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Corrosion in a Molten Salt Environment

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Corrosion in a Molten Salt Environment

A thesis submitted in partial fulfillment of the requirement for the degree of Bachelor of Science in Physics from The College of William and Mary

by

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Corrosion in a Molten Salt Environment

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Abstract

Molten salt can be used to transfer and store energy, but presents challenges in terms of corrosion. In this research, we investigate the early stages of corrosion on stainless steel exposed to molten nitrate salts. Specifically, we are interested in the initial attack on grains and grain boundaries, including the effects of internal stress and/or a weakened passivation layer. We have developed procedures for exposing stainless steel coupons to a mixture of molten sodium nitrate and potassium nitrate at 565°C. Following exposure to molten salt, samples are analyzed using field emission scanning electron microscopy (FE-SEM) and energy-dispersive X-ray spectroscopy (EDX). We have captured a series of images showing the nanoscale growth of corrosion on 316SS from zero to 30 minutes of exposure. Based on our EDX data, we hypothesize that corrosion on plateau areas at these short time scales is proportional to \((1 - e^{t/\tau})\) for a characteristic time \(\tau\). We conclude that EDX appears to be a suitable method for estimating the level of corrosion on stainless steel exposed to molten salt.
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1 Introduction

1.1 Concentrating solar power

Molten salt can be used to transfer and store energy in concentrating solar power (CSP) plants. Examples of commercial molten salt CSP facilities include the Crescent Dunes plant near Tonopah, Nevada, and the Gemasolar plant near Seville, Spain [1,2]. In a typical plant design, an array of mirrors directs sunlight onto a receiver mounted on a central tower. The receiver heats the molten salt, which then is used to generate steam to run a turbine. Alternatively, the heated molten salt can be stored for later use when the sun is not out; this is a key advantage of molten salt CSP over photovoltaic solar power [3].

Although molten salt has desirable thermodynamic characteristics for CSP, the types of alloys and the varieties of salt used in plant designs must be chosen carefully to minimize the effects of corrosion. Further, the thermal cycling in CSP plants leads to strain on components—of chief concern is the receiver, which has been called a “low-cycle, high-strain amplitude fatigue environment” [4].

1.2 Alloys and molten salt

Research by Goods and Bradshaw has focused extensively on mass loss of stainless steel coupons exposed to molten salt [5]. Their exposures lasted for 120 to 7000 hours. More relevant here is the analysis of the corrosion formed on 304 and 316 stainless steel (hereafter, 304SS and 316SS). They found that the corrosion layer formed on the coupons included structures resembling hematite (Fe$_2$O$_3$), magnetite (Fe$_3$O$_4$), and sodium ferrite (NaFeO$_2$).

It is worth noting that the molten salt can decompose somewhat at temperatures around 550°C. It has been found that in an equimolar mixture of sodium nitrate (NaNO$_3$) and potassium nitrate (KNO$_3$), similar to the mixture we are using, the main reaction is given by $\text{NO}_3^- \equiv \text{NO}_2^- + \frac{1}{2}\text{O}_2$, where NO$_2^-$ is the nitrite ion [6]. It is this release of oxygen which
promotes the corrosion of stainless steel when exposed to the molten nitrates [7].

1.3 Our research

In this research, we focus on the microscopic surface behavior of corrosion, rather than mass loss. We are interested in the initial attack of corrosion on stainless steel over short time scales, on the order of hours or even minutes. Specifically, we seek to determine whether corrosion begins on the grain boundaries or surfaces, and whether a weakened passivation layer and/or internal stresses play a role in these processes.

2 Methods

2.1 General procedures

Figure 1: The oven setup, with steel drip tray and 10 mL alumina crucibles.

We are using an oven capable of the 500°C to 600°C temperatures typical in CSP facilities. Our stainless steel exposures occur at 565°C, a temperature typical of previous research and industrial use. With the built-in temperature controller reading 500°C, an external IR thermometer was in good agreement at 503°C. We are using samples of 304SS, 316SS, and
347SS. We immerse the stainless steel samples in crucibles of the widely used NaNO₃-KNO₃ (60%-40% by weight) molten salt mixture, commonly referred to as solar salt.

