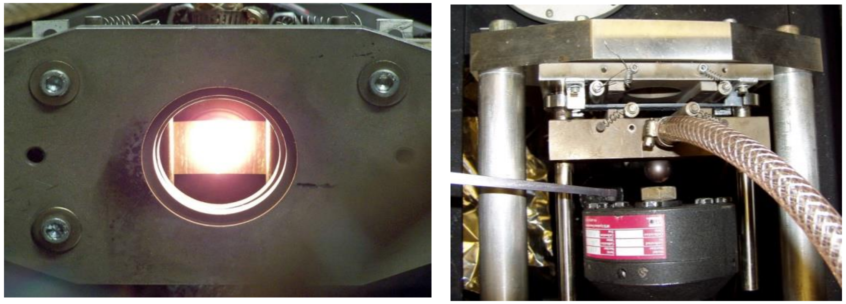
After 50hr Cyclic Testing

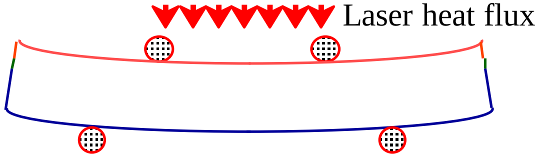
High Heat Flux Thermomechanical Testing for EBC Development

- High heat flux and combined thermal-mechanical loading capabilities established to allow SiC/SiC system performance data to be obtained under simulated operating conditions
- A 1000 Hz high heat flux HCF testing rig is being established this year



High heat flux tensile TMF and rupture testing

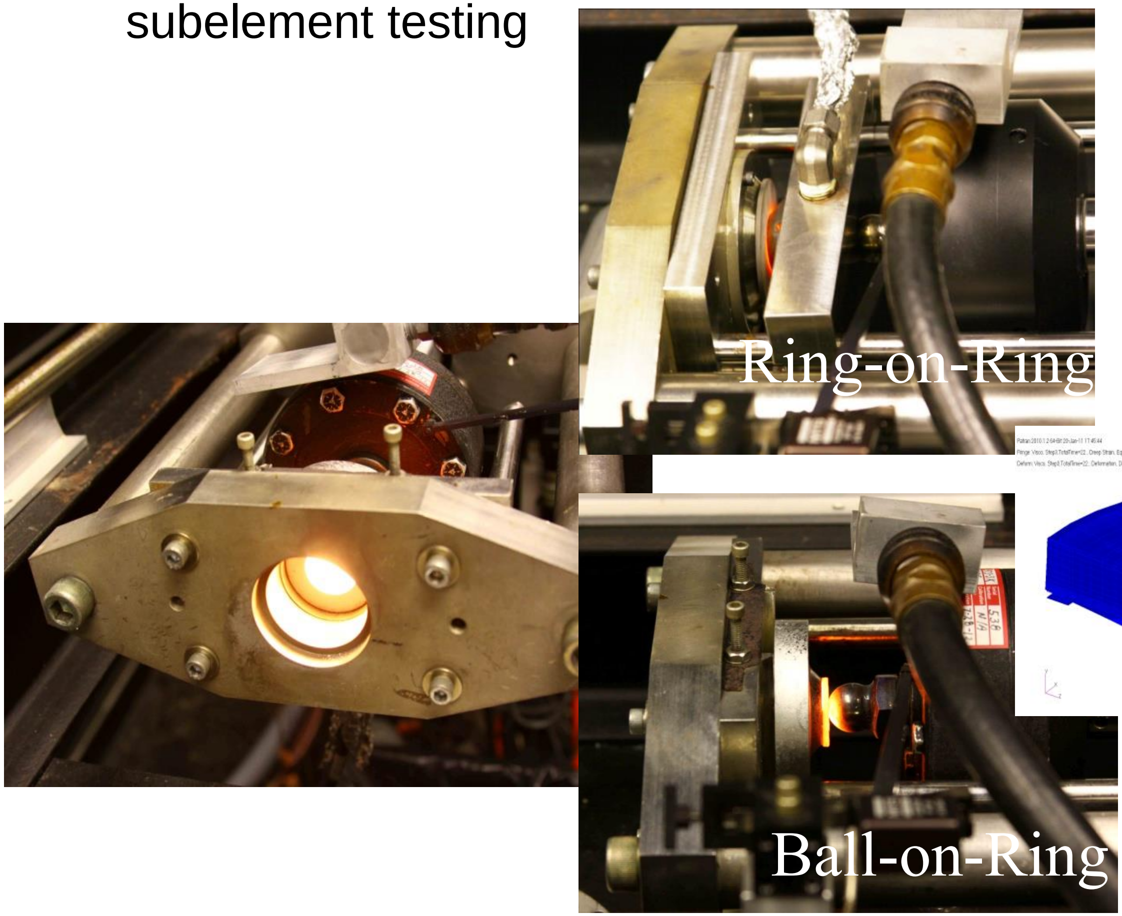


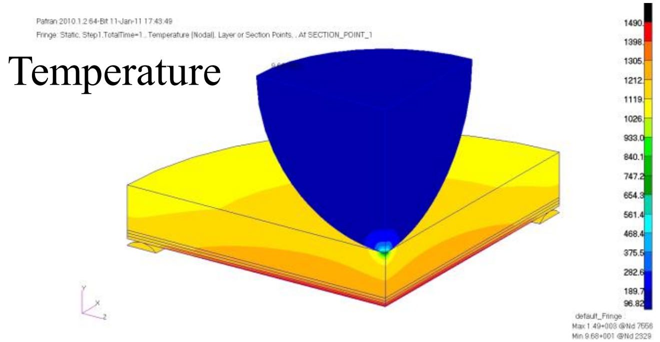


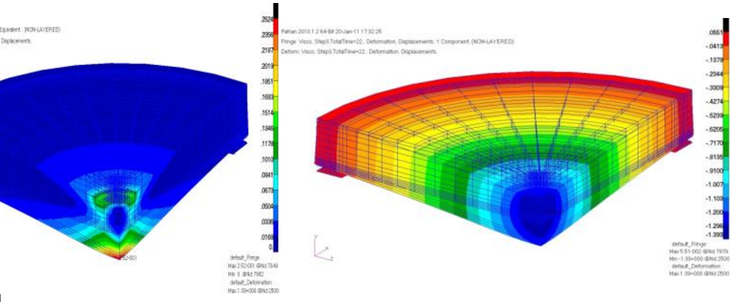
High heat flux flexural TMF testing: HCF, LCF, interlaminar and biaxial strengths

High Heat Flux Biaxial Testing for EBC Development

- Allows very high temperature, high heat flux cooled thermal gradient testing of CMC-EBC under engine equivalent biaxial stress conditions
- Capable of fatigue testing up to 100 hz
- Accommodates 1” diameter and 2” diameter disc test specimens, and subelement testing



