Corrosion Performance of Structural Alloys and Weldments in Simulated Oxy-fuel Environments

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1. Corrosion of base alloys
2. Mechanistic understanding of corrosion
3. Performance of weldment specimens
Corrosion of Base Alloys
## Current List of Alloys in the Study

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Fe</th>
<th>Other</th>
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</thead>
<tbody>
<tr>
<td>153MA</td>
<td>0.05</td>
<td>18.4</td>
<td>9.5</td>
<td>0.6</td>
<td>1.4</td>
<td>0.2</td>
<td>Bal</td>
<td>N 0.05, Nb 0.07, V 0.2</td>
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<tr>
<td>253MA</td>
<td>0.09</td>
<td>20.9</td>
<td>10.9</td>
<td>0.6</td>
<td>1.6</td>
<td>0.3</td>
<td>Bal</td>
<td>N 0.19, Ce 0.04</td>
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<tr>
<td>800H</td>
<td>0.08</td>
<td>20.1</td>
<td>31.7</td>
<td>1.0</td>
<td>0.2</td>
<td>0.3</td>
<td>Bal</td>
<td>Al 0.4, Ti 0.3</td>
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<tr>
<td>MA956</td>
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<td>-</td>
<td>-</td>
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<td>Bal</td>
<td>Al 4.5, Ti 0.5, Y2O3 0.6</td>
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<tr>
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<td>26.7</td>
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<td>0.5</td>
<td>-</td>
<td>Bal</td>
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<tr>
<td>WASP</td>
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<td>Bal</td>
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<td>0.75</td>
<td>4.3</td>
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<td>Co 12-15, Ti 2.6-3.25, Al 1.0-1.5, Zr 0.1</td>
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<tr>
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<td>0.9</td>
<td>Co 12.5, Al 1.2, Ti 0.3</td>
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<tr>
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<td>-</td>
<td>-</td>
<td>-</td>
<td>3.0</td>
<td>Nb 5.0, Al 0.5, Ti 0.9, B 0.002</td>
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<td>2.5</td>
<td>Nb 3.7, Al 0.2, Ti 0.2</td>
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<tr>
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<td>1.2</td>
<td>W 14, Al 0.3, La 0.015</td>
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<td>602CA</td>
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<td>62.6</td>
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<td>0.1</td>
<td>-</td>
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<td>Al 2.3, Ti 0.13, Zr 0.19, Y 0.09</td>
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<tr>
<td>693</td>
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<td>Al 3.3, Nb 0.67, Ti 0.4, Zr 0.03</td>
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<tr>
<td>214</td>
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<td>Bal</td>
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<td>Al 3.7, Zr 0.01, Y 0.006</td>
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<tr>
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<td>0.06</td>
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<td>6</td>
<td>0.7</td>
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<td>58</td>
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<td>0.5</td>
<td>10</td>
<td>5</td>
<td>Al 1.5, Ti 3.1, Co11, B 0.006</td>
</tr>
</tbody>
</table>
Laboratory Test Details

Key variables: Temperature, time, alloy composition

Materials: Fe- and Ni-base alloys

Environments: Gas A: 68.1%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂
              Gas B: 95%CO₂-0.99%SO₂-3.97%O₂

Ash mixture
Eastern ash: 90% (SiO₂:Al₂O₃:Fe₂O₃ = 1:1:1) and 10%(Na₂SO₄:K₂SO₄ = 1:1)
Western ash: 36%SiO₂-16%Al₂O₃-9%Fe₂O₃-29%CaO and 10%(Na₂SO₄:K₂SO₄ = 1:1)

Test temperature range: 750 °C

Test times: up to 6,300 h

Specimen evaluation: weight change
                      scanning electron microscopy
                      energy dispersive X-ray analysis
                      X-ray diffraction
                      synchrotron nanobeam analysis
Incubation period for ash corrosion

Alloy 253
1200h before incubation time

3600h after incubation time

Corrosion rate = \frac{\text{Depth}}{\text{Total exposure time} - \text{incubation time}}
Effect of surface condition on incubation time

Incubation time: Strongly affected by surface condition
Short term test results not reliable due to the effect of incubation time.

Eastern ash, Gas A: 68.1%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂, 750°C

General character of localized corrosion

Polished Non-polished

602CA, 300h

Weight loss (mg/cm²)

Non-polished

Polished

0 500 1000 1500 2000
t (h)
How to determine incubation time?