Before being placed in the oven, the crucibles, lids, stainless steel samples, and any additional hardware are cleaned using Fisherbrand Sparkleen detergent and de-ionized (DI) water in an ultrasonic bath, followed by three rinses of DI water and a final rinse of high purity isopropanol.

Although not implemented for the earliest runs, in accordance with the literature we decided to use a pre-heating method to remove any water from the solar salt. This involves heating the solar salt is at 200°C for 4 hours prior to going to 565°C. (Solar salt melts at approximately 222°C [6].)

Following exposure, samples are rinsed with DI water to remove any remaining solar salt. For the initial runs, when mass loss was a concern, crucibles and samples were weighed before and after exposure. Samples are subsequently analyzed using a field-emission scanning electron microscope (FE-SEM) for surface imaging and energy-dispersive X-ray spectroscopy (EDX) for compositional analysis.

2.2 Coupon samples

Our primary samples are rectangular coupons approximately 2 cm × 1 cm cut from stainless steel sheets. The sheet of 347SS was 0.64 mm thick (converted from specified 0.025 inch); sheets of 304SS and 316SS were 0.76 mm thick (converted from specified 0.030 inch). Each coupon has a series of notches along its top edge for easy identification of its material (see Figure 14), and the coupons are able to fit into the FE-SEM without needing to be cut. For intermediate length exposures (20 hours), these coupons were placed vertically in small 10 mL alumina crucibles filled with granulated solar salt (which melted as the oven reached its set temperature) and covered with alumina lids.

A revised exposure method was developed for short exposures (4 to 30 minutes). This procedure involves first drying and melting the solar salt in 10 mL alumina crucibles. Once
at temperature, a crucible’s lid is removed and a 1/4-20 screw is placed across the crucible rim as a sample holder; two nuts and two washers clamp the coupon in place (see Figure 2). The screw, nuts, and washers are made from 316SS. This setup allows for quick placement and retrieval of the sample assembly using tongs.

3 Experimental Development

3.1 C-ring samples and exposures

Initially, we considered using “c-ring” samples cut from sections of pipe, since they allow for stress to be applied via a nut and bolt across each c-ring. These were prepared as described in ASTM designation G38-01 [8]. Two rings can be clamped (via the bolt through each) into a holder, which is suspended through the oven’s top vent and can be lowered toward a crucible of molten salt. Following exposure, the rings are sectioned using a right-angle grinder with a cutoff wheel in order to produce samples small enough to be analyzed.

An initial test using c-ring samples revealed several issues. The amount of solar salt in our crucible had decreased by 66%; we later attributed this to the incompatibility of solar
salt with the glazed porcelain crucibles. The c-ring samples proved difficult to use, namely that their initial machining resulted numerous scratches and hence produced inconsistent samples to start with. Our setup of lowering the rings through the top of the oven did not allow for precise movements, and based on later results of coupons heated in air (middle row in Figure 14), leaving the coupons in air at 565°C could possibly alter the steel surface before molten salt exposure. The optimal exposure method for would be similar to that described above; i.e., opening the oven and using tongs to place a c-ring assembly over an appropriately sized crucible once the salt is already molten. If there is concern about water on the c-ring surface, perhaps the best solution would be to pre-heat the c-ring assemblies in a second oven at a more moderate temperature such as 200°C.