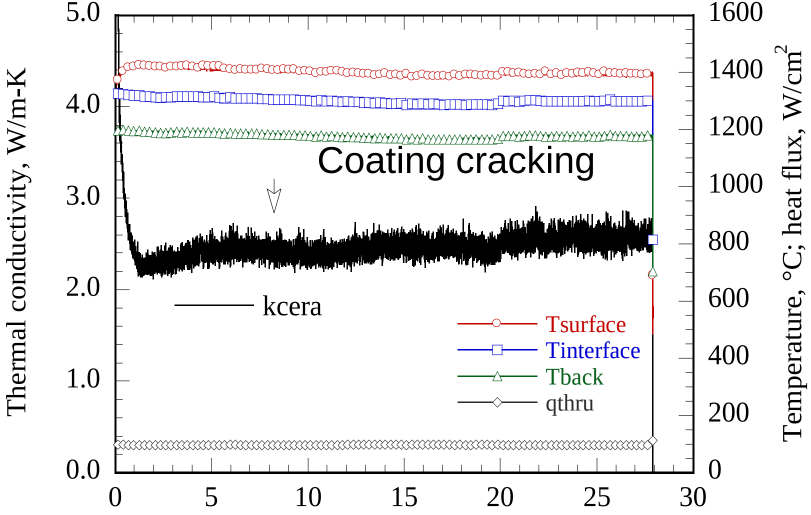


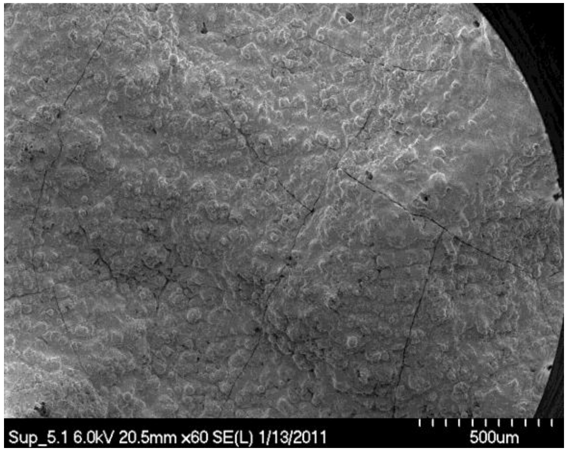


Axial total creep displacements under heat flux testing can be measured, and modeled by FEM analysis

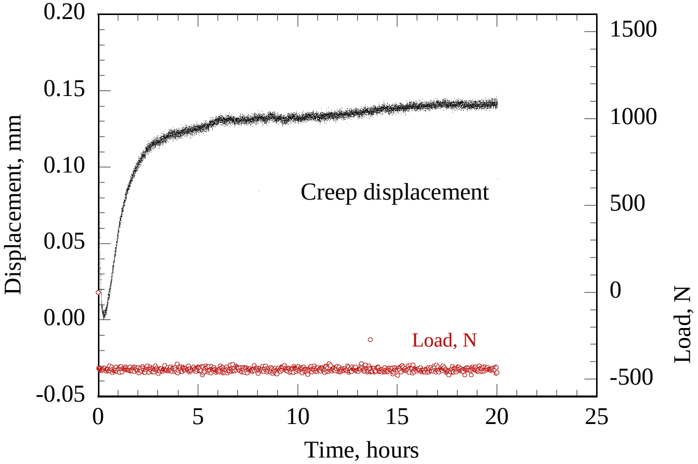
A Two-layer Yb_2SiO_5/$Yb_2Si_2O_7$ Ytterbium Silicate EBC on SiC-SiC CMC

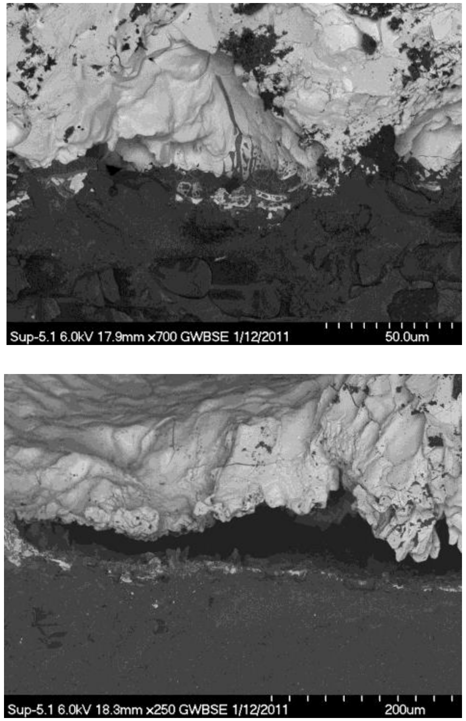
- Tested $T_{surface}$1420°C and $T_{interface}$ 1315°C, load 445 N (stress ~200 MPa)
- Excellent correlations between thermal conductivity and creep strain response due to coating failure





