Evaporative Metal Bonding of APMT to Nickel Superalloys

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The University of North Dakota (UND) Energy & Environmental Research Center (EERC) and Department of Mechanical Engineering are working with Siemens Energy to test a new method for joining high-temperature alloys for use in advanced high-hydrogen-gas-burning turbines.

- Developed models for designing clamping fixtures and zinc diffusion.
- Thin plates of oxidation- and spallation-resistant Kanthal APMT™ have been bonded to high-strength CM247LC and Rene® 80 using evaporative metal (EM) bonding.
- Bonded parts, with and without thermal barrier coatings (TBCs), will be tested for oxidation, corrosion, and spallation resistance at Siemens Energy.
- Gasifier sampling to determine appropriate corrosion conditions.
Characterization of Combusted Syngas Contaminants

- Information to be used in designing later corrosion testing – contaminants will not be similar to gasifier fly ash.
- Collection of microcontaminants in combusted syngas created in two pilot-scale gasifiers.
- Analysis of captured microcontaminants by SEM.
EERC Pilot-Scale Gasifiers

Entrained-Flow Gasifier (EFG)
1800°C, 300 psi

Fluid-Bed Gasifier (FBG)
800°C, 600 psi
Method 29
Sampling System
Eagle Butte Coal Ash Composition

The diagram represents the composition of Eagle Butte Coal Ash using the ternary phase diagram. It shows the percentage of CaO, SiO₂, and Al₂O₃. The specific point on the diagram indicates the concentration of these components in the ash.

- **CaO** (Calcium Oxide) is on one axis, ranging from 0% to 100%.
- **SiO₂** (Silicon Dioxide) is on another axis, also ranging from 0% to 100%.
- **Al₂O₃** (Aluminium Oxide) is on the third axis, ranging from 0% to 100%.

The point on the diagram suggests a specific composition of these components in the coal ash.
Advanced Technique Analysis for Eagle Butte Fly Ash

- Plot of individual fly ash minerals.
- Fly ash minerals behave differently on a particle-by-particle basis.
Results of FBG Particulate Analyses

- In the quenched syngas, the particulates are predominantly 0.1–0.5 μm in diameter.
- We were not able to get good energy-dispersive x-ray analyses of the small predominant particles.
- XPS shows that the average composition of the syngas particles is very close to that of the polycarbonate filter and is most likely carbonaceous soot.
- In the combusted syngas, the carbonaceous particles are more spherical than in the syngas and slightly larger, typically 0.2–2 μm.
- The combusted particles show more O, N, and S than the noncombusted particles.
- Ion etching shows that the increased O, N, and S were confined to the surface of the particles.
Results of EFG Particulate Analyses

• No submicron particulates were seen on the syngas filter either because the filter had softened or there is just less soot formed due to the lower tar formation in an EFG.

• Flakes of iron oxide were collected from the syngas that came from system surfaces. They contained some C, Na, Cl, S, and Zn.

• Combusted syngas contained 0.1 to 0.3 micron soot particles.

• Some soot is collected even when burning only natural gas, but particles are smaller and fewer than when burning syngas.
\[ \frac{\partial C}{\partial t} = D \left[ \frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2} \right] \]

3-D Diffusion with Constant Diffusivity

No analytical solution exists for the combination of initial and boundary conditions present in the experimental setup (midline symmetry assumed):

- **100% Zn** (initial)
- **0% Zn** (boundary)
• A finite difference algorithm was implemented within MATLAB to solve the diffusion equation.
• The ‘hopscotch’ iterative solver was implemented to improve accuracy and computational efficiency.
• Algorithm assumes initial midline concentration of Zn, assumes constant diffusivity, uses a rectangular geometry and allows for different mesh size in each direction (x, y, z).
- ~15 wt% initial centerline composition for model
- D for Zn in APMT ~2.7 E-12 m²/s
- D for Zn in Rene 80/CM 247 ~4 E-14 m²/s
Jig Assembly for Fabrication of Samples
Grooved Backing Plate
Normal Stresses in the Plane for the APMT for CM247LC and Rene 80 at 1200°C
TZM Mo Jig Equivalent Stresses for CM247LC and Rene 80 at 1200°C
Base Metal–TZM Mo Jig Normal Stresses for CM247LC and Rene 80 at 1200°C
Assembled Jib in Preparation to Bond the APMT Plate to the Superalloy Block
## Alloy Compositions

### Composition of Kanthal APMT, wt% – Dispersion-Strengthened

<table>
<thead>
<tr>
<th></th>
<th>Cr</th>
<th>Al</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>APMT</td>
<td>22</td>
<td>5</td>
<td>3</td>
<td>0.4</td>
<td>0.7</td>
<td>Balance</td>
</tr>
</tbody>
</table>

