Calcium-Magnesium Aluminosilicate (CMAS) Interactions with Advanced Environmental Barrier Coating Material

Valerie L. Wiesner and Narottam P. Bansal
Materials & Structures Division
NASA Glenn Research Center

11th International Conference on Ceramic Materials and Components for Energy and Environmental Applications
Vancouver, B.C., Canada
June 18, 2015
Environmental Barrier Coatings for Ceramic Matrix Composites

- Improve air-breathing turbine efficiency by replacing metal-based components with ceramic matrix composites (CMCs)
- Environmental barrier coatings (EBCs) protect CMC components from oxidation and corrosion in hot section of gas turbine engines
  - Rare-earth silicates

Target: 1482ºC

Molten CMAS Damage to Protective Coatings

• Particulates (i.e. sand, volcanic ash) ingested by engine melt into Calcium-Magnesium-AluminoSilicate (CMAS) glass above 1200°C

• Molten CMAS degrade EBCs

➢ Need EBC materials resistant to CMAS glass attack above >1200°C
High-Temperature Interactions between EBC Material and CMAS Glass

Objective:

• Evaluate thermo-chemical interactions between yttrium disilicate ($Y_2Si_2O_7$) EBC material and a desert sand glass at temperatures 1200ºC-1500ºC

• **Yttrium Disilicate ($Y_2Si_2O_7$)**
  – Comparable coefficient of thermal expansion to silicon-based CMCs
  – Water vapor resistance

• **Desert Sand (CMAS) Glass**
  – Actual sand sample
  – Relevant CMAS composition to aviation

Preparation of Desert Sand Glass

- As-received desert sand melted into glass
  - Heated at 10\(^\circ\)C/min to 1550\(^\circ\)C (1h)
  - Quenched melt in water
  - Grind glass frit in planetary mill zirconia milling media
  - Pass through sieve (<297 µm)

- Chemical analysis of glass by inductively coupled plasma atomic emission spectrometry (ICP-AES)

<table>
<thead>
<tr>
<th>Composition (mol.%)</th>
<th>CaO</th>
<th>MgO</th>
<th>Al(_2)O(_3)</th>
<th>SiO(_2)</th>
<th>K(_2)O</th>
<th>Fe(_2)O(_3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desert sand glass</td>
<td>27.8</td>
<td>4</td>
<td>5</td>
<td>61.6</td>
<td>1</td>
<td>0.6</td>
</tr>
<tr>
<td>Common CMAS glass(^1,2)</td>
<td>33</td>
<td>9</td>
<td>13</td>
<td>45</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

- Desert sand glass comprised of CMAS and trace oxides

Evaluate sand (CMAS) glass interactions with $Y_2Si_2O_7$ material

- **CMAS glass on hot-pressed $Y_2Si_2O_7$ substrate**
  - Load substrate with CMAS glass $\sim 35$ mg/cm$^2$
  - 20h heat treatments at 1200$^\circ$C, 1300$^\circ$C, 1400$^\circ$C and 1500$^\circ$C in air
  - Evaluate microstructure and composition of $Y_2Si_2O_7$/CMAS glass interface with SEM/EDS and EPMA

- **Cold-pressed pellet of $Y_2Si_2O_7$ and CMAS glass**
  - 80 wt.% $Y_2Si_2O_7$, 20 wt.% CMAS glass
  - 20h heat treatments at 1200$^\circ$C, 1300$^\circ$C, 1400$^\circ$C and 1500$^\circ$C in air
  - Analyze resulting phases using XRD
CMAS Glass on $\text{Y}_2\text{Si}_2\text{O}_7$ Substrate

Heat $>1200^\circ\text{C}$, CMAS glass melts and penetrates/reacts with $\text{Y}_2\text{Si}_2\text{O}_7$ substrate

1. CMAS glass infiltration into $\text{Y}_2\text{Si}_2\text{O}_7$ substrate
2. Thermo-chemical interactions of $\text{Y}_2\text{Si}_2\text{O}_7$/CMAS glass
SEM Cross-Section of CMAS/$Y_2Si_2O_7$ substrate

