



# **Calcium-Magnesium-Aluminosilicate (CMAS) Infiltration and Cyclic Degradations of Thermal and Environmental Barrier Coatings in Thermal Gradients**

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## Abstract

In a continuing effort to develop higher temperature capable turbine thermal barrier and environmental barrier coating systems, Calcium-Magnesium-Aluminosilicate (CMAS) resistance of the advanced coating systems needs to be evaluated and improved. This paper highlights some of NASA past high heat flux testing approaches for turbine coatings assessments in the CMAS environments. One of our emphases has been focused on the thermal barrier - environmental barrier coating composition and testing developments. The effort has included the CMAS infiltrations in high temperature and high heat flux turbine engine like conditions using advanced laser high heat flux rigs, and subsequently degradation studies in laser heat flux thermal gradient cyclic and isothermal furnace cyclic testing conditions. These heat flux CMAS infiltration and related coating durability testing are essential where appropriate CMAS melting, infiltration and coating-substrate temperature exposure temperature controls can be achieved, thus helping quantify the CMAS-coating interaction and degradation mechanisms. The CMAS work is also playing a critical role in advanced coating developments, by developing laboratory coating durability assessment methodologies in simulated turbine engine conditions and helping establish CMAS test standards in laboratory environments.

### Acknowledgements

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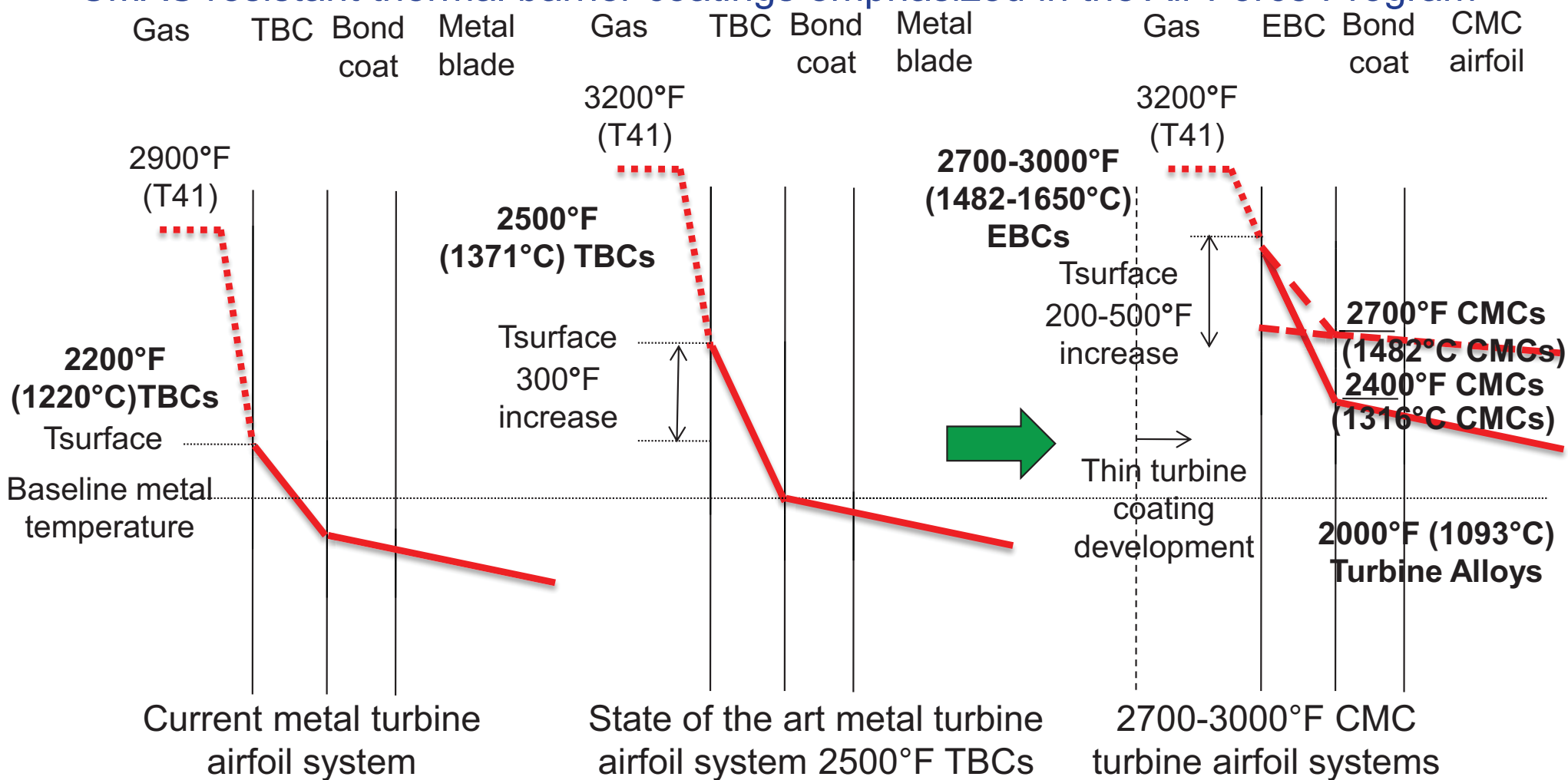
# Objectives

- **Develop advanced rig testing capability in understanding Calcium-Magnesium-Aluminosilicate (CMAS) Infiltration and Cyclic Degradations**
  - Including test rig test validation and comparisons
  - Helping establish robust hot-section component CMAS test standards and methodologies
- **Establish preliminary CMAS and coating property data information**
- **Evaluate turbine engine thermal and environmental barrier coatings in heat flux rig, burner rig, and cyclic testing**
  - Helping understand CMAS, erosion, and oxidation failures, and their interactions
- **Support NASA and Air Force CMAS composition development and CMAS resistant coating development**