The initial c-ring experiment (with the anomalous salt loss) resulted in a dark, nearly black coating on the c-rings and other hardware (Figure 4). This dark coating is in agreement with Goods and Bradshaw’s observation of magnetite [5]. We saw no noticeable difference in tabletop SEM images between the stressed and unstressed c-rings. Additionally, as illustrated in Figure 8, the Phenom tabletop SEM produced subpar results at higher magnifications, so we switched to using the Hitachi FE-SEM exclusively.
In light of the above, we decided to focus on corrosion of coupon samples while optimizing the other experimental variables. Given the results and technical knowledge gained from working with coupon samples, and assuming the initial c-ring surface could be undamaged in machining or otherwise made uniform (perhaps using electropolishing), the molten salt project would now be equipped to revisit the effects of applied stress on stainless steel.
Figure 5: Tabletop SEM image of 304SS c-ring exposed to molten solar salt for approximately 1 week under stress.
Figure 6: Tabletop SEM image of 304SS c-ring exposed to molten solar salt for approximately 1 week without stress.
Figure 7: Tabletop SEM image of 304SS c-ring in as received condition.
Figure 8: Tabletop SEM image of 304SS c-ring in as received condition, showing limitations of the instrument at high magnification.
3.2 Hirox

Before performing our short exposures, we wanted to determine the thickness of the black coating on the coupons exposed to molten salt for intermediate durations (20 hours to several days). Using a 316SS coupon exposed for 3.5 days as a prototype, we found that scratching the surface using a rotary tool would leave deep grooves. However, on regions immediately adjacent to these grooves, the black coating would chip off, revealing the familiar plateau and boundary structure seen on the as received coupons. Using a Hirox digital microscope, we obtained a depth profile along the boundary between the coated and chipped-away regions, from which we estimated that the coating thickness of this particular sample was about 1 µm.

![Hirox image](image)

Figure 9: Hirox image (composite, for better depth of field) of 316SS coupon exposed to molten salt for 3.5 days. From left to right, the coated, chipped, and scratched regions are visible.

We used a Zeiss optical microscope to check the coupons after being scratched, to ensure that the requisite chipped regions were present. It turned out that the coatings on 304SS
Figure 10: Hirox 3D rendering of sample surface.

Figure 11: Depth profile along cross-section plane shown in Figure 10.

and 347SS did not chip as easily. Also, our short exposures on 316SS resulted in a coating which is almost certainly too thin to measure using this technique.
### 3.3 TOF-SIMS

With assistance from Amy Wilkerson at the Applied Research Center, we have obtained preliminary data on our 316SS samples using time-of-flight secondary ion mass spectrometry (TOF-SIMS). The compositional data showed high levels of polydimethylsiloxane (PDMS), a common contaminant, which is not surprising given that the samples were handled several times during FE-SEM/EDX analysis and then needed to be cut to fit into the TOF-SIMS chamber.

Examples of TOF-SIMS ion count plots are shown in Figures 12 and 13. Presently it is unclear whether the TOF-SIMS data are useful for dealing with the variations in EDX data discussed in Section 4.4. The plots of ion counts either appear to lack discernible features (Figure 12) or show features much larger than anything we have seen on the FE-SEM (Figure 13). The latter is likely due to contamination; the region in the upper right corner of each plot in Figure 13 likely still has a layer of solar salt on it, based on its high sodium content.

If the project is to continue using TOF-SIMS, a better handling protocol should be established. This would likely include rinsing the samples with IPA immediately before loading into TOF-SIMS (although a comparison of exposed coupons rinsed with DI water and IPA versus only DI water should be done first using FE-SEM and EDX) and storing the samples in cleaned glass containers. Also, if both TOF-SIMS and FE-SEM/EDX are to be performed on the same sample, performing TOF-SIMS first would likely reduce the risk of contamination.
Figure 12: Negative SIMS on 316SS coupon exposed to molten salt for 4 minutes.
Figure 13: Positive SIMS on 316SS coupon exposed to molten salt for 8 minutes.
4 Results

4.1 Intermediate exposures

We have exposed coupons of 304SS, 316SS, and 347SS to molten solar salt for 20 hours and have compared the FE-SEM images to those of as-received coupons (see Figures 15 to 20). On the as-received coupons, and on some of the exposed coupons, a structure of boundary and plateau areas is visible. We hypothesize that these boundaries and plateaus are related to the grain structure of the alloys. We performed EDX on both such areas on each coupon, except on the 316SS coupon exposed to solar salt, as its boundaries and plateaus were entirely obscured by corrosion growth. The results are shown in Table 1.