Eastern ash, Gas A: 68.1%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂, 750°C

Cut sample to obtain scale thickness vs Significant weight change

Realistic method+ not change surface condition
Weight Change data after exposure

Western ash:
16%Al₂O₃, 36%SiO₂, 9%Fe₂O₃, 29%CaO, 5%K₂SO₄, 5%Na₂SO₄

Gas: 68.1%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂

Eastern ash:
30%Al₂O₃, 30%SiO₂, 30%Fe₂O₃, 5%K₂SO₄, 5%Na₂SO₄
Effect of ash chemistry on incubation time

Western ash: 16%Al$_2$O$_3$, 36%SiO$_2$, 9%Fe$_2$O$_3$, 29%CaO, 5%K$_2$SO$_4$, 5%Na$_2$SO$_4$

Eastern ash: 30%Al$_2$O$_3$, 30%SiO$_2$, 30%Fe$_2$O$_3$, 5%K$_2$SO$_4$, 5%Na$_2$SO$_4$

Gas: 68.14%CO$_2$-26.9%H$_2$O-0.99%SO$_2$-3.97%O$_2$
Effect of ash chemistry on corrosion rate

Western ash: 16% Al$_2$O$_3$, 36% SiO$_2$, 9% Fe$_2$O$_3$, 29% CaO, 5% K$_2$SO$_4$, 5% Na$_2$SO$_4$

Eastern ash: 30% Al$_2$O$_3$, 30% SiO$_2$, 30% Fe$_2$O$_3$, 5% K$_2$SO$_4$, 5% Na$_2$SO$_4$

Gas: 68.14% CO$_2$-26.9% H$_2$O-0.99% SO$_2$-3.97% O$_2$

CaO addition reduces corrosion rates of most Co-containing and Ni-base alloys
Mechanistic Understanding of Corrosion Processes
Ash corrosion mechanism

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Melting temperature (°C)</th>
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<tbody>
<tr>
<td>$\text{K}_2\text{Al(SO}_4\text{)}_3$</td>
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</tr>
<tr>
<td>$\text{Na}_3\text{Al(SO}_4\text{)}_3$</td>
<td>646</td>
</tr>
<tr>
<td>$\text{K}_3\text{Fe(SO}_4\text{)}_2$</td>
<td>618</td>
</tr>
<tr>
<td>$\text{Na}_3\text{Fe(SO}_4\text{)}_3$</td>
<td>624</td>
</tr>
<tr>
<td>$\text{KFe(SO}_4\text{)}_2$</td>
<td>694</td>
</tr>
<tr>
<td>$\text{NaFe(SO}_4\text{)}_2$</td>
<td>690</td>
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</table>

Do all sulfates decompose?

<table>
<thead>
<tr>
<th></th>
<th>Western</th>
<th>Eastern</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{SiO}_2$</td>
<td>36.04</td>
<td>40.35</td>
</tr>
<tr>
<td>$\text{Fe}_2\text{O}_3$</td>
<td>5.86</td>
<td>28.33</td>
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<tr>
<td>$\text{Al}_2\text{O}_3$</td>
<td>16.84</td>
<td>22.56</td>
</tr>
<tr>
<td>$\text{CaO}$</td>
<td>21.61</td>
<td>2.62</td>
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<tr>
<td>$\text{MgO}$</td>
<td>5.06</td>
<td>0.69</td>
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<td>$\text{SrO}$</td>
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<td>0.09</td>
</tr>
<tr>
<td>$\text{BaO}$</td>
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<td>0.11</td>
</tr>
<tr>
<td>$\text{Na}_2\text{O}$</td>
<td>1.69</td>
<td>0.41</td>
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<tr>
<td>$\text{K}_2\text{O}$</td>
<td>0.5</td>
<td>1.28</td>
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<tr>
<td>$\text{TiO}_2$</td>
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<td>1.04</td>
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<tr>
<td>$\text{MnO}_2$</td>
<td>0.02</td>
<td>0.05</td>
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<tr>
<td>$\text{P}_2\text{O}_5$</td>
<td>1</td>
<td>0.22</td>
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<tr>
<td>$\text{SO}_3$</td>
<td>9.09</td>
<td>2.25</td>
</tr>
</tbody>
</table>
Decomposition of sulfates

$$K_3Al(SO_4)_3 = KAl(SO_4)_2 + K_2SO_4$$

$$4KAl(SO_4)_2 = 2K_2SO_4 + 2Al_2O_3 + 6SO_2 + 3O_2$$

Gas: 68.14%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂
Change of the kinetics of decomposition of sulfates