# 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
- CMAS resistant thermal barrier coatings emphasized in the Air Force Program



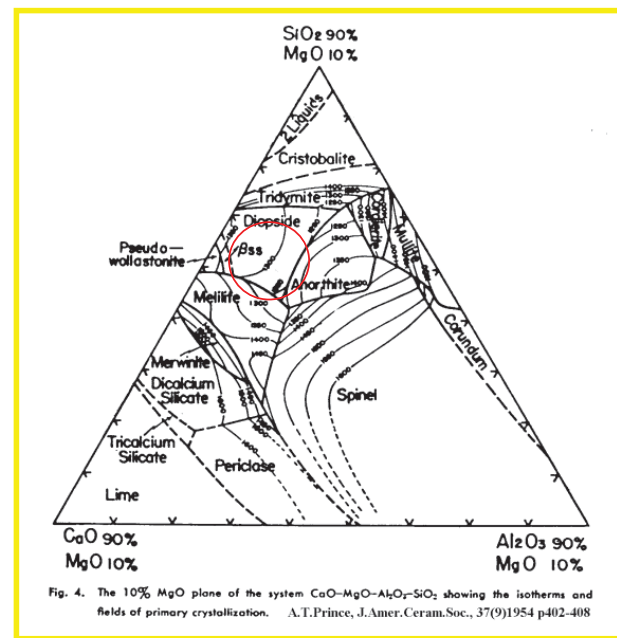
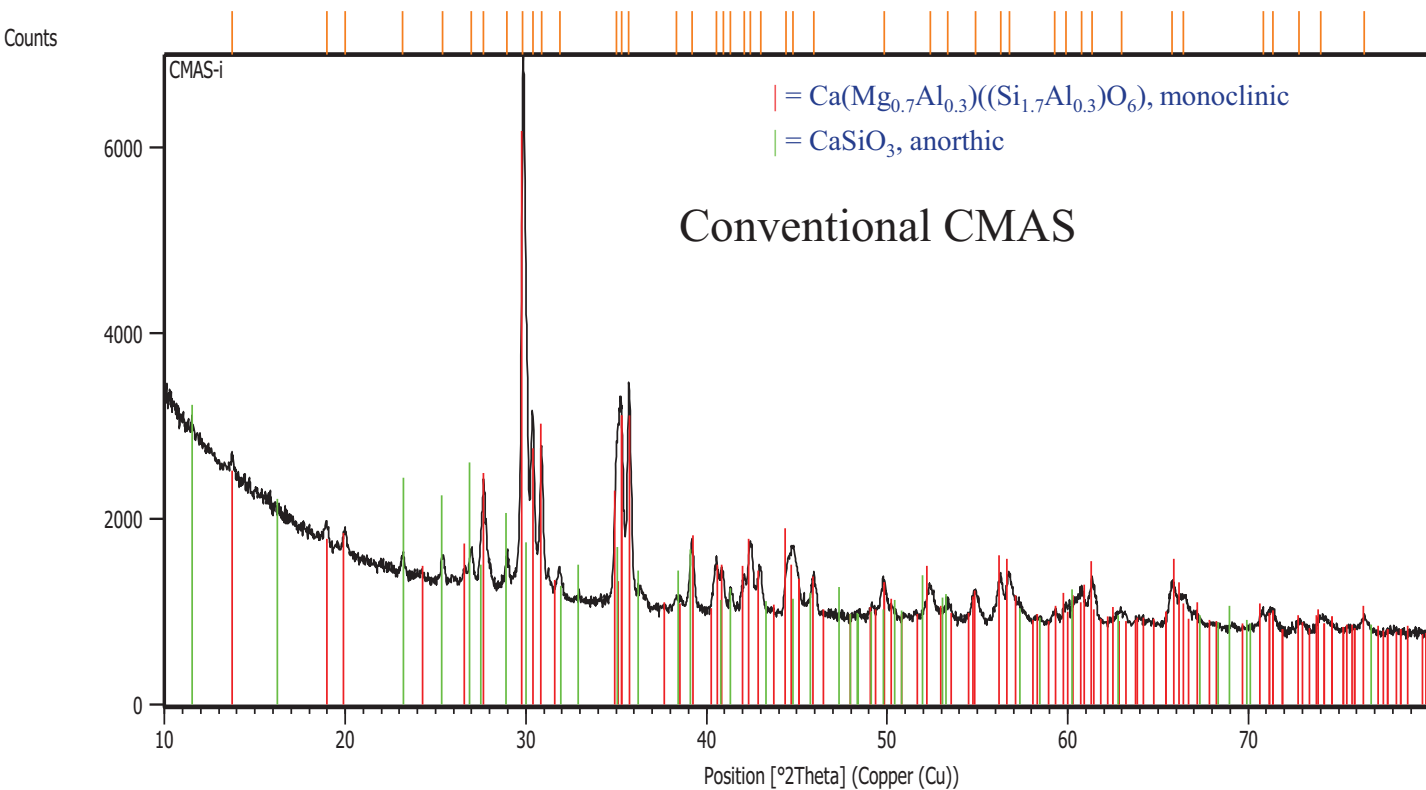


# Major CMAS Resistant Coating Research and Developments Emphasis

- **Thermal and environmental barrier coatings need improved erosion, impact and calcium-magnesium-alumino-silicate (CMAS) resistance**
  - Interactions with interface oxidation, interface reactions, and/or environment degradation, erosion, and fatigue
  - Advanced test methods and advanced coating compositions
  - Coating durability and life predictions