Surface

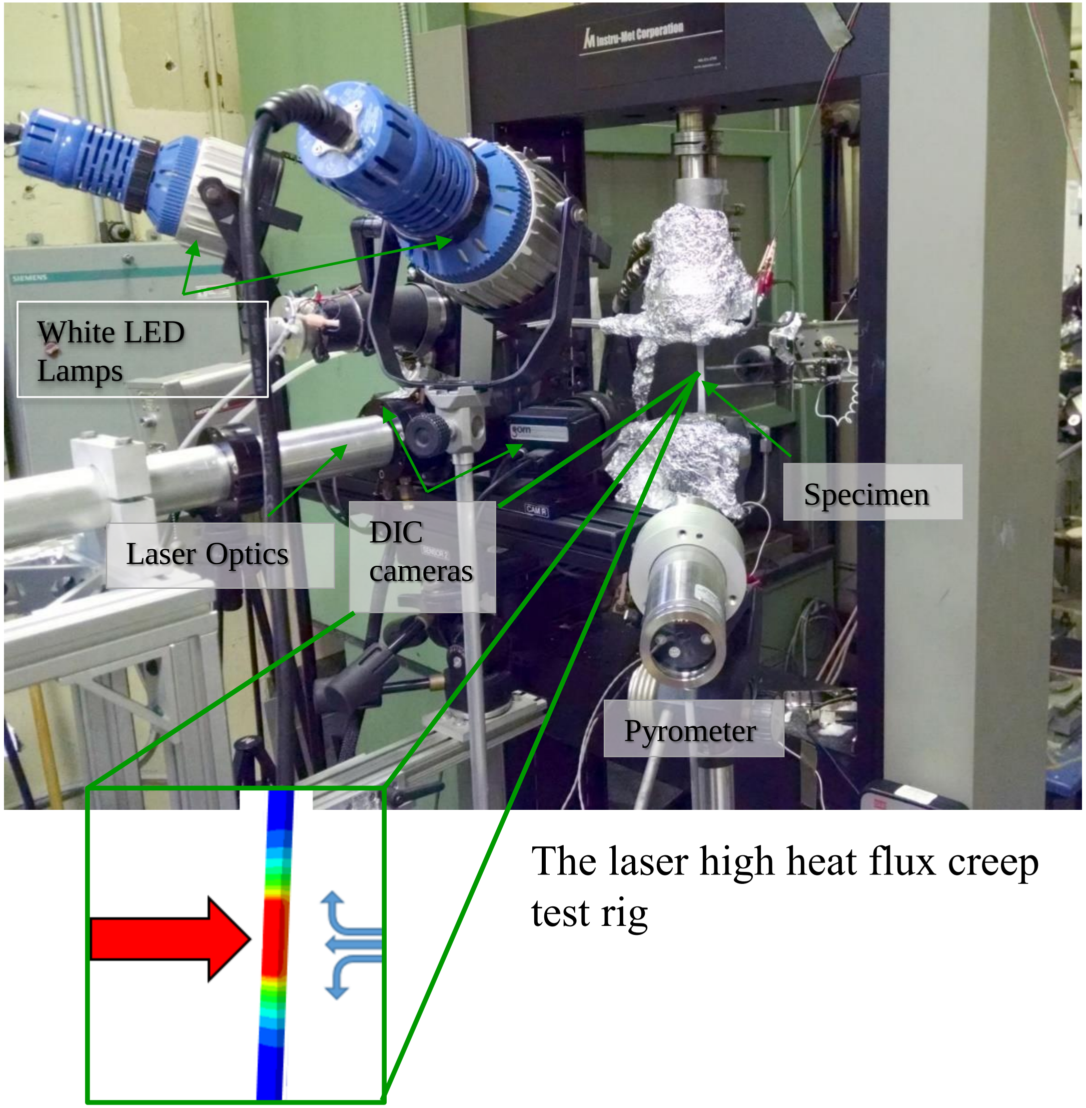




Cross sections

EBC Coated CMC Rupture Strength Tests under Heat Flux Using Digital Image Correlation (DIC) Strain Measurements

- A coated CVI-MI specimen shown in heat flux uni-axial tension rig
- Digital Image Correlation (DIC) is used to determine localized strain fields at high temperatures
- Using Y_2O_3 paint
- Acoustic Emission (AE) and Electrical Resistance (ER) also incorporated

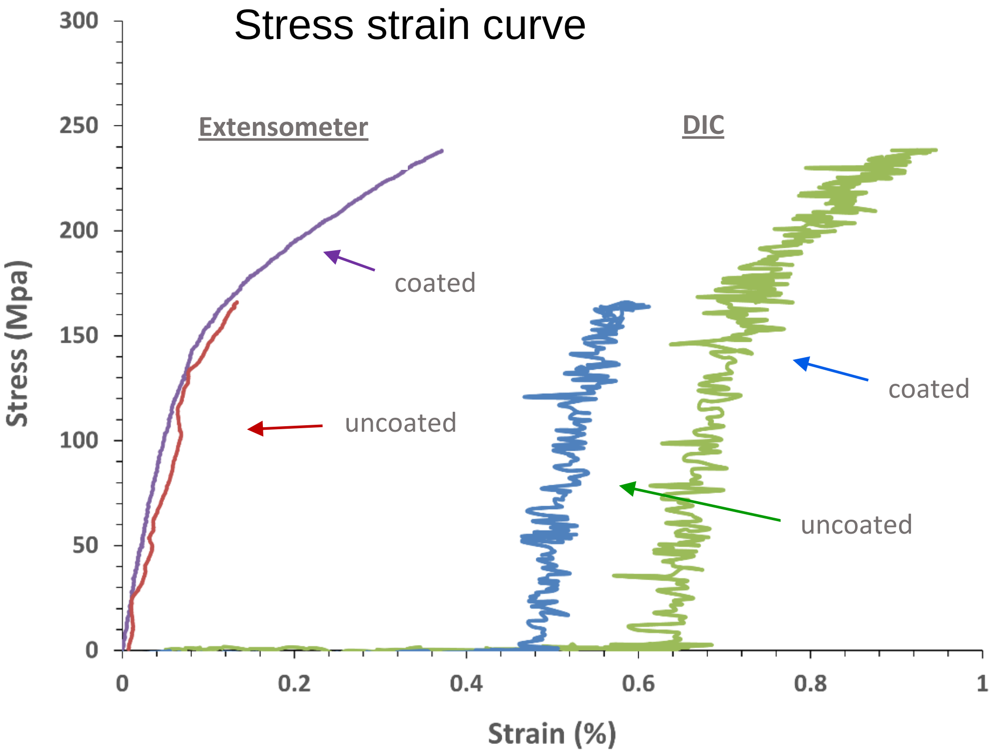


The laser high heat flux creep test rig

With Matt Appleby et al, Surface and Coatings Technology, 284 (2015) 318–326.

High Pressure Burner Rig Pre-exposed EBC-CMC Specimens (10 atm, 2400°F, 30 h) Fast Strength Tested in the Laser Heat Flux Tensile Rig at High Temperature

- The EBC HfO_2-Si coated CVI-MI CMC specimen shown near intact
- As comparison, the uncoated CVI-MI specimen exposed indicates more severe degradation of composite properties
- Oxidation and embrittlement of the MI-CVI CMC in HPBR lead to the lowers strength of the uncoated specimen



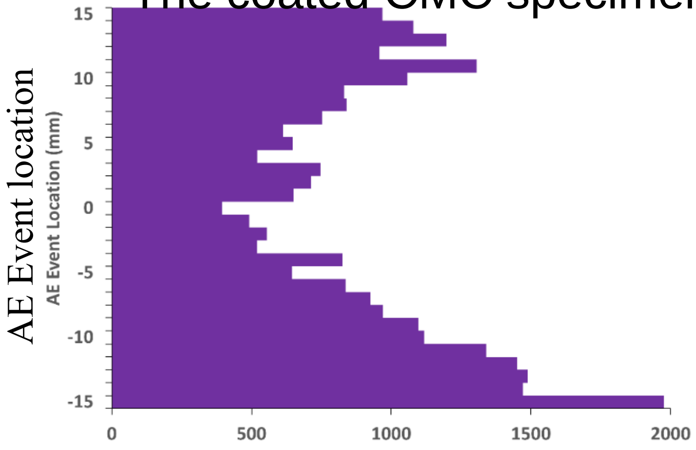
With Matt Appleby et al, Surface and Coatings Technology, 284 (2015) 318–326.

Specimen	Surface Temp. (°C)	Back Temp. (°C)
coated	1230	1070
uncoated	1200	1010

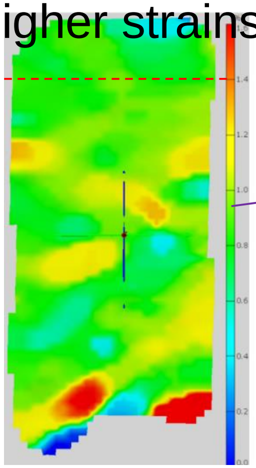
Specimen	E (GPa) Extensometer	E (GPa) DIC	σ_{UTS} (MPa)	ε_{fail} (%)
coated	241	266	238	0.371
uncoated	146	221	166	0.134

AE Waveform Analysis and DIC Failure Maps

- Energy distribution of AE events compared in specimen gage section with corresponding DIC strain mapping at failure stress
- The coated CMC specimen showed higher strains and AE event energies