4KAl\((\text{SO}_4)_2\) = 2K\(_2\)SO\(_4\) + 2Al\(_2\)O\(_3\) + 6SO\(_2\) + 3O\(_2\)

If a stable K\(_3\)Al\((\text{SO}_4)_3\) exists at 750\(^\circ\)C, low stability sulfates such as KAl\((\text{SO}_4)_2\), Na\(_3\)Al\((\text{SO}_4)_3\), and K\(_3\)Fe\((\text{SO}_4)_3\) would not simply decompose at ~650\(^\circ\)C

Peak at 700\(^\circ\)C?

\(p_{O_2} = 0.04\) atm

\[ K = \frac{1}{p_{SO_2}^6 \cdot p_{O_2}^3} \] without solvent

\[ K = \frac{C_{\text{KAl(SO}_4)_2}}{p_{SO_2}^6 \cdot p_{O_2}^3} \] in K\(_3\)Al\((\text{SO}_4)_3\) solution

![Graph of weight change vs temperature for different compositions of x%KAl\((\text{SO}_4)_2\) - (100-x)%K\(_3\)Al\((\text{SO}_4)_3\) in the temperature range of 500 to 1000\(^\circ\)C. Peaks at 835\(^\circ\)C, 915\(^\circ\)C, 650\(^\circ\)C, 825\(^\circ\)C, 975\(^\circ\)C, 912\(^\circ\)C, and 790\(^\circ\)C are indicated.]
Why corrosion rate with high CaO ash is lower?

$$2\text{Al}_2\text{O}_3 + 6\text{K}_2\text{SO}_4 + 6\text{SO}_2 + 3\text{O}_2 = 4\text{K}_3\text{Al(SO}_4)_3$$ melting T $690 \degree $ C

$$\text{CoO} + \text{SO}_2 + 1/2\text{O}_2 = \text{CoSO}_4$$ melting T $735 \degree $ C

$$\text{NiO} + \text{SO}_2 + 1/2\text{O}_2 = \text{NiSO}_4$$ NiSO$_4$-Na$_2$SO$_4$ melting T $671 \degree $ C

$$3\text{CaO} + 2\text{K}_3\text{Al(SO}_4)_3 = 3\text{CaSO}_4 + 3\text{K}_2\text{SO}_4 + \text{Al}_2\text{O}_3$$ melting T $1460 \degree $ C $1069 \degree $ C

$$3\text{CaSiO}_3 + 2\text{K}_3\text{Al(SO}_4)_3 = 3\text{CaSO}_4 + 3\text{K}_2\text{SO}_4 + 3\text{SiO}_2 + \text{Al}_2\text{O}_3$$

$$3\text{CaCO}_3 + 2\text{K}_3\text{Al(SO}_4)_3 = 3\text{CaSO}_4 + 3\text{K}_2\text{SO}_4 + 3\text{CO}_2 + \text{Al}_2\text{O}_3$$

Solid state reaction

$$\text{CoSO}_4 + \text{CaCO}_3 = \text{CaSO}_4 + \text{CoO} + \text{CO}_2$$

$$\text{NiSO}_4 + \text{CaCO}_3 = \text{CaSO}_4 + \text{NiO} + \text{CO}_2$$

$$\text{CoSO}_4 + \text{CaSiO}_3 = \text{CaSO}_4 + \text{CoO} + \text{SiO}_2$$

$$\text{NiSO}_4 + \text{CaCO}_3 = \text{CaSO}_4 + \text{NiO} + \text{CO}_2$$
Is there an inhibitor for ash corrosion?

