## **Development of Refractory Silicate-YSZ Dual Layer TBCs**

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### **Research Area**

• Materials (TBC)

# Objective

• Improve TBC durability by incorporating an environmental barrier with low oxygen conductivity and high hot corrosion resistance.

## **Relevance to ATS**

TBC's with enhanced environmental resistance provide longer lifetime and higher temperature capability, which in turn increase the efficiency and performance of gas turbine engines.

# Approach

- 1. Selection of Ceramic Environmental Barrier
  - Mullite, Glass Ceramics, Rare-Earth Silicates
- 2. Application of Coatings
  - APS, Sputtering, SPPS
- 3. Testing and Data Analysis
  - Thermal Cycling, Na<sub>2</sub>SO<sub>4</sub> Hot Corrosion
  - XRD, SEM.

# **Selection Of Barrier Coatings**

- Low Oxygen Conductivity
- Hot Corrosion Resistance
- Chemical Compatibility
- Thermal Expansion Coeff.

Barrier	Mullite BAS, CAS	CeAS	La <sub>2</sub> SiO <sub>5</sub> , SmAS, SLB
CTE (10 <sup>-6/o</sup> C)	3 ~ 5	7~8	9 ~ 12

CTE (YSZ) : **9~10** 10<sup>-6</sup>/°C

# **Application Of Coatings**

#### **Barrier**

- Sputtering, SPPS (oxygen barrier)
  - on top of bond coat
  - thin coating  $(1 \sim 10 \ \mu m)$
- APS (hot corrosion barrier)
  - on top of YSZ
  - thick coating  $(50 \sim 75 \,\mu m)$

#### **Bond Coat**

• LPPS -NiCrAlY (5 mil)

#### **Top Coat**

- APS
  - -YSZ (10 mil)

#### **Substrate**

- CMSX4+Y
  - 1" Dia., 1/8" thick
  - all coatings on one face

### **Testing & Data Analysis**

- Annealing: 4h in Ar-5% H<sub>2</sub>, 1100°C
- Cyclic Oxidation: 1h & 20h cycle @ 1080-1100°C
- Hot Corrosion : 900°C,  $O_2/100$ ppm SO<sub>2</sub>,

 $3 \text{ mg/cm}^2 \text{ Na}_2 \text{SO}_4 \text{ film}$ 

• Post-Test Characterization: XRD, SEM

## Results

- Oxygen Barrier Coatings by Sputtering
  - Applied at the bond coat/YSZ interface
  - Low CTE: Mullite, BAS, CeAS (L)
  - High CTE: CeAS (H), SmAS, Sm<sub>2</sub>SiO<sub>5</sub>



- •1 **m** oxygen barrier shows the best durability
  - presumably due to enhanced compliance
- No clear evidence of reduced TGO thickness (Except for mullite)
  - similar TBC life to standard YSZ TBC
  - low coating quality is an issue (porosity, discontinuity)



#### • Similar TBC life to standard YSZ TBC at 1080°C

- despite reduced TGO thickness by a factor or twopresumably due to high modulus

### • Significantly reduced life at 1000°C • not understood at this point (duplicate test is underway)

![](_page_10_Picture_0.jpeg)

#### As-sputtered 3mmCeAS(H)

![](_page_10_Picture_2.jpeg)

**3mm** CeAS(H)/YSZ after 18 cycles of 20h cycle test at 1100°C

![](_page_11_Figure_0.jpeg)

![](_page_11_Figure_1.jpeg)

**3mm** SmAS/YSZ after 16 cycles of 20h cycle test at 1100°C

### Comparison of TGO thickness (20h cycle, 1100°C)

![](_page_12_Picture_1.jpeg)

**Standard YSZ after 17 cycles** 

3mm CeAS/YSZ after 18 cycles

![](_page_12_Figure_4.jpeg)

3mm SmAS/YSZ after 16 cycles 3mm Sm<sub>2</sub>SiO<sub>5</sub>/YSZ after 14 cycles

# **Conclusions** (Oxygen Barrier)

- Thin oxygen barrier (1 $\mu$ m) performs better than thick oxygen barriers (> 3  $\mu$ m)
  - presumably due to enhanced compliance of thinner coatings
- No clear evidence that oxygen barrier reduces TGO thickness (Except for mullite oxygen barrier)
  - similar TBC life to standard YSZ TBC
  - key issues (limitations)
    - low quality coating (porosity, discontinuity, phase instability)
    - high modulus