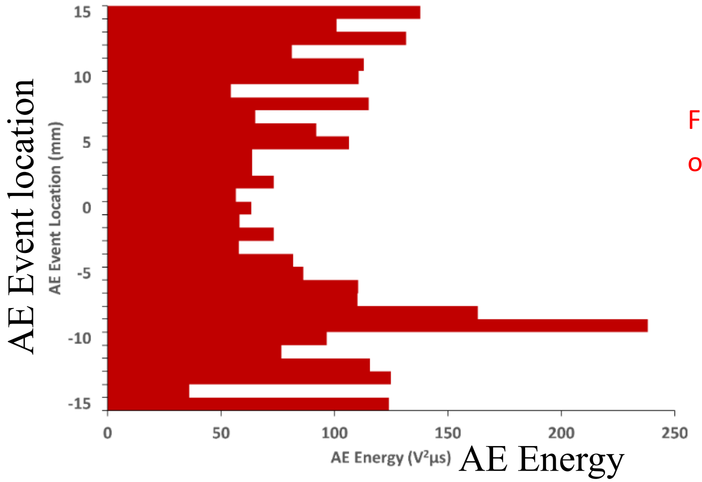
Failure Plane



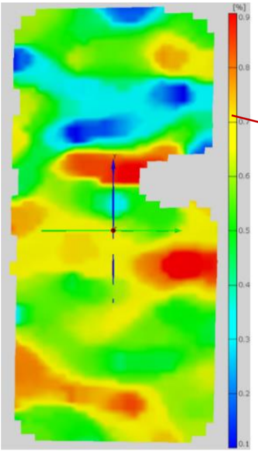
COATED

Nominal thermomechanical strain: 0.96%

🟩



Failed below area of DIC interest



UNCOATED

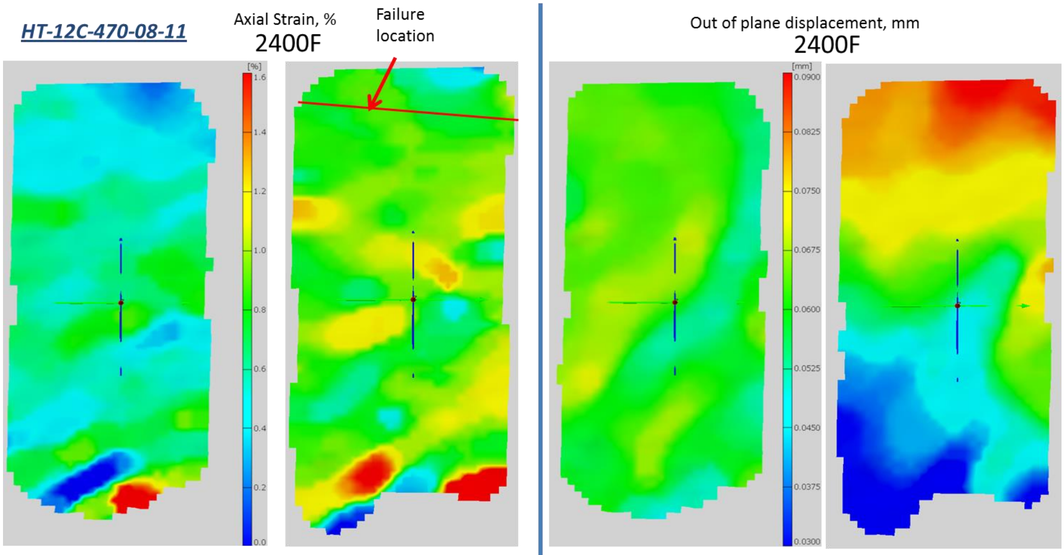
Nominal thermomechanical strain: 0.71%

🟨

With Matt Appleby et al, Surface and Coatings Technology, 2015

DIC Full-Field Strain Measurements in Heat Flux Tensile Tests

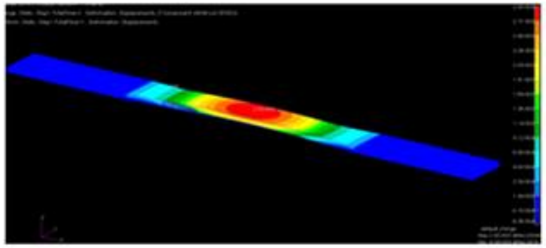
– DIC strain measurements of heat flux thermal gradient tensile testing



After heat up Just prior to failure After heat up Just prior to failure

- Thin EBC HfO_2-Si CVI-MI CMC
- Axial, in-plane strains ~ 0.96%
- Out of plane deflection ~ 0.05-0.09 mm
- Tests help validate FEM models

Example of EBC-CVI-SMI CMC tensile loading in-plane (axial) and out-of-plane strain distribution



Modeling of Heat-Flux Tensile Creep associated bending

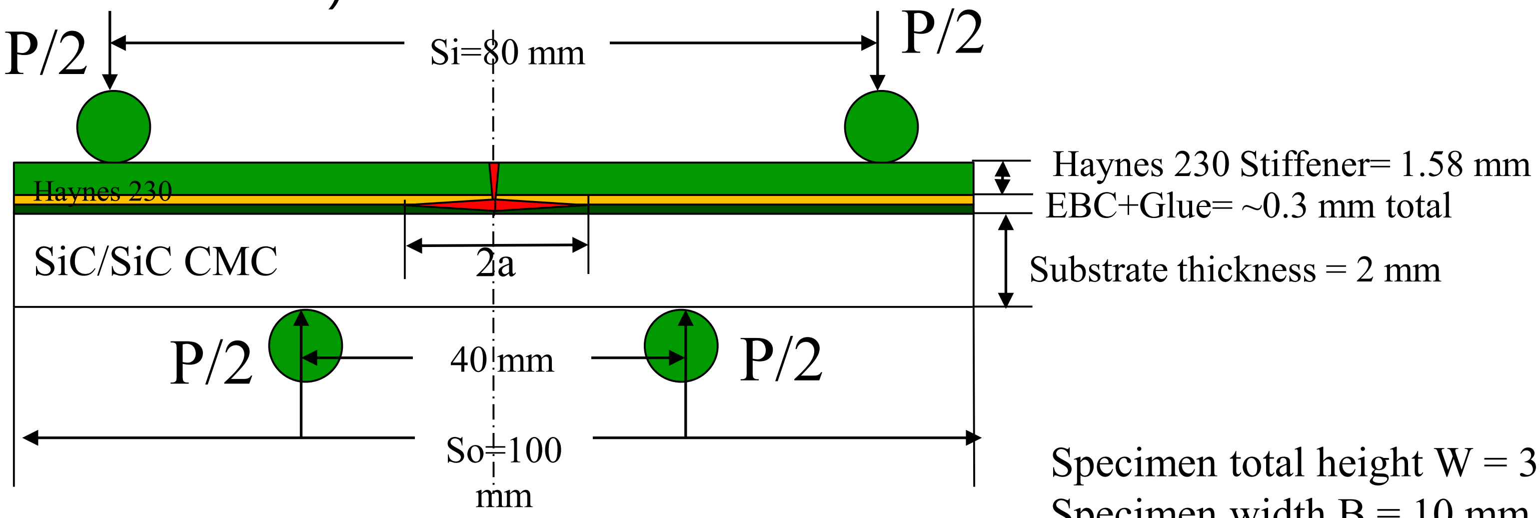
Side view of thicker EBC coated tensile specimen as heated. Top=1450C, Bottom=1200C, intermediate=1300C

Deformation under P=5400N. Due to bending, bottom surface carries higher load and EBC finally is in tension.