Method to terminate ash corrosion:
convert low-melting-temperature sulfates to high melting temperature sulfates

\[
\begin{align*}
K_3\text{Al(SO}_4\text{)}_3 & \quad \text{CaSO}_4 & \quad K_2\text{SO}_4 & \quad K_2\text{SO}_4 - K_2\text{Ca}_2(\text{SO}_4)_3 \\
\text{melting T:} & \quad 690^\circ \text{C} & \quad 1460^\circ \text{C} & \quad 1069^\circ \text{C} & \quad 875^\circ \text{C}
\end{align*}
\]
X-ray diffraction of simulated Eastern and Western ashes after exposure to Gas A at 750°C for 300h

- ♥: CaSO₄
- ♠: K₃Al(SO₄)₃
- *: SiO₂
- ◊: K₂Ca₂(SO₄)₃
- ▽: Fe₂O₃
- ◇: Al₂O₃
- ♦: CaSiO₃
Carburization after incubation time

EDX mapping of the cross section of Alloy 740 after exposure to Gas B at 750°C for 3600h.
What does it incubate for?

Combination of sulfidation, oxidation and carburization

Parabolic rate law

Effects of gas contents

SO₂ CO₂

Crack defects

Linear rate law

Metallic networks

Weight loss (mg/cm²) vs. time (h)

0 500 1000 1500 2000 2500 3000

0 40 80 120 160
Self-assembled silicon oxide layer on alloy surface to resist ash corrosion

EDX of the cross section of Alloy 446 after exposure to the ash with a high calcium content (simulating US Western coal) plus alkali sulfates in Gas A (with steam) at 750°C for 3600 h.
Alloy corrosion rates under oxy-fuel and air combustion

Penetration rates of alloys after subtracting incubation period of alloys after exposure to coal ash and gas environments of oxy-fuel and air-combustion.

A: Fe-based alloys with Cr contents ~ 18 wt% and Ni-based Alloy 230 at 750°C.
B: Alloy 800 at 725°C in air combustion, 750°C in oxy-fuel, and 800°C in air combustion.
The rate at 700°C is taken from Castello. The corrosion rates of Alloy 617 exposed to a simulated air combustion environment are taken from Baker.

Alloy corrosion rates after incubation time under oxy-fuel combustion are generally higher than in air combustion.

Controversy results on complicate localized corrosion?

Test time: test results obtained below incubation time are not reliable and not realistic for long term application.
Temperature: ANL data at 750°C, other literature data at lower temperature, most from short time test without deleting incubation time.
Project Summary

- It is necessary to obtain corrosion rate after incubation time for long term application.
- The observed corrosion rates are similar to those reported from the UK research results under Task 2 in US/UK collaboration.
- Alloy corrosion rates after incubation time under oxy-fuel combustion condition are generally higher than in air combustion due to higher SO\textsubscript{2} and CO\textsubscript{2} concentrations under oxy-fuel combustion conditions.
- It is possible to reduce the long term alloy corrosion rate by increasing the calcium concentration in ash.
- It is possible to reduce the long term alloy corrosion rate by selecting alloys with adequate chemical composition as cladding or coating.
Corrosion Behavior of Weldments
### 3. Performance of weldment specimens

#### Weldment specimens

<table>
<thead>
<tr>
<th>Weldment</th>
<th>Alloys</th>
<th>Filler</th>
<th>ID</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>230</td>
<td>230-H</td>
<td>7-7814</td>
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<tr>
<td>B</td>
<td>214</td>
<td>214-H</td>
<td>6-7434</td>
</tr>
<tr>
<td>C</td>
<td>282</td>
<td>282-H</td>
<td>4-8352</td>
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<tr>
<td>D</td>
<td>263</td>
<td>263-SM</td>
<td>HT5371PKII</td>
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<tr>
<td>E</td>
<td>617</td>
<td>617-SM</td>
<td>XX2312UK</td>
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<td>F</td>
<td>740</td>
<td>740-SM</td>
<td>HT31305X</td>
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<tr>
<td>G</td>
<td>R41</td>
<td>R41-H</td>
<td>9-8317</td>
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<tr>
<td>H</td>
<td>WASP</td>
<td>WASP-H</td>
<td>9-6506</td>
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<tr>
<td>I</td>
<td>617</td>
<td>617-H</td>
<td>5-8806</td>
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<tr>
<td>J</td>
<td>263</td>
<td>263-H</td>
<td>1-9434</td>
</tr>
</tbody>
</table>