The coupons exposed to solar salt for 20 hours obtained a dark, nearly black coating, as seen in Figure 14. Based on Bradshaw and Goods [5] and due to its dark color, this coating is likely magnetite. EDX on these coupons revealed a depletion of chromium and nickel on the surfaces of the exposed coupons, relative to the as-received coupons.

As an indicator of the level of corrosion, we define the simplified oxygen composition as

\[
S = \frac{\text{atomic % oxygen}}{\text{atomic % iron} + \text{atomic % oxygen}}.
\]

On the coupons where boundaries and plateaus remained at least somewhat visible (304SS and 347SS), the value of \( S \) is higher on the plateaus than on the boundaries. This seems to imply that there is more corrosion on the plateaus than on the boundaries at time scales around 20 hours.
Figure 14: Comparison of coupons. Rows (top to bottom): as received, heated in oven without salt for 20 hours (as a control), heated in oven in solar salt for 20 hours. Columns (left to right): 304SS, 316SS, 347SS.

Table 1: EDX comparison of 304SS, 316SS, and 347SS, as-received and exposed for 20 hours to solar salt.
Figure 15: FE-SEM image of as-received 304SS.

Figure 16: FE-SEM image of 304SS exposed to molten solar salt for 20 hours.
Figure 17: FE-SEM image of as-received 316SS.

Figure 18: FE-SEM image of 316SS exposed to molten solar salt for 20 hours.
Figure 19: FE-SEM image of as-received 347SS.

Figure 20: FE-SEM image of 347SS exposed to molten solar salt for 20 hours.
4.2 Short exposures

Based on the FE-SEM images at 20 hours, we assessed that much shorter exposures were needed. Exposures of 4, 8, 15, and 30 minutes, along with an as-received coupon, using 316SS are shown in Figures 21 to 25. Table 2 shows the EDX results for these coupons.

<table>
<thead>
<tr>
<th>Exposure</th>
<th>Area</th>
<th>C</th>
<th>O</th>
<th>Mg</th>
<th>Si</th>
<th>S</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
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<tr>
<td>none</td>
<td>boundary</td>
<td>0.00</td>
<td>2.74</td>
<td>0.64</td>
<td>0.41</td>
<td>1.36</td>
<td>16.40</td>
<td>68.93</td>
<td>9.52</td>
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<tr>
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<td>25.95</td>
<td>8.23</td>
<td>0.00</td>
<td>0.86</td>
<td>1.11</td>
<td>11.14</td>
<td>45.42</td>
<td>7.28</td>
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<tr>
<td>4 minutes</td>
<td>boundary</td>
<td>22.86</td>
<td>12.16</td>
<td>0.71</td>
<td>0.99</td>
<td>1.67</td>
<td>9.77</td>
<td>45.48</td>
<td>6.37</td>
</tr>
<tr>
<td>4 minutes</td>
<td>plateau</td>
<td>23.04</td>
<td>11.07</td>
<td>0.64</td>
<td>1.09</td>
<td>1.40</td>
<td>11.18</td>
<td>44.81</td>
<td>6.78</td>
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<tr>
<td>8 minutes</td>
<td>boundary</td>
<td>27.67</td>
<td>15.47</td>
<td>0.36</td>
<td>0.86</td>
<td>1.07</td>
<td>8.71</td>
<td>40.48</td>
<td>5.36</td>
</tr>
<tr>
<td>8 minutes</td>
<td>plateau</td>
<td>18.04</td>
<td>14.18</td>
<td>0.43</td>
<td>2.09</td>
<td>1.90</td>
<td>11.17</td>
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<tr>
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<td>boundary</td>
<td>16.60</td>
<td>11.59</td>
<td>0.78</td>
<td>0.68</td>
<td>1.16</td>
<td>11.17</td>
<td>52.00</td>
<td>6.02</td>
</tr>
<tr>
<td>15 minutes</td>
<td>plateau</td>
<td>15.46</td>
<td>16.13</td>
<td>0.40</td>
<td>1.68</td>
<td>1.52</td>
<td>11.98</td>
<td>46.25</td>
<td>6.57</td>
</tr>
<tr>
<td>30 minutes</td>
<td>boundary</td>
<td>10.56</td>
<td>27.36</td>
<td>0.81</td>
<td>1.31</td>
<td>1.58</td>
<td>8.99</td>
<td>43.09</td>
<td>6.31</td>
</tr>
<tr>
<td>30 minutes</td>
<td>plateau</td>
<td>17.07</td>
<td>21.39</td>
<td>0.40</td>
<td>1.61</td>
<td>1.57</td>
<td>9.91</td>
<td>41.87</td>
<td>6.18</td>
</tr>
</tbody>
</table>