### Composition of CM247 LC, wt% – Gamma Prime-Strengthened

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Al</th>
<th>Ti</th>
<th>Co</th>
<th>Mo</th>
<th>Ta</th>
<th>W</th>
<th>Nb</th>
<th>Hf</th>
<th>Mn</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>CM247LC</td>
<td>–</td>
<td>Balance</td>
<td>8.1</td>
<td>5.6</td>
<td>0.7</td>
<td>9.5</td>
<td>0.5</td>
<td>3.2</td>
<td>9.5</td>
<td>0.1</td>
<td>1.4</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

### Composition of Rene 80, wt% – Gamma Prime-Strengthened

<table>
<thead>
<tr>
<th></th>
<th>Cr</th>
<th>C</th>
<th>Mo</th>
<th>W</th>
<th>Ti</th>
<th>Nb</th>
<th>Co</th>
<th>Al</th>
<th>B</th>
<th>Fe</th>
<th>Zr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rene 80</td>
<td>14.2</td>
<td>0.16</td>
<td>4.0</td>
<td>4.1</td>
<td>5.1</td>
<td>0.03</td>
<td>9.4</td>
<td>3.0</td>
<td>0.02</td>
<td>0.10</td>
<td>0.04</td>
<td>Balance</td>
</tr>
</tbody>
</table>
Bond Line Between CM247LC (bottom) and APMT (top) at 100× Magnification
Microstructure of EM Joints

- Scanning electron microscopy (SEM) photo (top) and x-ray map (bottom).
- Needle growth and interdiffusion to create a joint stronger than the APMT.
- Nickel diffuses up to 700 µm into APMT.
- Iron diffuses 200 µm into CM247LC.
### Morphologies and Compositions in the APMT and the CM247LC Near the Bond Line at 1000×

#### Table of Element Compositions

<table>
<thead>
<tr>
<th>Element Description</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>O</th>
<th>Al</th>
<th>Mo</th>
<th>Hf</th>
<th>Ta</th>
<th>Ti</th>
<th>Mn</th>
<th>Zr</th>
<th>Co</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>C: Large, dark gray areas in APMT; from 0 to 30 µm into APMT</td>
<td>18.8</td>
<td>57.8</td>
<td>4.4</td>
<td>0.0</td>
<td>14.2</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>4.8</td>
<td>0.0</td>
</tr>
<tr>
<td>F: Large, light gray regions in APMT; present from 0 to 40 µm</td>
<td>41.2</td>
<td>29.1</td>
<td>13.7</td>
<td>0.0</td>
<td>2.6</td>
<td>1.1</td>
<td>1.0</td>
<td>1.5</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>5.3</td>
<td>4.5</td>
</tr>
<tr>
<td>G: Large, dark gray regions in CM247LC; present from 0 to 225 µm into CM247LC</td>
<td>15.1</td>
<td>58.7</td>
<td>2.8</td>
<td>0.0</td>
<td>12.8</td>
<td>0.0</td>
<td>1.8</td>
<td>3.1</td>
<td>0.4</td>
<td>0.3</td>
<td>0.0</td>
<td>5.0</td>
<td>0.0</td>
</tr>
<tr>
<td>H: Large, white regions in CM247LC; present from 40 to 260 µm</td>
<td>14.5</td>
<td>7.0</td>
<td>8.8</td>
<td>0.0</td>
<td>0.9</td>
<td>2.6</td>
<td>3.5</td>
<td>57.0</td>
<td>0.7</td>
<td>0.7</td>
<td>0.0</td>
<td>4.3</td>
<td>0.0</td>
</tr>
<tr>
<td>I: Large, light gray regions between precipitates in CM247LC</td>
<td>31.5</td>
<td>34.8</td>
<td>13.5</td>
<td>0.0</td>
<td>3.4</td>
<td>1.5</td>
<td>2.5</td>
<td>3.6</td>
<td>0.9</td>
<td>0.8</td>
<td>0.0</td>
<td>7.5</td>
<td>0.0</td>
</tr>
</tbody>
</table>
Precipitates at the Bond Line of a CM247LC–APMT Joint at 5000×

<table>
<thead>
<tr>
<th>Element, wt%</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>O</th>
<th>Al</th>
<th>Mo</th>
<th>Hf</th>
<th>Ta</th>
<th>Ti</th>
<th>Mn</th>
<th>Zr</th>
<th>Co</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>A: Small white specks found along bond line</td>
<td>7.1</td>
<td>12.8</td>
<td>4.9</td>
<td>0.0</td>
<td>2.7</td>
<td>0.0</td>
<td>27.8</td>
<td>25.0</td>
<td>4.3</td>
<td>0.0</td>
<td>0.0</td>
<td>2.2</td>
<td>13.2</td>
</tr>
<tr>
<td>B: Black specks found along bond line</td>
<td>6.6</td>
<td>8.2</td>
<td>2.9</td>
<td>38.8</td>
<td>32.6</td>
<td>0.8</td>
<td>7.2</td>
<td>0.0</td>
<td>1.0</td>
<td>0.0</td>
<td>0.0</td>
<td>1.9</td>
<td>0.0</td>
</tr>
</tbody>
</table>
Precipitates Within the APMT Near a CM247LC–APMT Joint at 5000×