Alloys 214, 282, 263, R41, 740 are testing together with weldments

*H – Haynes International

**SM – Special Metals
Hardness of Weldments before exposure

Weldment A 230-Haynes

Weldment B 214-Haynes

Weldment C 282-Haynes

Weldment D 263-263
Hardness of Weldments before exposure

Weldment E 617-617

Weldment F 740-740

Weldment G R41-Haynes

Weldment H 263-263
Hardness of Weldments before exposure

617-Haynes

Weldment I

Weldment J

263-Haynes

Hardness (HV)

Distance (mm)
Photos of weldments after 300h exposure to 750°C

Gas composition: 68.14%CO₂-26.9%H₂O-0.99%SO₂-3.97%O₂

Western ash: 16%Al₂O₃, 36%SiO₂, 9%Fe₂O₃, 29%CaO, 5%K₂SO₄, 5%Na₂SO₄

Eastern ash: 30%Al₂O₃, 30%SiO₂, 30%Fe₂O₃, 5%K₂SO₄, 5%Na₂SO₄

Eastern ash sticks to sample surface

$$2\text{Fe}_2\text{O}_3 + 6\text{K}_2\text{SO}_4 + 6\text{SO}_2 + 3\text{O}_2 = 4\text{K}_3\text{Fe}({\text{SO}_4})_3 \text{ melting T 694 °C}$$

$$2\text{Al}_2\text{O}_3 + 6\text{Na}_2\text{SO}_4 + 6\text{SO}_2 + 3\text{O}_2 = 4\text{K}_3\text{Al}({\text{SO}_4})_3 \text{ melting T 654 °C}$$

$$3\text{CaCO}_3 + 2\text{K}_3\text{Fe}({\text{SO}_4})_3 = 3\text{CaSO}_4 + 3\text{K}_2\text{SO}_4 + 3\text{CO}_2 + \text{Fe}_2\text{O}_3$$

$$3\text{CaCO}_3 + 2\text{K}_3\text{Al}({\text{SO}_4})_3 = 3\text{CaSO}_4 + 3\text{K}_2\text{SO}_4 + 3\text{CO}_2 + \text{Al}_2\text{O}_3$$

melting T 1460 °C 1069 °C
Weight Change data after exposure

Without CaO

With CaO

---

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
</tr>
</thead>
<tbody>
<tr>
<td>230</td>
<td>214</td>
<td>282</td>
<td>263</td>
<td>617</td>
<td>740</td>
<td>R41</td>
<td>WASP</td>
<td>617</td>
<td>263</td>
</tr>
</tbody>
</table>
Incubation time for weldments

![Incubation time graph for different weldment alloys with and without CaO. The graph shows the incubation time in hours for each alloy, comparing the cases with and without CaO addition. The alloys are labeled A to J, with specific incubation times indicated for each.]
Weldment D (263) without CaO
Weldment F (740) without CaO
Weldment I (617H) without CaO
Weldment D (263) with CaO
Weldment F(740) with CaO
Weldment I (617H) with CaO
It is possible to reduce the long term corrosion rate by using alloys with smaller grain size.
Hardness data after exposure for 2400h

With CaO

Weldment E

With CaO

Weldment F

Without CaO

Weldment E

Without CaO

Weldment F
Carburization after incubation time

Weldment E(617) without CaO

Molybdenum carbide  Chromium carbide

Cross scan

Hardness (HV)

0 0.5 1 1.5 2 2.5

d (mm)
Carburization after incubation time

EDX map of Weldment E(617) without CaO
Summary continued

• The ash corrosion rates at fusion zone of weldments are generally less than at base alloys.

• Carburization after incubation time can increase the hardness.

• The performance of Alloy 740 weldment is the best among the weldments tested.

• It is possible to reduce the long term corrosion rate of weldment by increasing the calcium concentration in ash.

• It is possible to reduce the long term alloy corrosion rate by using alloys with smaller grain size or by using alloy compositions as cladding or coating.
Future Plans for the ANL research project

• Complete corrosion evaluation of structural alloys in oxy-fuel environments containing different ashes, alkali sulfates, and chlorides. This includes a range of coal ash chemistry and gas environments at temperatures up to 750 °C. Test has been performed in oxy-fuel gas environment with 200 ppm HCl at 750°C for 300h. Long term exposures are being continued.

• Experimentation to mitigate corrosion of structural alloys in both advanced steam-cycle and oxy-fuel combustion systems
  - Conventional coatings
  - Ash additives
  - Alloy surface modification using nano-structures
  - Study alloys with nano-grain size
Thank you

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