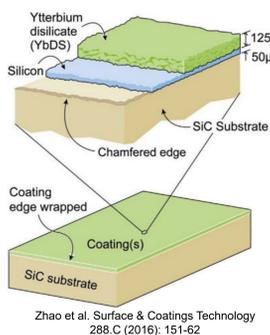


Reactions between molten calcium-magnesium-aluminosilicates (CMAS) and ytterbium disilicate (YbDS) environmental barrier coatings (EBCs) are investigated. EBCs are used to protect ceramic matrix composites (CMCs) from volatilization at high temperatures. An oxy-acetylene torch is used to ablate samples at 1400 °C. Ablation introduces a thermal gradient on samples, simulating use in gas turbine blades. The effect of cyclic exposure on CMAS-coating interactions and the effect of CMAS reaction as a function of time are investigated. X-ray diffraction (XRD) was used to examine the products of these reactions. XRD results indicated longer ablation test times as well as cyclic ablation exposures increase reaction phase formation. Thermal modeling is also used to determine the magnitude of the thermal gradient across samples and to predict thermal gradients of different materials.

This work is sponsored by Rolls-Royce Corp., Indianapolis, IN

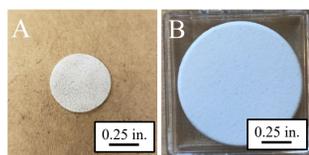
## Project Background

To increase efficiency of gas turbines, Rolls-Royce aims to increase operating temperatures by replacing nickel-based superalloy turbine blades with reaction-bonded silicon carbide (RBSiC) CMCs. CMCs require an EBC to protect turbine blades from volatilization by water vapor degradation in high temperature combustion environments. YbDS ( $\text{Yb}_2\text{Si}_2\text{O}_7$ ) has been identified as a potential EBC due to its similar coefficient of thermal expansion (CTE) of RBSiC. CMAS sands pose a threat to EBC life. A reaction of molten CMAS and YbDS creates a new phase (ytterbium silicate oxyapatite, or apatite) that could cause failure of the EBC.<sup>1</sup> An oxy-acetylene torch is used to ablate samples at 1400 °C. Heat cycling is used to simulate the take-off and landing of a gas turbine. The evolution of EBC-CMAS reactions as a function of time is also of interest. Thermal modeling is also used to analyze heat transfer and predict thermal gradients in different EBCs.



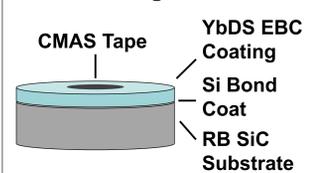
Zhao et al. Surface & Coatings Technology 288.C (2016): 151-62

## Experimental Procedure



CMAS tape (A) with concentration 4 mg/cm<sup>2</sup> was thermally bonded to YbDS-coated RBSiC samples with a Si bond coat (B). Four proprietary EBC coating types were tested (see table below).

### Sample Assembly Diagram

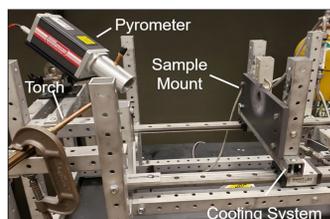


Coating	Spray parameter	Thermal process history
A	Recipe 1	Process 1
B	Recipe 1	Process 2
C	Recipe 2	Process 1
D	Recipe 2	Process 2

### Ablation Rig:

To simulate gas turbine operating conditions, an oxyacetylene torch mounted to a steel structure was used to ablate samples held in a graphite mount.

Two pyrometers measure temperatures of the front and back of samples. This structure is known as an ablation rig. The ablation rig was used to ablate samples at 1400 °C ± 25 °C.



Ablation Rig

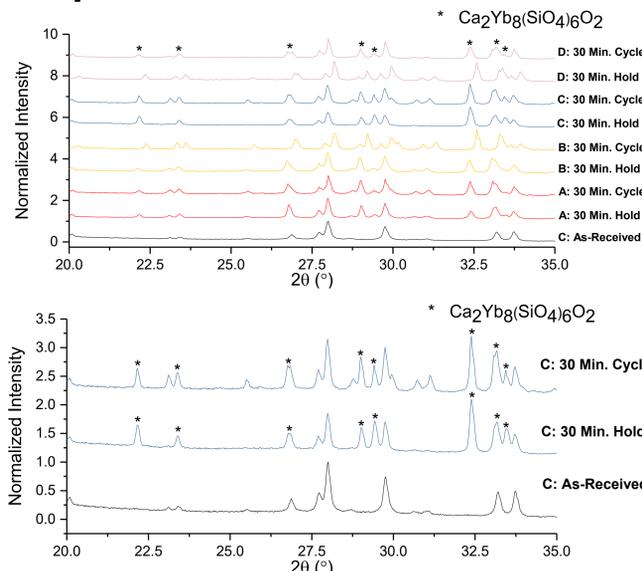
### Cyclic Study:

- Performed 5 min hold, cooled sample to 500 °C for 1 min
- Repeated 6 times
- Performed 30 min hold w/o cycling
- Tested all coatings

### Time Dependent Study:

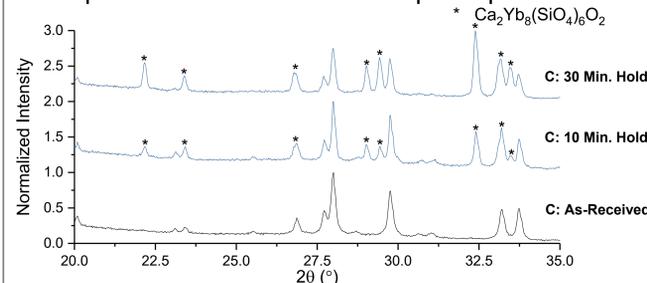
- Tested CMAS reactivity over time
- Performed 10 min and 30 min ablation tests
- Tests performed using coating C