# Phase Studies of Calcium Magnesium Alumino-Silicate (CMAS) Materials Used for This Study

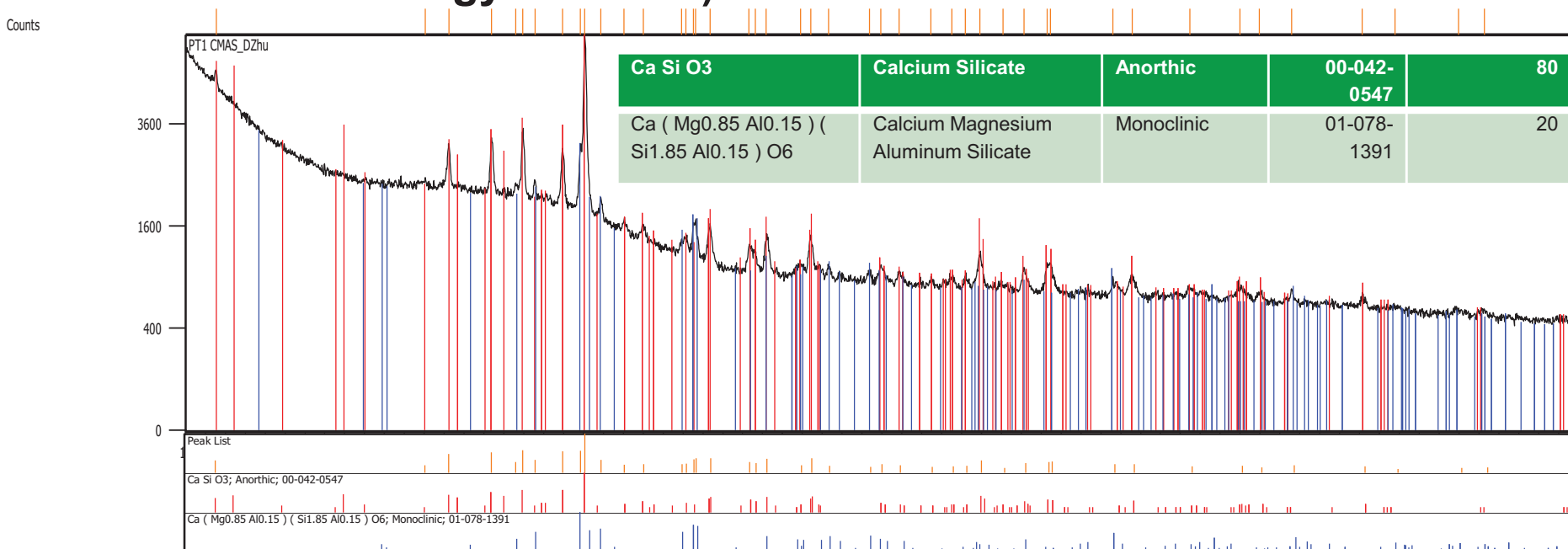
- Conventional CMAS
- Air Force developed and specially processed CMAS (processed by Powder Technology Inc. - PTI)



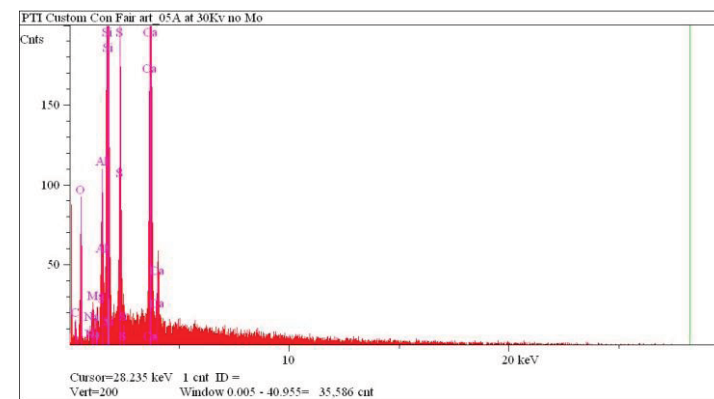
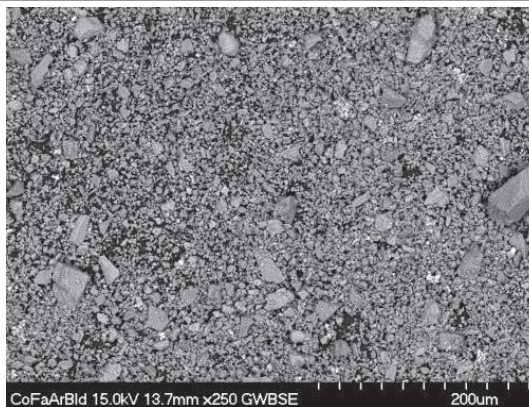
Conventional CMAS for thermal barrier coating testing

# Phase Studies of Calcium Magnesium Alumino-Silicate (CMAS) Materials Used for This Study - Continued

- Conventional CMAS
- Air Force developed and specially processed CMAS (processed by Powder Technology Inc. - PTI)