## Results

#### •Hot Corrosion Barrier Coatings by APS

-Applied on top of YSZ

$-CaOSiO_2$	- La <sub>2</sub> SiO <sub>5</sub>
-Mullite	- SLB
-BAS	CeAS
– SmAS	- ZTY

### 48 Na<sub>2</sub>SO<sub>4</sub> Hot Corrosion at 900°C

![](_page_15_Figure_1.jpeg)

#### • CaO·SiO<sub>2</sub>, BAS, Mullite:

- dense, continuous overlay coating
- no chemical degradation, cracking or spallation

#### 48 h Na<sub>2</sub>SO<sub>4</sub> Hot Corrosion at 900°C

![](_page_16_Figure_1.jpeg)

![](_page_16_Figure_2.jpeg)

- **SLB**: high porosity
- La<sub>2</sub>SiO<sub>2</sub> : severe cracking
- **ZTY**: caused cracking in YSZ

# **Repeated 48h** Na<sub>2</sub>SO<sub>4</sub> Hot Corrosion at 900°C (salt reapplied between each cycle)

![](_page_17_Figure_1.jpeg)

#### • CaO·SiO<sub>2</sub>, BAS, Mullite:

- significantly enhanced hot corrosion resistance compared to YSZ
- need to eliminate edge effect for accurate assessment of hot corrosion durability

# **Repeated 48h** Na<sub>2</sub>SO<sub>4</sub> Hot Corrosion at 900°C (salt reapplied between each cycle)

![](_page_18_Picture_1.jpeg)

• Standard YSZ TBC: severe degradation of both YSZ and substrate

#### • CaO·SiO<sub>2</sub>, BAS, Mullite:

- significantly enhanced hot corrosion resistance compared to YSZ
- some cracks around the edge (edge effect needs to be eliminated in future test)
- •SLB, SmAS: severe cracking of the entire coating

#### Standard YSZ TBC after 3 cycles of 48h Hot Corrosion at 900°C

![](_page_19_Figure_1.jpeg)

- Degradation of YSZ near the bond coat/YSZ interface where YSZ TBC typically fails under thermal cycling
  - YSZ grains readily come off during the polishing
  - this type of degradation disappears in the presence of overlay coatings
  - combination of salt attack & high stress?

### CaO·SiO<sub>2</sub>/YSZ TBC after 4 cycles of 48h Hot Corrosion at 900°C

![](_page_20_Figure_1.jpeg)

A continuous layer of CaSO<sub>4</sub> & some residual salt on the surface
- solid CaSO<sub>4</sub> is expected to limit further reaction

# BAS /YSZ TBC after 4 cycles of 48h Hot Corrosion at 900°C

![](_page_21_Picture_1.jpeg)

- Multiple phases in the BAS coating
- A continuous layer of  $BaSO_4$  & some residual salt on the surface
  - solid BaSO<sub>4</sub> is expected to limit further reaction
- Some Na-Al silicates detected in the coating

### Mullite/YSZ and SLB/YSZ TBCs after 3 cycles of 48h Hot Corrosion at 900°C

![](_page_22_Picture_1.jpeg)

- No evidence of chemical reaction
- cracking is an issue
- plasma sprayed mullite prone to cracking
- process optimization necessary

- Entire coating spalled
- Unstable phase?

### **Conclusions** (Hot Corrosion Barrier)

- CaO·SiO<sub>2</sub>, mullite and BAS show the best potential as a hot corrosion barrier coating
  - significantly improved hot corrosion resistance
  - high density, good adherence and crack-resistance
  - CaO·SiO<sub>2</sub> & BAS show limited chemical reaction in Na<sub>2</sub>SO<sub>4</sub>
  - mullite shows no chemical reaction in  $Na_2SO_4$
- Rare earth silicates, SLB and ZTY show cracking
  - cracking in rare earth silicates is due to phase instability
  - cracking of SLB and ZTY is under investigation

# Collaboration

- Industry (Solar Turbines, Westinghouse, GEAE)
  - provided some superalloy substrates and guidance on testing and evaluation
- NASA Glenn Research Center
  - provided plasma spray and sputtering facilities

# **Future Activities for FY00**

- Process and characterize thin oxygen barrier coatings of Ge-doped silica and mullite
- Process optimization of hot corrosion barrier coatings.
- Investigate long-term hot corrosion and air thermal cycling of hot corrosion barrier coatings
- Burner rig test of selected dual layer coating systems
- Make recommendation of dual layer coating systems for scale up.