Table 2: EDX comparison of 316SS coupons at multiple durations of exposure to molten solar salt.

The coupons exposed for 30 minutes or less remained shiny and metallic in appearance, but obtained various colors of tints as in Figure 26. This is likely due to a very thin corrosion coating which generates thin-film interference effects. As would be expected, the EDX results show a trend of increasing oxygen content over time.
Figure 21: FE-SEM image of as-received 316SS (0 minutes of exposure).

Figure 22: FE-SEM image of 316SS exposed to molten solar salt for 4 minutes.
Figure 23: FE-SEM image of 316SS exposed to molten solar salt for 8 minutes.

Figure 24: FE-SEM image of 316SS exposed to molten solar salt for 15 minutes.
Figure 25: FE-SEM image of 316SS exposed to molten solar salt for 30 minutes.

Figure 26: Comparison of 316SS coupons. Top row (left to right): as received, exposed to solar salt for 4 minutes, exposed to solar salt for 8 minutes. Bottom row (left to right): exposed to solar salt for 15 minutes, exposed to solar salt for 30 minutes.
4.3 Model for finding coating thickness

Figure 27: Plot of simplified oxygen composition $S$ on boundaries and plateaus of 316SS coupons exposed to molten salt.

The values of $S$ for 316SS for short exposures and also 20 hours are plotted in Figure 27. Note that we use the same point for both data series at 20 hours, since by then the boundaries and plateaus were indistinguishable on the 316SS. Fitting the data to a simple model where the amount of unoxidized iron near the sample surface decreases exponentially with time, and hence the amount of oxidized iron goes as $(1 - e^{t/\tau})$, we hypothesize that corrosion at these very short time scales on the plateau areas has a characteristic time $\tau$ of 53 minutes. Although an analogous fit of the boundary data is shown, these data contain too much noise for us to draw a conclusion on the boundary areas. It is possible that corrosion in the these areas follows a different functional form than the plateaus.
The penetration depth of electrons in an SEM sample can be approximated by

\[ d \approx \frac{(10 \mu g/cm^2)(E_0)^{1.35}}{\rho} \]

where \( E_0 \) is the incident electron energy in keV and \( \rho \) is the sample density [9]. Since all of our FE-SEM and EDX analysis used 15 keV electrons, and taking \( \rho \approx 7.87 \) g/cm\(^3\) using the density of iron [10], we obtain \( d \approx 0.49 \) \( \mu \)m.

For purposes of our model, we assume that our EDX data provides us with average composition data over a volume \( V = a^2d \) where \( a^2 \) is the area over which the FE-SEM beam rasters. Before any molten salt exposure, we would expect \( S \) to be close to zero, so further investigation is needed to explain our seemingly high as-received values for oxygen composition. At long times, assuming we obtain only magnetite (Fe\(_3\)O\(_4\)) for example, we would expect \( S \) to saturate at \( \frac{4}{4+3} \approx 0.57 \). Therefore, for a given observation \( S_i \), we can form the following weighted average

\[ S_i \approx S_{saturate} \frac{x_i}{d} + S_{initial} \frac{d - x_i}{d} \]

where \( x_i \) is the sample coating thickness which we are solving for. If we assume \( S_{initial} = 0 \), we get

\[ x_i \approx d \frac{S_i}{S_{saturate}}. \]

From the data in Table 1, we can calculate the value of \( S \) for 316SS after 20 hours of exposure:

\[ S = \frac{47.62}{31.82 + 47.62} \approx 0.60 \]

which is in reasonable agreement with our expectation for \( S_{saturate} \) for pure magnetite.