Air Force CMAS





# Thermal and Environmental Barrier Coating Systems Being Studied

## — Turbine Thermal Barrier Coatings

- EB-PVD 7YSZ/PtAl on Rene N5
- EB-PVD t'  $Gd_2Zr_2O_7$ /PtAl+Hf on Rene N5
- EB-PVD 7YSZ/PtAl+Hf on Rene N5 (Howmet)
- EB-PVD t' low k  $ZrO_2-1.6Y_2O_3-1.2Gd_2O_3-1.2Yb_2O_3$ /PtAl+Hf on Rene N5 (Howmet)
- EB-PVD t' low k  $ZrO_2-1.6Y_2O_3-1.2Gd_2O_3-1.2Yb_2O_3$ /PtAl+Hf on Rene N5
- EB-PVD an advanced  $HfO_2$ /PtAl on Rene N5 (DVTI)
- NASA *In-house PS-PVD low k t'  $ZrO_2$ /PtAl on Rene N5*
- NASA *In-house PS-PVD  $Gd_2Zr_2O_7$ /PtAl on Rene N5*

## — Environmental barrier coating systems

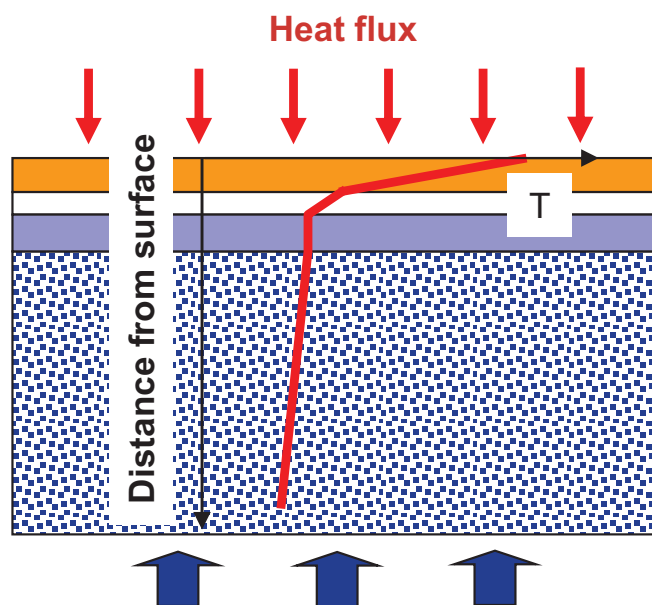
- Ytterbium mono-silicate and ytterbium di-silicate/ $HfO_2$ -Si/SiC/SiC ceramic matrix composite
- Ytterbium mono-silicate and ytterbium di-silicate/ $HfO_2$ -Si/SiC/SiC ceramic matrix composite



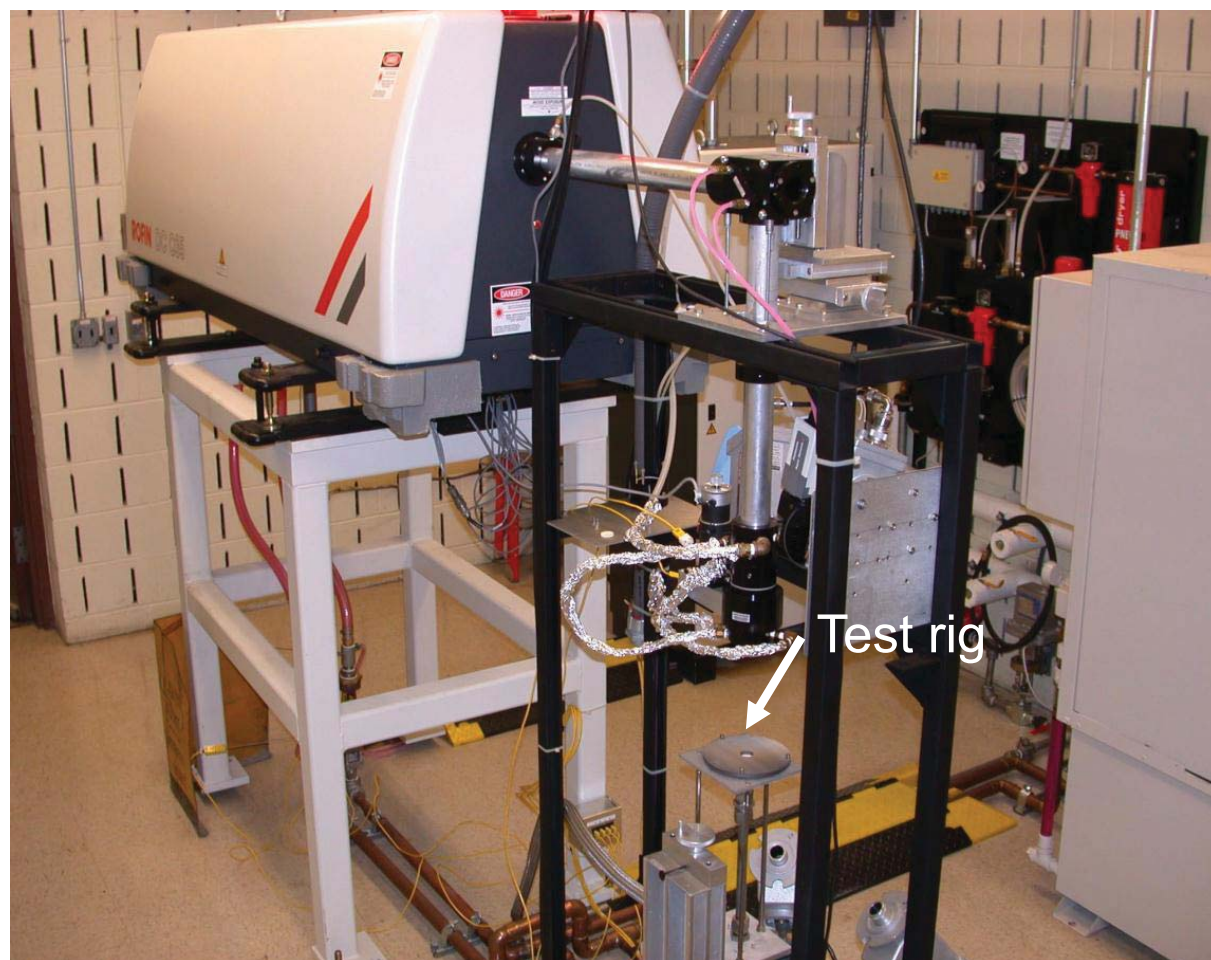
## High Heat Flux CO<sub>2</sub> Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development