## Apatite Phase Formation



### Cyclic Exposure Study:

- Top graph show all different  $\text{Yb}_2\text{Si}_2\text{O}_7$  coating/CMAS tape samples heated to 1400 ± 25°C and either held or cycled for 30 minutes
- Coating C chose as representative spectra on second graph
- Locations marked \* indicate locations of Silicate Oxyapatite ( $\text{Ca}_2\text{Yb}_8(\text{SiO}_4)_6\text{O}_2$ ) phase peaks.<sup>2</sup>
- Intensity of apatite phase peaks indicate a more complete transformation of the apatite phase



### Time Dependent Study:

- Graph above show spectra for  $\text{Yb}_2\text{Si}_2\text{O}_7$  coating C/CMAS tape heated to 1400 ± 25°C and held for either 10 or 30 continuous minutes
- Locations marked \* indicate locations of Silicate Oxyapatite ( $\text{Ca}_2\text{Yb}_8(\text{SiO}_4)_6\text{O}_2$ ) phase peaks.<sup>2</sup>
- Intensity of apatite phase peaks increase with an increase in continuous amount of time held, indicating a more complete transformation of apatite into the  $\text{Yb}_2\text{Si}_2\text{O}_7$  coating

## References

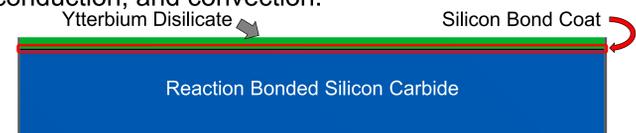
- Zhao et al. "Molten Silicate Reactions with Plasma Sprayed Ytterbium Silicate Coatings." Surface & Coatings Technology 288.C (2016): 151-62
- F. Stolzenburg, et al, "The Interaction of Calcium-Magnesium-Aluminosilicate with Ytterbium Silicate Environmental Barrier Materials." Surface & Coatings. 284 (2015) 44-50
- B.T. Richards, et al. "Response of Ytterbium Disilicate-Silicon Environmental Barrier Coatings to Thermal Cycling in Water Vapor." Acta Materialia, vol. 106, 2016, pp. 1-14.

## Acknowledgments

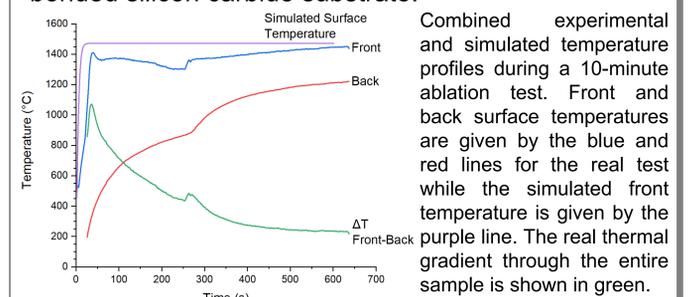
Special thanks to some of the members of the Trice group: Andrew Schlup, Dr. Jorge Ramirez-Velasco, and Averyonna Kimery.

## Thermal Modeling

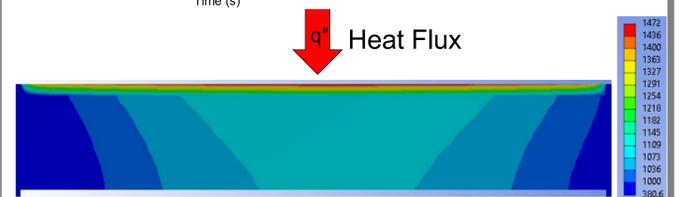
Two-dimensional thermal modeling was used to estimate the internal thermal gradients within the samples during an ablation test. ANSYS Mechanical Finite Element Analysis Software was used to create the model. The model accounts for radiation, conduction, and convection.



Modeled geometry of a tri-layered ablation sample. The green top layer represents the YbDS EBC coating. The black middle layer highlighted by a red rectangle represents the internal silicon bond coat layer. The blue bottom layer represents the reaction bonded silicon carbide substrate.



Combined experimental and simulated temperature profiles during a 10-minute ablation test. Front and back surface temperatures are given by the blue and red lines for the real test while the simulated front temperature is given by the purple line. The real thermal gradient through the entire sample is shown in green.



Simulation internal temperature plot showing thermal gradient in the EBC surface layer and total gradient through the sample during an ablation test with an applied heat flux density of 1.4  $\frac{\text{W}}{\text{mm}^2}$ . A  $\Delta T$  of 372°C can be seen across the EBC layer.

The goal of this model is to allow us to correlate the CMAS infiltration depth with the respective thermal gradient through the sample.

## Recommendations/Conclusions & Future Work

### Recommendations/Conclusions

- Longer ablation times lead to an increase of apatite formation
- Exposure of sample to 6, 5 minute cycles, leads to more apatite formation than 1, 30 minute, hold
- An increase in apatite formation could lead to failure of the EBC due to cracking, spallation, etc.<sup>3</sup>
- Sources of error for 2-D thermal model
  - Applied heat flux, 2-D constraints, simulation limitations, and thermal property data inputs

### Future Work

- Additional testing
  - Longer cycle times, longer continuous testing
  - Comparative isothermal tests
- SEM analysis of ablated samples
  - Determine CMAS infiltration depth
- 3D thermal modeling
  - Determine internal temperature gradients for comparison with CMAS infiltration