Now, as an example, we consider the plateau data for the 316SS coupon exposed to molten salt for 4 minutes, and using \( S_{saturate} = 0.60 \), we calculate
\[ S = \frac{11.07}{44.81 + 11.07} \approx 0.20 \]

\[ x = 0.49 \, \mu m \left( \frac{0.20}{0.60} \right) = 0.16 \, \mu m. \]

Note that the formula for \( x_i \) is only valid for \( S_i \leq S_{\text{saturate}} \).

### 4.4 Variation in EDX data

As seen in Figure 27, there is variation present in the EDX data. All EDX data so far has been taken using a 200 second duration; a useful future experiment would be to choose a fixed sample area and take EDX data over a selection of durations, i.e., 200, 400, 600, and 800 seconds. Longer durations would increase the number of x-ray counts and thus should reduce the \( \frac{\sqrt{N}}{N} \) variation present in the x-ray emission process. Also, there is some instability in the FE-SEM electron beam current, especially when the electron gun has been recently switched on for the first time on a given day. In the future, monitoring the beam current more closely could improve the consistency of the EDX data.

It is also possible that the composition of the corrosion coating is not uniform over the entire area exposed to molten salt. As another test, I would recommend doing EDX over approximately 5 small areas of a single plateau and looking at the variation of \( S \) among those measurements. (The aforementioned could also be done over 5 boundary areas.) The average from the small areas of the plateau could then be compared to a measurement of \( S \) taken from a larger scanning area covering the majority of the plateau (Figure 28). Finally, assuming the larger scanning area produces results which agree with the smaller subareas, I would recommend comparing EDX data using a large scanning area on several far-apart plateau areas (Figure 29).
Figure 28: Illustration of a possible experiment to examine variations in EDX data across a single plateau area.
Figure 29: Illustration of a possible experiment to examine variations in EDX data across a wide area of a sample. Ideally, the areas could be several millimeters apart, rather than the \(~100\ \mu m\) shown here.
4.5 Magnesium accretion

It is worth noting that the exposed samples in Table 1 show enhanced magnesium levels relative to their as-received counterparts. In fact, on the 316SS coupon exposed for 20 hours, we found a crystal which contained 30% (atomic) magnesium.

![Figure 30: EDX data for the large octahedral crystal shown above revealed a magnesium content of approximately 30% (atomic). Other constituents were oxygen (53%), iron (15%), and chromium (2%).](image)

EDX on granules of NaNO₃ and KNO₃ used in our solar salt showed no magnesium. Therefore, the magnesium is likely coming from impurities in the alumina crucibles or from some sort of contamination on the stainless steel itself (despite magnesium not being a specified constituent of any of the three stainless steels). Further research would be needed to determine the source of this apparent magnesium accretion and its effects, if any, on the corrosion process.
5 Conclusion

In this research, we have examined the early stages of corrosion of stainless steel in molten solar salt. We have developed and tested procedures for exposing stainless steel samples to molten solar salt. Based on our EDX data, we hypothesize that the corrosion on plateau areas proceeds according to \((1 - e^{t/\tau})\) at these short time scales, with \(\tau = 53\) minutes. In the future, electropolishing or otherwise pre-treating stainless steel samples would provide a better comparison of corrosion on grains and grain boundaries, in order to investigate the effects of internal stress and/or a weakened passivation layer. Although further tests are needed, we have shown that EDX is a promising method for measuring the level of corrosion of stainless steel in molten salt.
Acknowledgements

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