- Initial work has focused on NASA high heat flux rig CMAS infiltration and durability testing

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<sup>2</sup>-K

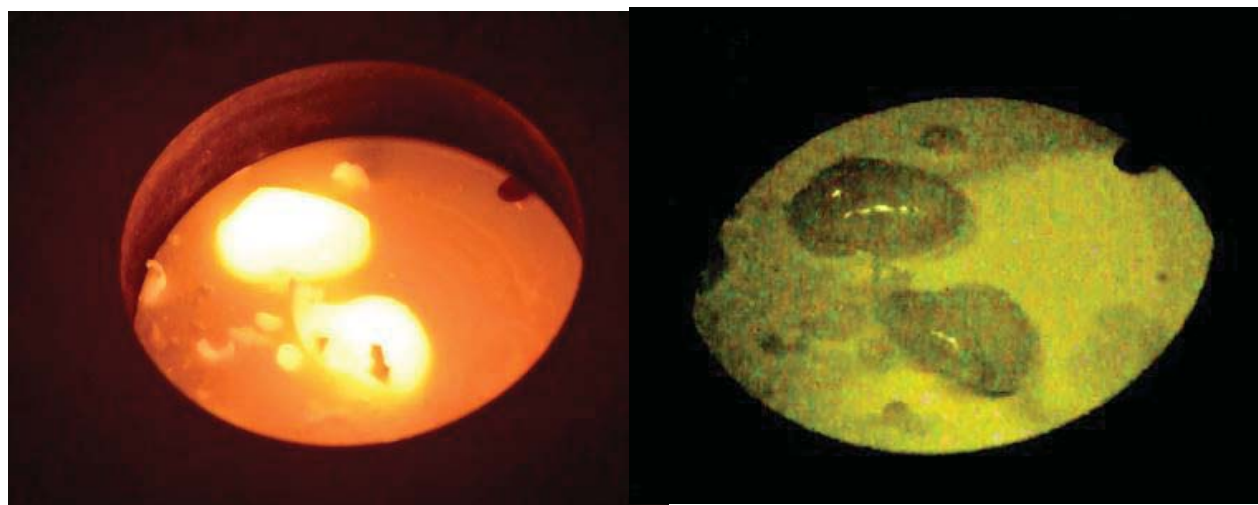


## High Heat Flux CO<sub>2</sub> Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development

- Direct Laser heat flux infiltrated thermal barrier coatings (Conventional CMAS)



- Direct Laser heat flux infiltrated thermal barrier coatings (Air Force/PTI CMAS)

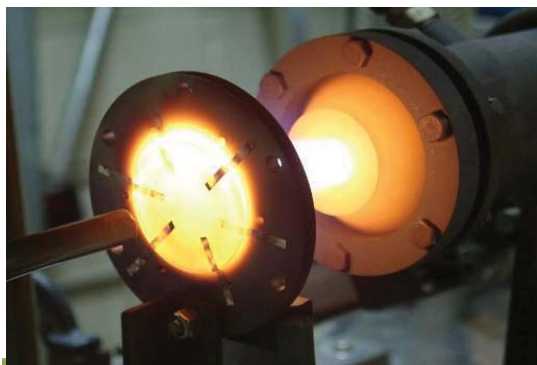


EB-PVD Low k  $\text{ZrO}_2\text{-}4\text{mol}\%\text{Y}_2\text{O}_3\text{-}3\text{mol}\%\text{Gd}_2\text{O}_3\text{-}3\text{mol}\%\text{Yb}_2\text{O}_3$   
/PtAl/Rene N5 (Howmet Processing-  
Run 3844, ID 15H1)

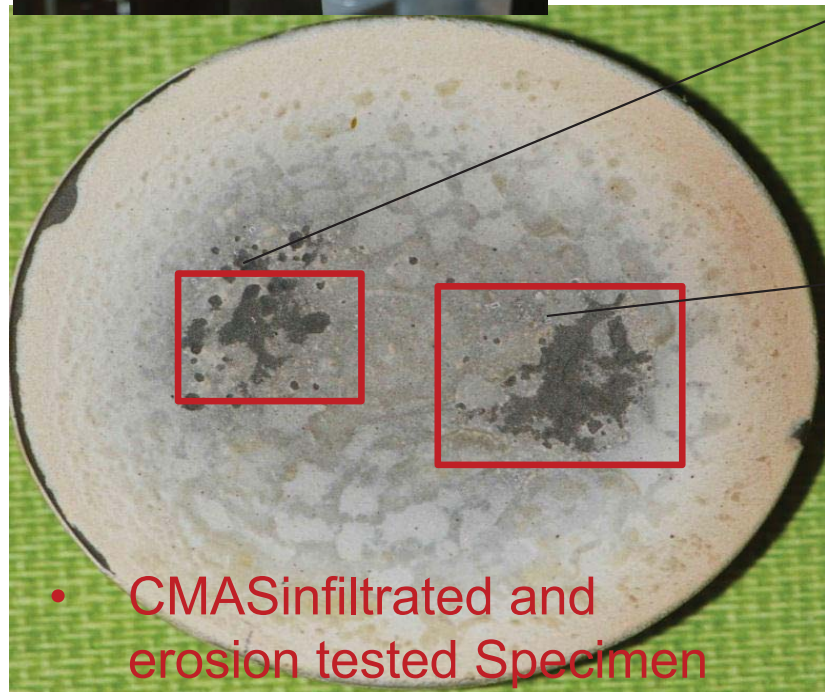


## CMAS Laser Infiltrated and Related Burner Erosion Failure

- Localized spallation and high erosion rates in high CMAS concentration areas:



- Mach 0.5 gas velocity
- Specimen testing temperature 2100°F

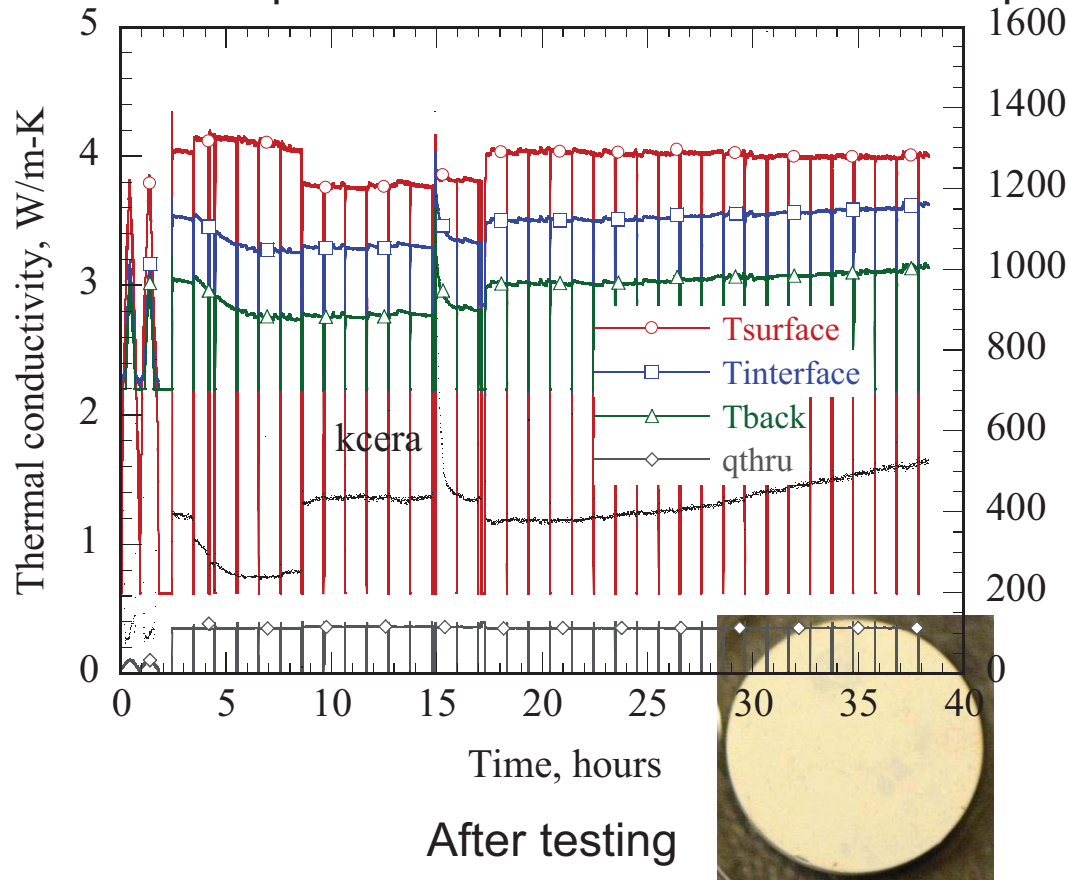


- CMAS infiltrated and erosion tested Specimen



## Laser High Heat Flux Rig For CMAS Thermal Gradient Cyclic Durability Testing

- An EB-PVD  $ZrO_2$ -2mol% $Y_2O_3$ -1.5mol% $Gd_2O_3$ -1.5mol% $Yb_2O_3$  TBC (PtAl/Rene N5 substrates) furnace tested at 1150°C 20 hr, then laser thermal gradient 2-cycle CMAS infiltration
- Continued laser thermal gradient cyclic tested at  $T_{surface}$  1280°C and  $T_{interface}$  1150°C for 38, 1 hr cycles with CMAS
- The specimen survived the test without spalling





## CMAS Laser + “Receptor” Heat Flux Infiltrated and Related Laser Rig Cyclic Failure

- Laser heat flux Receptor heat flux CMAS Infiltration
- Improved pressure infiltration and added additional dopants possible



YSZ 01 laser + receptor  
heat flux “pressure”  
infiltrated: C+Si dopants



YSZ 02 laser + receptor  
heat flux “pressure”  
infiltrated: C, Si dopants



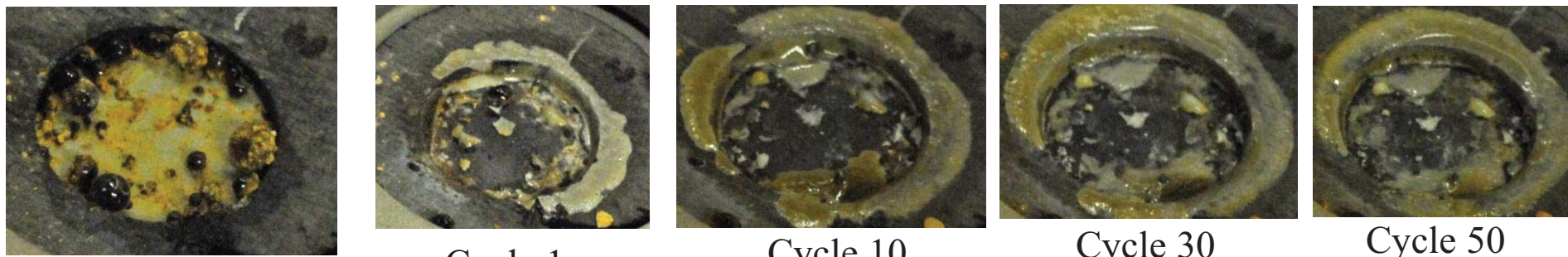
YSZ 03 laser + receptor  
heat flux “pressure”  
infiltrated: YSZ added