FEM-modeling of thermal gradient tensile specimen heating bending: Multilayer EBC coated tensile specimen modeled to help understand the EBC stress distributions in thermal gradient heating and mechanical tensile stress conditions, validating using DIC

EBC Delamination and CMC Interlaminar Toughness Testing and Modeling under Heat Flux Conditions

- Test configuration (Haynes alloy stiffener-Optional)/EBC (Ytterbium Silicate-Si/2D SiC/SiC CMC)



Specimen total height W = 3.88 mm
Specimen width B = 10 mm

Normalized Stress Intensity Factor – FEM modeled solutions

$$K_I = \frac{F_I(a)P\,(S_o - S_i)}{B\,W^{3/2}}$$

$$K_{II} = \frac{F_{II}(a)P\,(S_o - S_i)}{B\,W^{3/2}}$$

$$\varphi = atan\left(\frac{K_{II}}{K_I}\right)$$

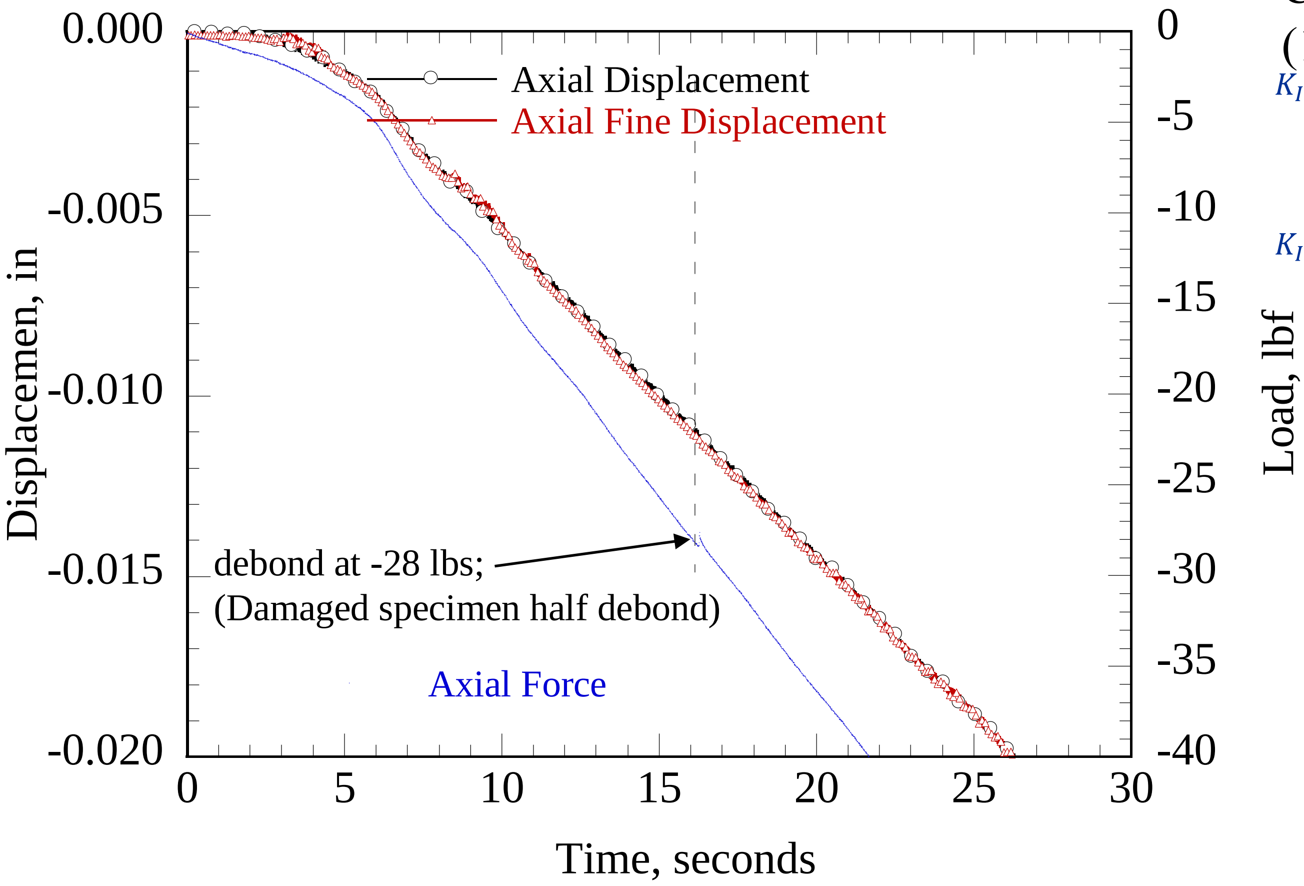
Mode Mixty 29.10 deg

The FEM Table numbers used (close to steady-state)

a/2 mm	a/Si	KI-norm	KII-Norm	Angle, deg	G-Norm
2	0.1	1.07	0.59	29.10	4.89E-05

EBC Delamination and CMC Interlaminar Toughness Testing and Modeling under Heat Flux Conditions - Continued

- Si bond coated specimen, failure load at -28 lbf, Room Temperature



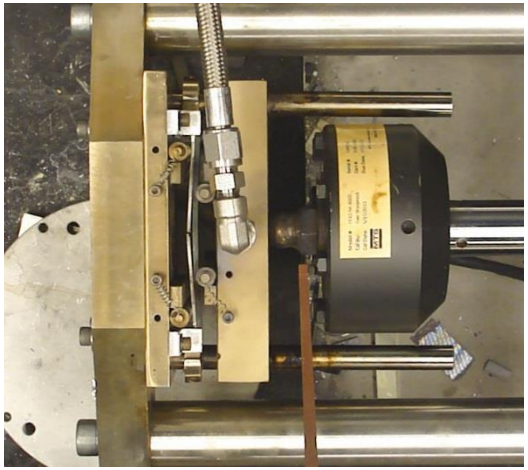
Critical load At -28 lbf (124.578N)

$$K_I = \frac{F_I(a)P\,(S_o - S_i)}{B\,W^{3/2}} = \frac{1.07 * 124.578 * 40 * 10^{-3}}{(10 * 10^{-3}) * (3.88 * 10\hat{}-3)^{3/2}}$$

$$K_I = 2.206\,MPa * m^{0.5}$$

$$K_{II} = \frac{F_{II}(a)P\,(S_o - S_i)}{B\,W^{3/2}} = \frac{0.59 * 124.578 * 40 * 10^{-3}}{(10 * 10^{-3}) * (3.88 * 10\hat{}-3)^{3/2}}$$

