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 Yb$_2$Si$_2$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**

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

\[
\text{Vollatily} \propto \sqrt[4]{\frac{P(H_2O)}{P_{\text{TOTAL}}}}^2
\]

- \( \nu \): gas velocity
- \( P(H_2O) \): water vapor pressure
- \( P_{\text{TOTAL}} \): total pressure


**Solar Turbine Engine Test**


**EBC**

- Gen 1 EBC (EPM: NASA-GE-PW-1997)
  - Silicon bond coat (mp = 1416°C)


- Next Gen EBC (NASA Developmental)
  - Bond coat mp > 1500°C

Key EBC Failure Modes

**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/Yb$_2$Si$_2$O$_7$)

2400F, 90% H$_2$O-O$_2$, 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>
<tbody>
<tr>
<td>Mass Spectrometer</td>
<td>$P(H_2O) = N/A$</td>
<td>recession (High pressure measurement of reaction products and Low pressure measurement of activities)</td>
</tr>
<tr>
<td></td>
<td>$v = N/A$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = N/A$</td>
<td></td>
</tr>
<tr>
<td>Steam TGA</td>
<td>$P(H_2O) = \text{up to } \sim 0.5\ \text{atm}$</td>
<td>recession (Initial screening of candidate materials).</td>
</tr>
<tr>
<td></td>
<td>$v \sim 10\ \text{cm/s}$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = N/A$</td>
<td></td>
</tr>
<tr>
<td>Mach 0.3 Burner rig</td>
<td>$P(H_2O) = \sim 0.1\ \text{atm}$</td>
<td>cmas, erosion, fod</td>
</tr>
<tr>
<td></td>
<td>$v = 230\ \text{m/s}$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = 1\ \text{atm}$</td>
<td></td>
</tr>
<tr>
<td>Steam cycling rig</td>
<td>$P(H_2O) = \text{up to } \sim 1\ \text{atm}$</td>
<td>Steam oxidation</td>
</tr>
<tr>
<td></td>
<td>$v = a\ \text{few cm/s}$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = 1\ \text{atm}$</td>
<td></td>
</tr>
<tr>
<td>High heat flux laser rig</td>
<td>$P(H_2O) = \text{ambient air}$</td>
<td>thermal fatigue in temp gradient</td>
</tr>
<tr>
<td></td>
<td>$v = \text{zero}$</td>
<td>thermo-mechanical fatigue in temp gradient</td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = 1\ \text{atm}$</td>
<td></td>
</tr>
<tr>
<td>Natural gas burner rig</td>
<td>$P(H_2O) \sim 0.5\ \text{atm}$</td>
<td>recession</td>
</tr>
<tr>
<td></td>
<td>$v \sim 250\ \text{m/s}$</td>
<td>thermal fatigue in temp gradient</td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} = 1\ \text{atm}$</td>
<td>(coupons, tensile bars, components)</td>
</tr>
<tr>
<td>CE-5 combustion rig</td>
<td>$P(H_2O) \sim 3\ \text{atm}$</td>
<td>steam oxidation w/ temperature gradient</td>
</tr>
<tr>
<td></td>
<td>$v \sim &gt;30\ \text{m/s}$</td>
<td>recession</td>
</tr>
<tr>
<td></td>
<td>$P_{\text{total}} \sim 30\ \text{atm}$</td>
<td>(coupons, tensile bars, components)</td>
</tr>
</tbody>
</table>

- 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(SiO_2)_{RE2Si2O7}</td>
<td>0.281</td>
<td>0.194</td>
</tr>
<tr>
<td>a(SiO_2)_{RE2SiO5}</td>
<td>0.000804</td>
<td>0.00298</td>
</tr>
<tr>
<td>a(SiO_2)<em>{RE2Si2O7} / a(SiO_2)</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>
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<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>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp</td>
<td>Up to ~3000F</td>
</tr>
<tr>
<td>Velocity</td>
<td>&gt;30 m/s</td>
</tr>
<tr>
<td>Water vapor</td>
<td>~3 atm</td>
</tr>
<tr>
<td>Pressure</td>
<td>~30 atm</td>
</tr>
</tbody>
</table>

Dual position configuration
Take-up spool

TC Probe
Air/H2O
Pyro
• 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<100C, 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

2600F (1427C) in 90% H2O, 100h/100 cycles

Yb2Si2O7-based
HfSiO4-based

80 µm

Bond Coat
CMC

80 µm

Bond Coat (Mullite-Based)

80 µm

CMC

20 µm

CMC

Bond Coat
TGO
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<100°C, 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)
Baseline vs. Modified $\text{Yb}_2\text{Si}_2\text{O}_7$

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