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

Current metal turbine airfoil system

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

2700-3000°F CMC turbine airfoil systems

Baseline metal temperature

Gas TBC Bond coat Metal blade Gas TBC Bond coat Metal blade Gas EBC Bond coat CMC airfoil

2200°F (1220°C) TBCs

2500°F (1371°C) TBCs

Tsurface 300°F increase

Tsurface 200-500°F increase

Thin turbine coating development

2700°F CMCs (1482°C CMCs)

2400°F CMCs (1316°C CMCs)

2000°F (1093°C) Turbine Alloys
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)

\[
\text{Position } [^\circ \text{2Theta}](\text{Copper (Cu)})
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\[
\text{CMAS-i} = \text{Ca(Mg}_{0.7}\text{Al}_{0.3})((\text{Si}_{1.7}\text{Al}_{0.3})\text{O}_6), \text{monoclinic}
\]

\[
\text{CMAS-i} = \text{CaSiO}_3, \text{anorhlc}
\]

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)

### Materials

- **Conventional CMAS**
- **Air Force CMAS**

#### Phase List

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**Image:**

- **Air Force CMAS**

![Image of Air Force CMAS](image-url)
Thermal and Environmental Barrier Coating Systems Being Studied

Turbine Thermal Barrier Coatings
- EB-PVD 7YSZ/PtAl on Rene N5
- EB-PVD t’ Gd$_2$Zr$_2$O$_7$/PtAl+Hf on Rene N5
- EB-PVD 7YSZ/PtAl+Hf on Rene N5 (Howmet)
- EB-PVD t’ low k ZrO$_2$-1.6Y$_2$O$_3$-1.2Gd$_2$O$_3$-1.2Yb$_2$O$_3$/PtAl+Hf on Rene N5 (Howmet)
- EB-PVD t’ low k ZrO$_2$-1.6Y$_2$O$_3$-1.2Gd$_2$O$_3$-1.2Yb$_2$O$_3$/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$_2$Zr$_2$O$_7$/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 CO₂ 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

Heat flux

Distance from surface

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

Test rig
High Heat Flux CO$_2$ Laser Rig and Testing for Thermal and Environmental Barrier Coatings Development

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

\[
\begin{align*}
\text{ZrO}_2-1.0\text{Y}_2\text{O}_3-1.5\text{Gd}_2\text{O}_3-1.5\text{Yb}_2\text{O}_3 \\
\text{ZrO}_2-1.6\text{Y}_2\text{O}_3-1.2\text{Gd}_2\text{O}_3-1.2\text{Yb}_2\text{O}_3 \\
\text{7YSZ}
\end{align*}
\]

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

\[
\begin{align*}
\text{EB-PVD Low k ZrO}_2-4\text{mol}\%\text{Y}_2\text{O}_3-3\text{mol}\%\text{Gd}_2\text{O}_3-3\text{mol}\%\text{Yb}_2\text{O}_3 \\
/\text{PtAl/Rene N5 (Howmet Processing-Run 3844, ID 15H1)}
\end{align*}
\]
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$_2$O$_3$-1.5mol%Gd$_2$O$_3$-1.5mol%Yb$_2$O$_3$ 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 $T_{\text{surface}}$ 1280°C and $T_{\text{interface}}$ 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°C and $T_{\text{interface}}$ 1170°C
Laser High Heat Flux Thermal Gradient Cyclic Tested Ytterbium Silicate EBC/HfO₂-Si Bond Coat on SiC/SiC CMC Substrate for 80, 1hr cycles at 1400°C

- Early partial EBC spallation; severe Yb/HfO₂-Si reactions observed

Infiltration, 35 mg/cm²

Cycle 1
Cycle 10
Cycle 30
Cycle 50

Cycle 80

Initial coating thermal conductivity

Thermal conductivity, W/m-K

Coating temperature, °C

Temperature, °C

Time, hours

Initial coating thermal conductivity chart

Graph showing thermal conductivity and coating temperature over time.
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
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