$$K_I = 1.216\,MPa * m^{0.5}$$

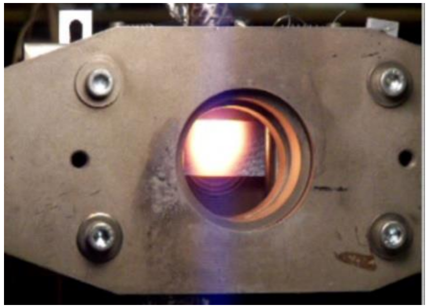


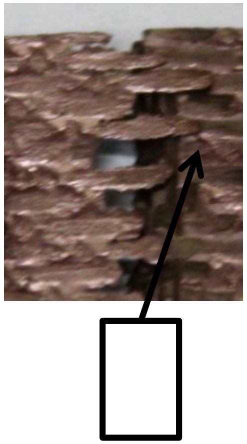
Debond

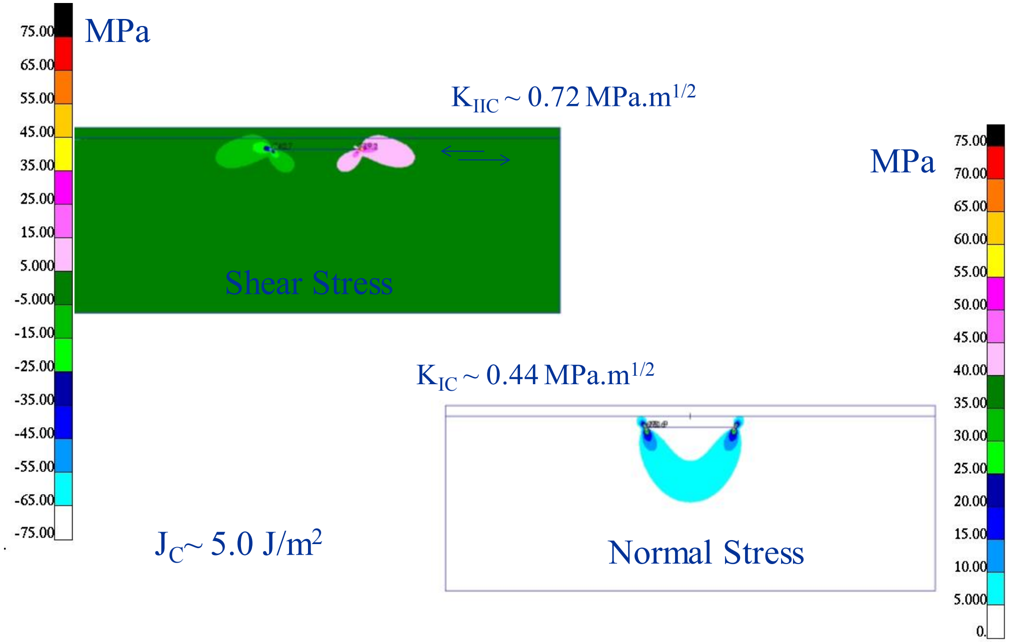
After test

EBC Delamination and CMC Interlaminar Toughness Testing and Modeling under Heat Flux Conditions - Continued

- The CMC-coating interlaminar fracture energy tested from a uncoated MI SiC/SiC CMC specimen under combined thermal heat flux and mechanical flexural loading

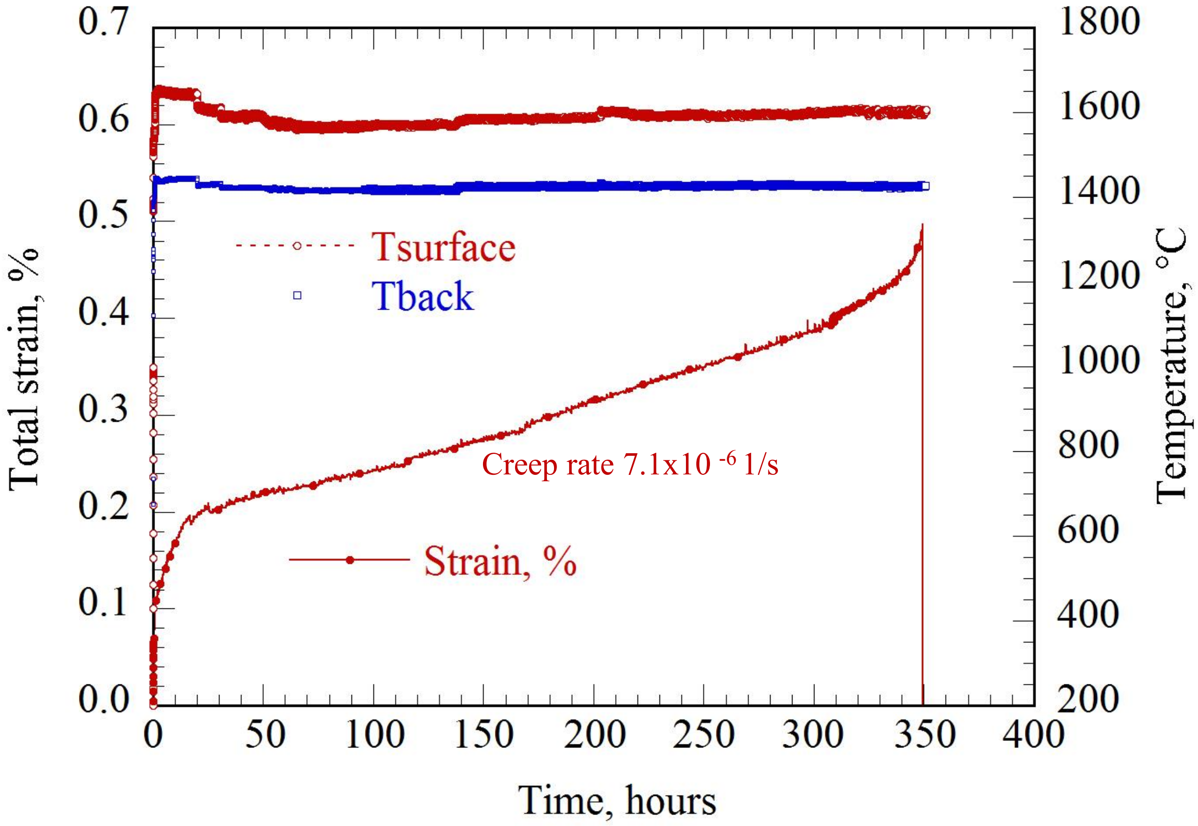






EBC Coated CMC 2650°F (1454°C) Creep Rupture Durability Test

- SiC/SiC CMC 12C-470-022 SiC/SiC CVI-MI CMC specimen
- Coated with 2700°F (1482°C) RESi and Rare Earth EBC
- Test temperatures: $T_{EBC\ surface}$ at 2850-3000°F (1600-1650°C), and $T_{cmc\ back}$ at ~2600°F (1426°C)