- Scanning electron microscopy (SEM) to evaluate cross-sections of heat treated CMAS glass/$Y_2Si_2O_7$ substrates

Interface between $Y_2Si_2O_7$ substrate/CMAS glass after heat treatment
SEM Cross-Section of CMAS/Y$_2$Si$_2$O$_7$ substrate

- CMAS glass penetration into Y$_2$Si$_2$O$_7$ substrates
  - Infiltration depth increases with temperature

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>Depth of CMAS Infiltration</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200°C for 20h</td>
<td>12.7 ± 2.5 µm</td>
</tr>
<tr>
<td>1300°C for 20h</td>
<td>80.9 ± 14.2 µm</td>
</tr>
<tr>
<td>1400°C for 20h</td>
<td>215.8 ± 17.6 µm</td>
</tr>
<tr>
<td>1500°C for 20h</td>
<td>217.6 ± 19.6 µm</td>
</tr>
</tbody>
</table>

Interface between Y$_2$Si$_2$O$_7$ substrate/CMAS glass after heat treatment

- CMAS glass on surface
  - ~13 µm
  - ~218 µm
SEM Cross-Section of CMAS/$Y_2Si_2O_7$ substrate

- CMAS glass penetration into $Y_2Si_2O_7$ substrates
  - Infiltration depth increases with temperature

- Thermo-chemical interactions
  - Precipitation of alternate phase in CMAS glass and infiltrated region

![Image of SEM cross-section](image)

- CMAS glass on surface
- Alternate phase
- $Y_2Si_2O_7$ substrate

1200°C
Interface between $Y_2Si_2O_7$ substrate/CMAS glass after heat treatment

1500°C
Alternate phase

150 µm
EDS Mapping of Interaction Region

- Yttrium incorporated into CMAS glass
  - Yttrium signal detected above substrate surface in glass
  - $\text{Ca}_2\text{Y}_8(\text{SiO}_4)_6\text{O}_2$ oxyapatite silicate phase expected

- Calcium infiltrated $\text{Y}_2\text{Si}_2\text{O}_7$ substrate
  - Depth of calcium infiltration corresponds to microstructural deformation in interaction region

Interface between $\text{Y}_2\text{Si}_2\text{O}_7$ substrate and CMAS glass after 20h heat treatment at 1500ºC
Quantification of Composition by EPMA

• Electron probe micro-analysis (EPMA)
  – Evaluate composition along line normal to substrate surface

BSE image of $Y_2Si_2O_7$ substrate and CMAS glass after 20h heat treatment at 1500ºC
Quantification of Composition by EPMA

- Electron probe micro-analysis (EPMA)
  - Evaluate composition along line normal to substrate surface
  - Quantify variation in elemental composition from CMAS glass through $Y_2Si_2O_7$ substrate after various heat treatments

- Ca detected throughout CMAS glass and interaction region
- No Ca in substrate
- Minimal Al or Mg in interaction region

Compare Ca content in specimens heat treated at different temperatures
Average CaO Content by EPMA

- CaO content in glass decreases with temperature
- CaO content in interaction region constant
  - Depth of interaction region increases with temperature
- No CaO detected in substrate
Identifying Alternate Phase using XRD

- Heat treat powder pellets containing 80 wt.% EBC powder ($Y_2Si_2O_7$) and 20 wt.% CMAS glass
- Evaluate reacted pellet using X-ray diffraction (XRD)

Alternate phase: $Ca_2Y_8(SiO_4)_6O_2$ oxyapatite silicate phase
Conclusions and Current Efforts

• Desert sand (CMAS) glass reacted with $Y_2Si_2O_7$ yielding $Ca_2Y_8(SiO_4)_6O_2$ oxyapatite silicate phase
  – Formed by dissolution of $Y_2Si_2O_7$ in CMAS glass followed by precipitation during cooling
  – Similar reaction observed for $Y_2SiO_5$

• Depth of CMAS infiltration increased with increasing heat treatment temperature
  – More significant pore formation and microstructural deformation in interaction region compared with $Y_2SiO_5$

➢ Evaluate other advanced EBC materials’ high-temperature interaction with desert sand (CMAS) glass