<table>
<thead>
<tr>
<th>Element, wt%</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>O</th>
<th>Al</th>
<th>Mo</th>
<th>Hf</th>
<th>Ta</th>
<th>Ti</th>
<th>Mn</th>
<th>Zr</th>
<th>Co</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>D: Small, white precipitates in APMT</td>
<td>8.3</td>
<td>3.3</td>
<td>3.0</td>
<td>0.0</td>
<td>0.6</td>
<td>0.2</td>
<td>23.8</td>
<td>42.8</td>
<td>14.5</td>
<td>0.1</td>
<td>2.7</td>
<td>0.7</td>
<td>0.0</td>
</tr>
<tr>
<td>E: Dark gray, small, and circular in APMT; present in APMT past 15 µm</td>
<td>22.7</td>
<td>49.5</td>
<td>6.5</td>
<td>0.0</td>
<td>13.4</td>
<td>0.3</td>
<td>1.0</td>
<td>2.8</td>
<td>0.2</td>
<td>0.2</td>
<td>0.0</td>
<td>3.4</td>
<td>0.0</td>
</tr>
</tbody>
</table>
Bond Line Between Rene 80 (bottom) and APMT (top) at 100× Magnification
Morphologies Near the Bond Line in an APMT–Rene 80 Joint at 1000×

<table>
<thead>
<tr>
<th>Element, wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
</tr>
<tr>
<td>J: Black and round precipitates in bond line; diffused up to 30 µm into base metal</td>
</tr>
<tr>
<td>K: Black precipitates in Rene 80; formed approx. 30–45 µm below bond line</td>
</tr>
<tr>
<td>L: Light gray, skinny, and long precipitates in Rene 80; formed approx. 45–60 µm below bond line</td>
</tr>
<tr>
<td>M: Gray regions in between other ppt.</td>
</tr>
</tbody>
</table>

![Image of micrograph showing morphologies near the bond line in an APMT–Rene 80 joint at 1000× magnification.](image-url)
Precipitates in the APMT Near an APMT–Rene 80 Joint at 5000×

<table>
<thead>
<tr>
<th></th>
<th>Element, wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>N: White specks in APMT</td>
<td>Fe 50.7, Ni 3.6, Cr 11.4, O 0.8, C 13.0, Al 2.5, Mo 0.0, Ti 2.0, Co 0.0, W 0.0, Zr 7.7, Y 0.0, Si 8.3</td>
</tr>
<tr>
<td>O: Black specks in APMT; from 0 to 30 µm</td>
<td>Fe 44.4, Ni 2.7, Cr 10.6, O 15.0, C 5.7, Al 12.6, Mo 1.3, Ti 0.0, Co 0.0, W 0.0, Zr 0.0, Y 7.7, Si 0.0</td>
</tr>
<tr>
<td>P: Gray region in APMT</td>
<td>Fe 68.3, Ni 3.8, Cr 15.5, O 0.0, C 5.2, Al 4.4, Mo 2.8, Ti 0.0, Co 0.0, W 0.0, Zr 0.0, Y 0.0, Si 0.0</td>
</tr>
<tr>
<td>Q: Gray region</td>
<td>Fe 69.7, Ni 4.8, Cr 15.3, O 0.5, C 3.0, Al 3.9, Mo 2.6, Ti 0.0, Co 0.0, W 0.0, Zr 0.0, Y 0.0, Si 0.2</td>
</tr>
</tbody>
</table>

The table lists the element concentrations in different regions of the APMT near an APMT–Rene 80 joint at a magnification of 5000×.
EM Bonding Initial Observations

• One bond for each superalloy was weak, likely due to oxidation during a month long period between sandblasting and bonding.
• Previous tests with CM247LC show bond is stronger than the APMT.
• The procedure allows all of the zinc to diffuse out down to the detection limit (0.1%) and evaporate from the surface.
• Most zinc diffusion occurs through the APMT.
• There is significant interdiffusion between the alloys, especially iron, nickel, tantalum, and hafnium, more with CM247LC than with Rene80.
• The interdiffusion causes precipitation of different phases near the bond line.
Future Work

- Siemens Energy will perform precipitation hardening procedure on bonded superalloys.
- TBC will be applied to the APMT layer on a portion of the samples.
- They will then perform their standard oxidation, spallation resistance, and hot corrosion tests on the samples and compare to previous results for unbonded samples.
- EERC will perform hardness tests and SEM analyses on nonhardened and hardened crosssections.
- EERC will perform SEM analyses on oxidized and corroded samples. Will aluminum cross from the superalloy (source) to the APMT (sink)?
Acknowledgments

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