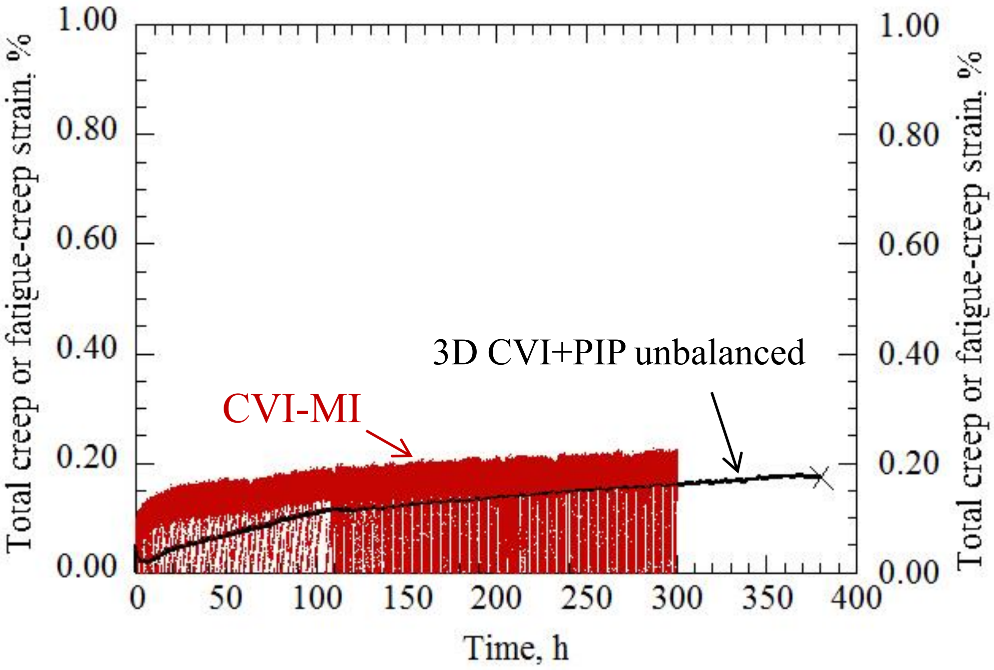
Advanced EBC Coated CMCs Demonstrated Creep and Fatigue Durability at ~2700°F

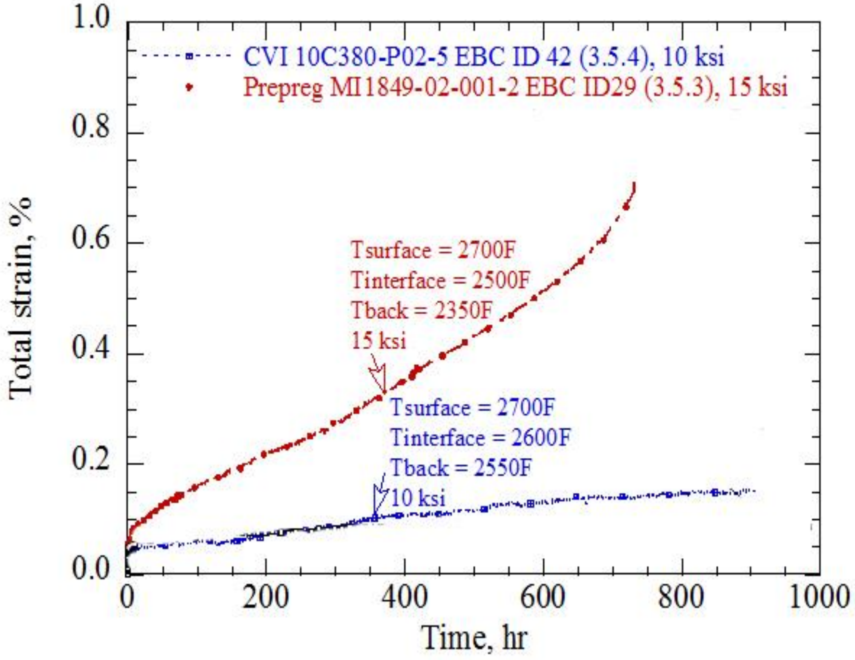
- Turbine airfoil EBC systems with advanced HfO_2-rare earth silicate and GdYbSi or NdYbSi bond coats tested with CVI-MI and CVI-PIP CMCs in laser heat flux rigs
- Demonstrated initial durability at 2700°F

- CVI-MI, Fatigue loading 69 MPa 10 ksi (69 MPa), R=0.05, with 1 h
- Thermal LCF
- $T_{EBC\text{-}surface}$ ~1537°C (~2800°F)
- $T_{bond\ coat}$ 1480°C (~2700°F)
- $T_{back\ CMC\ surface}$ ~1250°C (2282°F)

- 3D CVI+PIP unbalanced, Creep loading 50MPa (7.5ksi)
- $T_{EBC\text{-}surface}$ 1537°C (2800F)
- $T_{bond\ coat}$ 1480°C (~2700F)
- $T_{back\ CMC\ surface}$ 1271°C (2500F)

- In comparison with previously lower temperature creep tested EBC coated prepreg MI and CVI CMCs