YSZ 02 laser rig cyclic tested  
and spalled, after 50 hr test at  
 $T_{\text{surface}} 1230^{\circ}\text{C}$  and  $T_{\text{interface}} 1170^{\circ}\text{C}$

# Laser High Heat Flux Thermal Gradient Cyclic Tested Ytterbium Silicate EBC/HfO<sub>2</sub>-Si Bond Coat on SiC/SiC CMC Substrate for 80, 1hr cycles at 1400°C

– Early partial EBC spallation; severe Yb/HfO<sub>2</sub>-Si reactions observed



Infiltration, 35 mg/cm<sup>2</sup>

Cycle 1

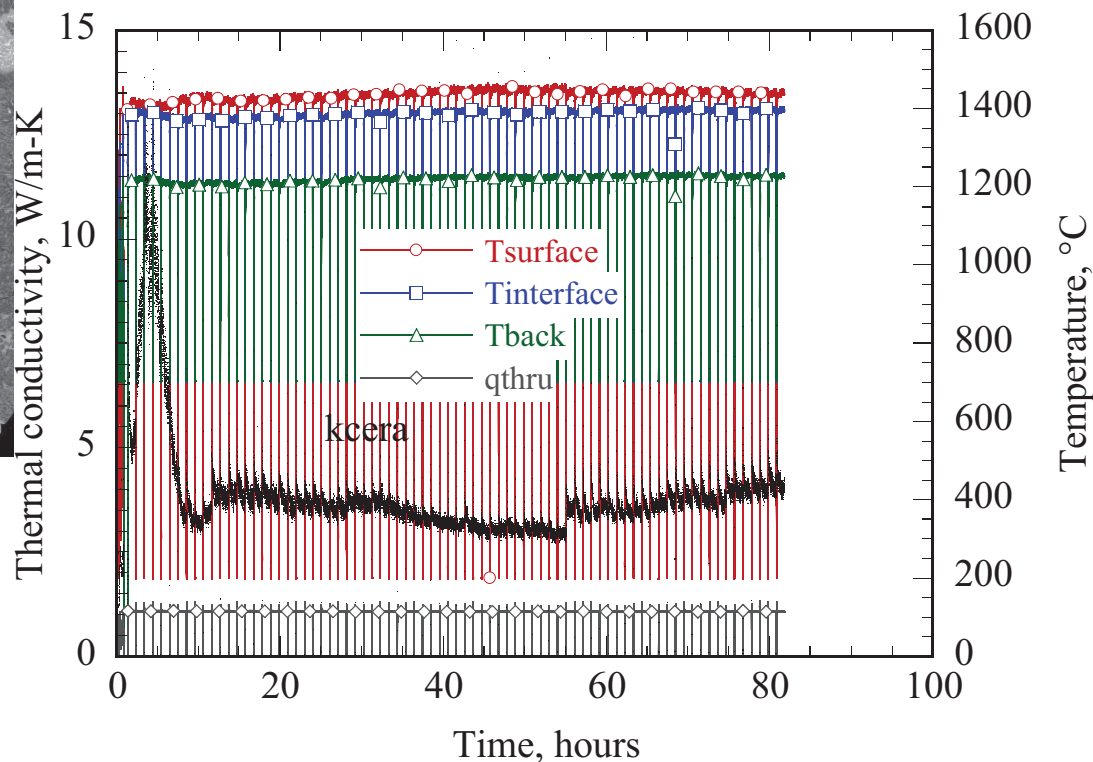
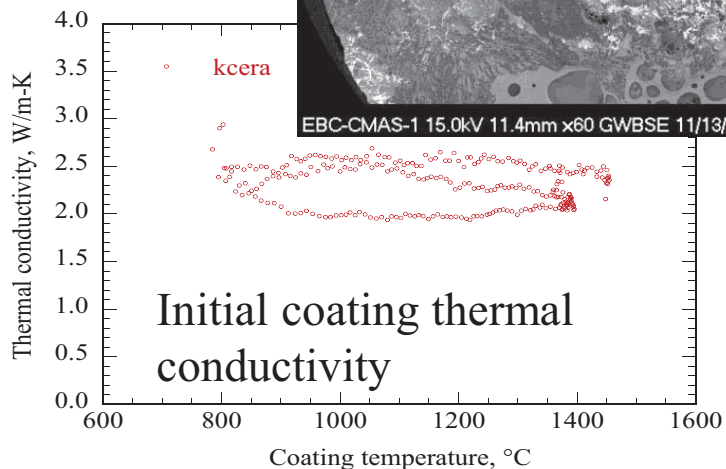
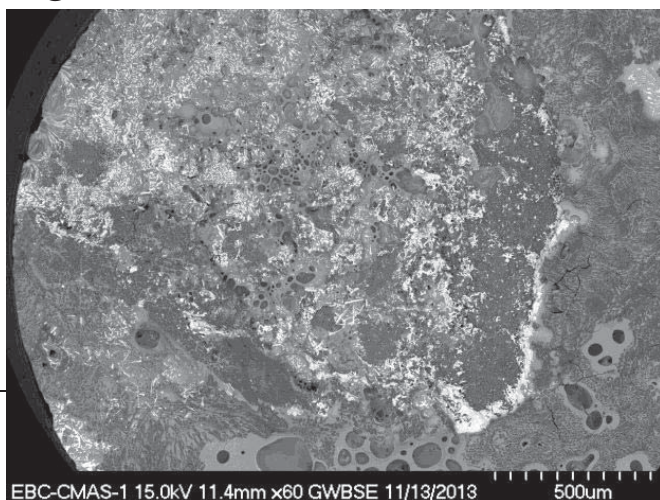
Cycle 10

