Current EBC Development and Testing at NASA

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Outline

• Background
• EBC Testing Facilities
• EBC Development
  1. **Slurry** EBCs *w/ 2700F Bond Coat Capability*
  2. **Plasma-Sprayed** Modified $\text{Yb}_2\text{Si}_2\text{O}_7$ EBCs *w/ Si Bond Coat*
Environmental Barrier Coating (EBC)

- An external coating to protect CMCs from rapid recession by H₂O
- Enabling technology for CMCs

**Recession Model**

\[
\text{SiO}_2 (s) + 2\text{H}_2\text{O} (g) = \text{Si(OH)}_4 (g)
\]

\[
\text{Volutility} \propto \frac{\nu^{1/2} \times P(\text{H}_2\text{O})^2}{P_{\text{TOTAL}}^{1/2}}
\]

ν : gas velocity
P(\text{H}_2\text{O}) : water vapor pressure
P_{\text{TOTAL}} : total pressure


**Solar Turbine Engine Test**


**Gen 1 EBC**


Silicon bond coat (mp = 1416°C)


**Gen 2 EBC**


**Next Gen EBC**

(NASA Developmental)

Bond coat mp > 1500°C

### Key EBC Failure Modes

<table>
<thead>
<tr>
<th>Steam oxidation</th>
<th>Recession by water vapor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Erosion</td>
<td>CMAS: (Ca-Mg-Al-Silicates)</td>
</tr>
</tbody>
</table>

**Synergy between failure modes likely leads to EBC failure**

Steam Oxidation-Induced EBC Failure

**15,144-h Solar Combustor Liner Engine Test**


**5,366-h Rig Test**

**NASA Steam Cycle Test (Si/Yb₂Si₂O₇)**

2400F, 90% H₂O-O₂, 550 hr/550 cycles

**Failure mode is similar to TBC:** Failure driven by the stored energy in the ceramic and TGO and the decrease in the toughness of the bond coat/TGO interface

• EBC Testing Facilities
## NASA EBC Test Rigs

<table>
<thead>
<tr>
<th>Rig</th>
<th>Capability</th>
<th>Failure modes to be tested</th>
</tr>
</thead>
</table>
| Mass Spectrometer            | $P(H_2O) = N/A$  
$v = N/A$  
$P_{total} = N/A$          | Recession (High pressure measurement of reaction products and Low pressure measurement of activities) |
| Steam TGA                    | $P(H_2O) = \text{up to } \sim 0.5 \text{ atm}$  
$v \sim 10 \text{ cm/s}$  
$P_{total} = 1 \text{ atm}$ | Recession (Initial screening of candidate materials).                                     |
| Mach 0.3 Burner rig          | $P(H_2O) = \sim 0.1 \text{ atm}$  
$v = 230 \text{ m/s}$  
$P_{total} = 1 \text{ atm}$ | CMAS, Erosion, FOD                                                                       |
| Steam cycling rig            | $P(H_2O) = \text{up to } \sim 1 \text{ atm}$  
$v = \text{a few cm/s}$  
$P_{total} = 1 \text{ atm}$ | Steam oxidation                                                                         |
| High heat flux laser rig     | $P(H_2O) = \text{ambient air}$  
$v = \text{zero}$  
$P_{total} = 1 \text{ atm}$ | Thermal fatigue in temp gradient  
Thermo-mechanical fatigue in temp gradient                                               |
| Natural gas burner rig       | $P(H_2O) \sim 0.5 \text{ atm}$  
$v \sim 250 \text{ m/s}$  
$P_{total} = 1 \text{ atm}$ | Recession  
Thermal fatigue in temp gradient  
(Coupons, Tensile bars, components)                                                    |
| CE-5 combustion rig          | $P(H_2O) \sim 3 \text{ atm}$  
$v \sim >30 \text{ m/s}$  
$P_{total} \sim 30 \text{ atm}$ | Steam oxidation w/ temperature gradient  
Recession  
(Coupons, Tensile bars, components)                                                    |

- Combinations of rigs to investigate synergy between failure modes
Steam TGA

Mass Spectrometer

<table>
<thead>
<tr>
<th></th>
<th>RE = Y</th>
<th>RE = Yb</th>
</tr>
</thead>
<tbody>
<tr>
<td>a(SiO₂)_{RE2Si2O7}</td>
<td>0.281</td>
<td>0.194</td>
</tr>
<tr>
<td>a(SiO₂)_{RE2SiO5}</td>
<td>0.000804</td>
<td>0.00298</td>
</tr>
<tr>
<td>a(SiO₂)<em>{RE2Si2O7}/a(SiO₂)</em>{RE2SiO5}</td>
<td>350</td>
<td>65</td>
</tr>
</tbody>
</table>
Steam Cycle Rig
(1 hr at Temp and 20 min at T<100°C)

- Temp: Up to ~2700°F
- Velocity: ~10 cm/s
- Water vapor: Up to ~0.9 atm
- Pressure: 1 atm

2400°F (1316°C) in 90% H2O-Bal O2, 100h/100 cycles

Silica scale is twice thicker on the backside
- Lower silica volatility on backside due to restricted gas flow

Silica scale was the same on both sides
- Gas velocity does not affect oxidation rates
Natural Gas Burner Rig

- Thermal cycling test under temp gradient
- Recession test
- Coupon and subcomponent test
- Feb in progress