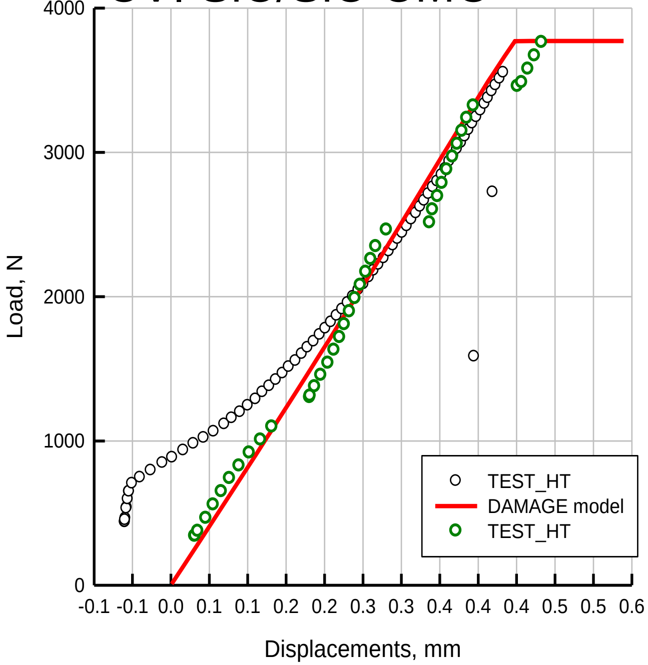




SiC/SiC Turbine Airfoil Trailing Edge Tests

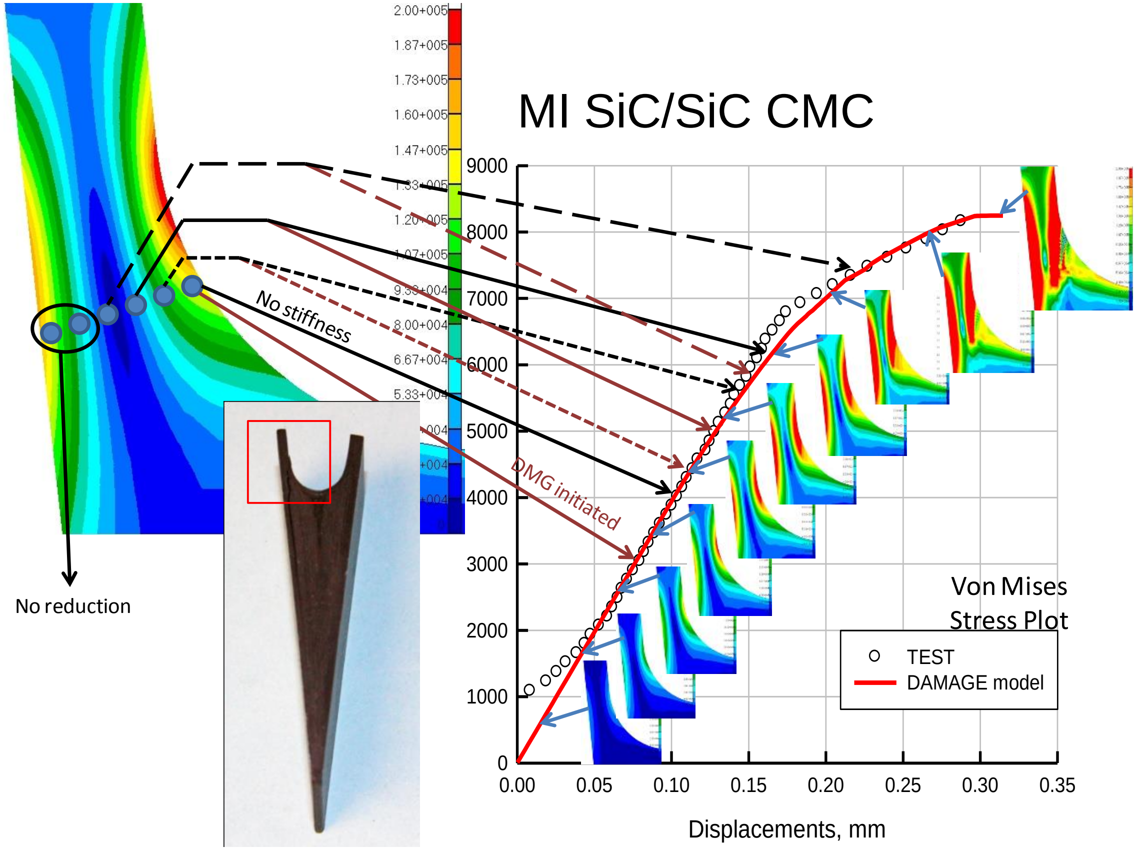
- Subelement wedge testing and high temperature tests, aiming at understanding the CMC and EBC degradation

CVI SiC/SiC CMC



Subelement Load-Displacement curve – CVI CMC trailing edge

MI SiC/SiC CMC



Subelement Load-Displacement curve – Prepreg MI CMC trailing edge

Summary and Future Directions

- **Advanced high heat flux creep rupture, fatigue and biaxial ball-on-ring rigs established for simulated EBC-CMC testing**
  - High temperature comprehensive testing capability
  - Real time coating degradation monitoring
  - Incorporated thermography, electrical resistance, acoustic emission for in-situ NDE
  - FEM models helped understand the testing

- **Long term creep rupture and fatigue behavior evaluated for EBCs-CMCs at 1482°C (2700°F)**

- **The heat flux thermomechanical testing capabilities crucial for the EBC-CMC materials development and life modeling**

Future plans

- HCF high heat flux rig with additional environmental testing capabilities (mixture controlled steam or vacuum capabilities)
- Additional full field strain measurement experiments, in particular at high temperatures
- Planned a multi-axial testing rig for CMC and EBC testing

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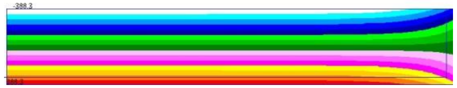
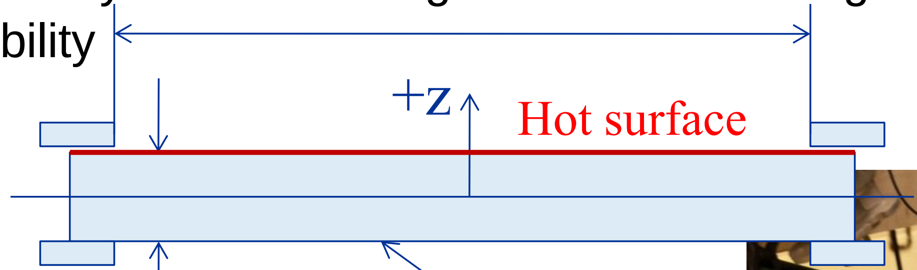
Laser Heat Flux Thermal Gradient Associated Stresses

- Through thickness gradient stresses in a disk test specimen
- Constrained subelements tests may have advantages in understanding complex stress effects on SiC/SiC durability

$$\sigma_r = \frac{\alpha\,\Delta T\,E}{2(1-\nu)} \cdot \left(\frac{-z}{t/2} \right)$$





+z

Hot surface

Cooled surface

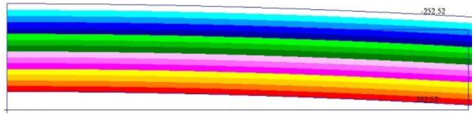
Specimen’s thickness, t

Independent of conductivity, thickness, and diameter!

Where α= Coefficient of Thermal Expansion, E= Modulus, ν = Poisson’s Ratio,
t = specimen thickness, z = axial distance from the mid section, a = radial distance to the edge support

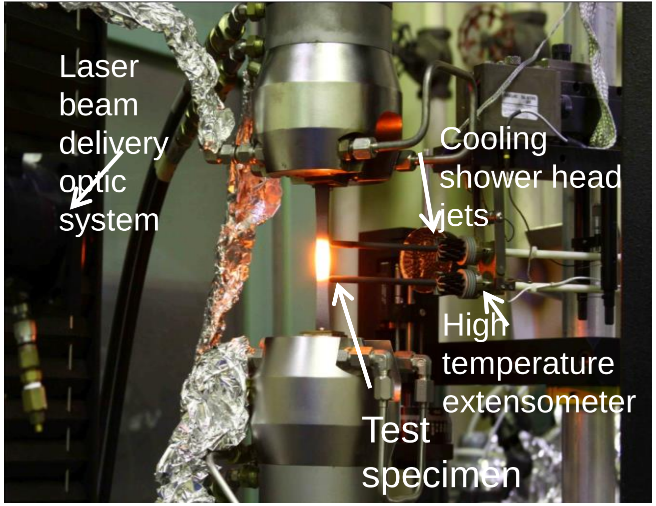
Equation when disk is free to bend

$$\sigma_r = \frac{\alpha\,\Delta T\,E}{2(1-\nu)} \cdot \left(\frac{-z}{t/2} \right) \cdot \left(1.0798 - 0.9935\left(\frac{t}{a} \right) - 5.4667\left(\frac{t}{a} \right)^2 \right)$$



Debonding and Interlaminar Studies at High Temperatures under Thermal Gradients – with Creep 10 ksi Loading

- Early EBC-SiC/SiC specimen thermal gradient creep test, completed 600 hr creep rupture testing at 69 MPa (10 ksi)
- Convoluted EBC and CMC degradation in creep testing EBC-CMC specimens, conductivity reductions



Laser high heat flux tensile rupture testing