Cycle 30

Cycle 50



Cycle 80

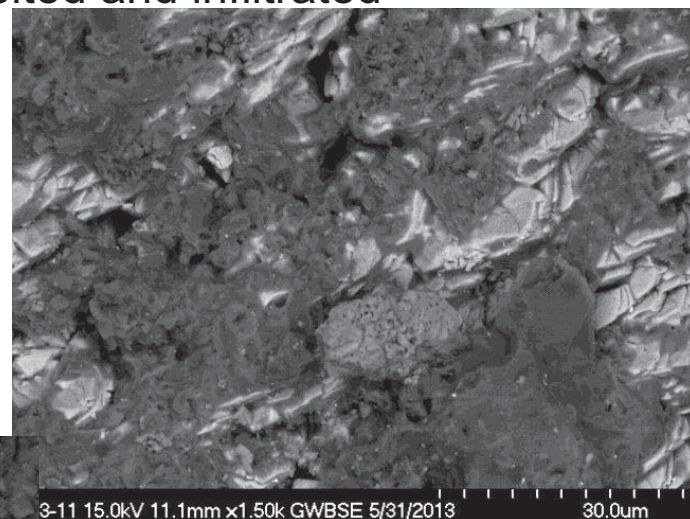




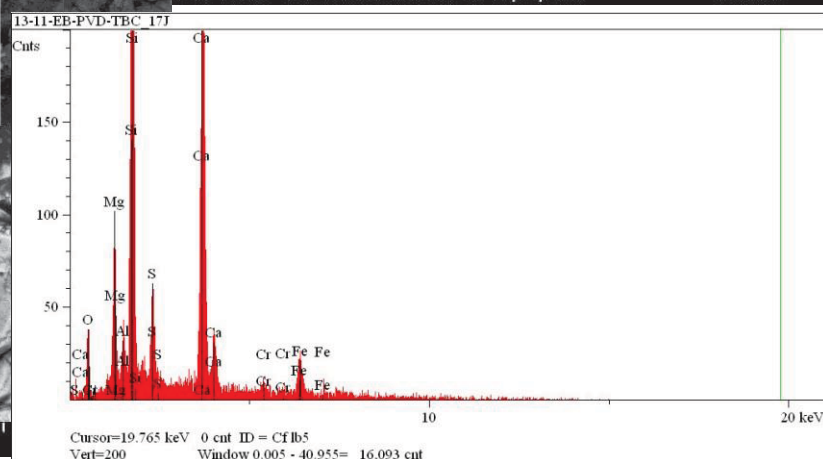
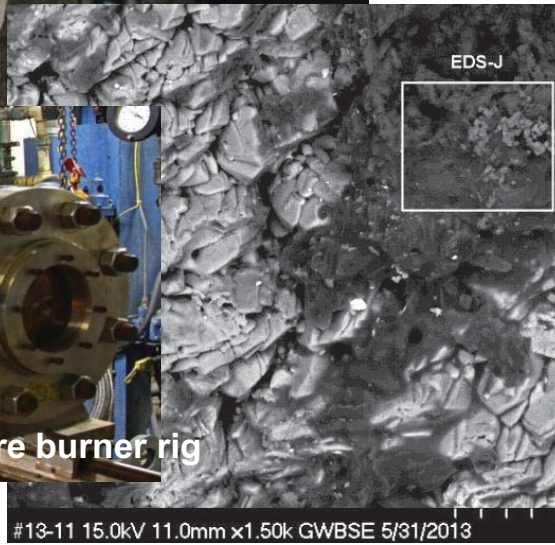
# High Velocity and High Pressure Burner Rig Tested for TBC Specimen CMAS Injection and Infiltration - Continued

- A 2" diameter thermal barrier coated disc (back cooled) used for the initial test
- Thermal barrier coating temperature at 1250°C
- Rig conditions:
  - Jet fuel & air combustion with mass air flow 1.5 lbm/s
  - 10 atmospheres (160 psi) pressure
  - CMAS injected after the burner nozzle but not fully melted and infiltrated

CMAS Infiltration test in a coated specimen



High pressure burner rig





## Summary

- Environmental barrier coating and thermal barrier coating systems, including ytterbium silicates, four-component and six-component low conductivity systems, gadolinium zirconate, were selected and initially tested for CMAS infiltration and durability
- CMAS compositions and phases identified
- CMAS infiltration methods, that is, a direct heat flux heating infiltration and a heat flux “receptor” heating infiltration, were established; and demonstrated effective using convectional and Air Force CMAS sands
- CMAS composition, deposition and aero aspects being initially investigated in high pressure burner rig environments
- CMAS infiltration and erosion degradation interaction mechanisms identified for turbine thermal barrier coatings, more detailed investigations in progress
- Furnace cyclic testing of infiltrated coating specimens planned