<table>
<thead>
<tr>
<th>Temp</th>
<th>Up to ~2700F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Velocity</td>
<td>~250 m/s</td>
</tr>
<tr>
<td>Water vapor</td>
<td>~0.5 atm</td>
</tr>
<tr>
<td>Pressure</td>
<td>1 atm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>P(total), atm.</th>
<th>%H2O</th>
<th>v, m/s</th>
<th>vapor flux</th>
<th>mass loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>steam tube</td>
<td>1</td>
<td>1</td>
<td>175</td>
<td>1.00</td>
<td>1</td>
</tr>
<tr>
<td>NG-O2</td>
<td>1</td>
<td>0.5</td>
<td>250</td>
<td>0.30</td>
<td>193</td>
</tr>
<tr>
<td>HPBR</td>
<td>15</td>
<td>0.1</td>
<td>30</td>
<td>0.24</td>
<td>155</td>
</tr>
<tr>
<td>HPBR</td>
<td>6</td>
<td>0.1</td>
<td>300</td>
<td>0.19</td>
<td>124</td>
</tr>
<tr>
<td>HPBR</td>
<td>6</td>
<td>0.1</td>
<td>185</td>
<td>0.15</td>
<td>97</td>
</tr>
<tr>
<td>HPBR</td>
<td>6</td>
<td>0.1</td>
<td>30</td>
<td>0.06</td>
<td>39</td>
</tr>
<tr>
<td>M0.3</td>
<td>1</td>
<td>0.1</td>
<td>100</td>
<td>0.01</td>
<td>5</td>
</tr>
<tr>
<td>CE-5</td>
<td>30</td>
<td>0.05</td>
<td>30</td>
<td>0.17</td>
<td>110</td>
</tr>
</tbody>
</table>

* per 1" dia. Sample

James Smialek, NASA

Sighting Glass
CE-5 Combustion Rig

• Coupon & Vane holder Designs
  • Button Sample Holder
    • (1) 1” dia button
    • Backside cooling
    • Fab in progress
  • Vane pack sample holder
    • (2) 3” x 3” vanes
    • Backside cooling
    • Fab in progress

• Flexible Configurations
  a) Either holder downstream as piggy-back to injector testing
  b) Coupon upstream + Vane downstream as stand alone testing
  c) Investigating “dog bone”, CMC panel, and combustor liner configurations

<table>
<thead>
<tr>
<th></th>
<th>Temp</th>
<th>Up to ~3000°F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Velocity</td>
<td>&gt;30 m/s</td>
</tr>
<tr>
<td></td>
<td>Water vapor</td>
<td>~3 atm</td>
</tr>
<tr>
<td></td>
<td>Pressure</td>
<td>~30 atm</td>
</tr>
</tbody>
</table>

Dual position configuration

Take-up spool
• EBC Development
  1. **Slurry** EBCs w/ 2700F Bond Coat Capability
     - EBC on CMC Coupons
     - EBC on SiC Heating Element
Slurry EBC Flow Diagram

Processing

- Planetary Mill (Submicron coating powders)
- Organic processing aids (Dispersant and Binder)
- Ball Mill (Coating powders + Processing aids + Solvent)
- Processing (Dip, Spin, Spin-Dip, Spraying, Paint)
- Drying (Burnout organic processing aids)
- Sintering (Consolidate coating)

Characterization

- Particle size analyzer
- Zeta potential
- Viscometer
- Dilatometer
- Test & Characterization
Steam Oxidation of Slurry EBCs on CMC at 2600F (1 hr at 2600F (1427°C) / 20 min at T<100°C, 90% H₂O)

- TGO (~4 µm at 100h) is thinner than PS-PVD baseline Yb₂Si₂O₇ by ~2.5x
- Optimization and long-term testing (future work)
Slurry EBC

As-Sintered Bond Coat

- Sintered at T>2700°F (1482°C)
- Good chemical compatibility between layers
- TGO ~4 μm
- Phase and chemical analysis in progress

2600°F (1427°C) in 90% H2O, 100h/100 cycles
Cyclic Oxidation of Slurry EBC on SiC Heating Element (1 hr at 2600F (1427C) / 20 min at T<100C, 90% H2O)

- High oxidation rate of uncoated SiC due to additives and high porosity
- EBC is effective in reducing oxidation rate in air and steam
• EBC Development

  2. Plasma-Sprayed Modified Yb$_2$Si$_2$O$_7$ EBCs (w/ Si Bond Coat)
Steam Oxidation of Si/Modified Yb$_2$Si$_2$O$_7$ at 2400F (1 hr at 2400F (1316°C) / 20 min at T<100C, 90% H2O)

- Some modified Yb$_2$Si$_2$O$_7$ EBCs significantly reduce steam oxidation rate of Si BC
- Optimization and long-term testing (future work)

Data Points: This study

Baseline vs. Modified $\text{Yb}_2\text{Si}_2\text{O}_7$

2400F (1316C) in 90% H2O, 500h/500 cycles

Modified $\text{Yb}_2\text{Si}_2\text{O}_7$
Summary

• H₂O is the predominant oxidant in EBC steam oxidation

• Oxidation-induced failure mechanism appears to be similar to TGO-driven TBC failure mechanism

• Potential for slurry-based 2700F (1427C) bond coat demonstrated
  • Phase/chemical analysis in progress
  • Optimization, long-term test, and CMAS study (future work)

• Modified APS Yb₂Si₂O₇ EBCs reduce TGO growth rates on Si bond coat at 2400F (1316C) by two orders of magnitude
  • Very effective in reducing oxidation rates
  • Phase/chemical analysis and CMAS study in progress
  • Optimization and long-term test (future work)
Acknowledgements

• Dagny Sacksteder (Summer Intern)
  - Help with slurry fabrication and SiC heating elements cycle test

• Bryan Harder (NASA)
  - Helpful discussion on EBC steam oxidation