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Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain Repository — Quarterly technical report No. 3

Dhanesh Chandra  
*University of Nevada, Reno, dchandra@scs.unr.edu*

Jaak J.K. Daemen  
*University of Nevada, Reno, daemen@mines.unr.edu*

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“Sub-surface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”

Quarterly Technical Report No. 3 (3rd Quarter of FY 2005)

Start Date of this Quarter, January 1, 2005 to March 31, 2005

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United States Department of Energy
Office of Civilian Defense Radioactive Waste Management
Yucca Mountain Project
1551 Hillshire Drive, Suite A
Las Vegas NV 89134-6321

and

Harry Reid Center
University of Nevada, Las Vegas
4505 Maryland Parkway
Las Vegas Nevada 89154-4009

-submitted by-
Metallurgical and Materials Engineering
Mail Stop 388
1664 N. Virginia Street
College of Engineering
University of Nevada, Reno
Reno NV 89557

Dhanesh Chandra, PI (Corrosion Team Leader)
Jaak Daemen (Co-PI)

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Statement of Work
The objective of this task is to conduct corrosion related research and predict the durability of rock-bolts and other underground metallic roof supports. In this period, we have performed oxidation of rock bolts made of high-strength low alloy steel, medium carbon steel (from the YM site), and Alloy 22 (reference material) specimens. Electrochemical and Stress corrosion tests were performed on the Alloy 22 samples.

General Statements
- Baseline Corrosion Rates have been established for Alloy 22 using YM waters.
- Rock bolts specimens have been obtained from manufacturers, as well as the raw materials for testing.
- Graduate students and Post Doctoral fellows have been trained for the QA work on the project.

Progress for the Period 01/01/05-03/31/05:
The SIP has been recently approved 12/17/04. We have started preliminary research on the following tasks:
Subtask 1: Selection of New High Strength Steels, Stainless Steels for Rock Bolts, Steel Sets and Perforated Roof Supports
Subtask 2: Electrochemical Corrosion Tests
Subtask 4: Environmentally Assisted Corrosion Tests
Subtask 7: Dry Oxidation Tests by Thermogravimetric analyses
Subtask 8: Microstructure and Phase Characterization Studies

In addition we are working on a manuscript on stress corrosion cracking of steels for underground support. A manuscript “Paper No. MS30802E” has been sent to Corrosion Journal (NACE) which is accepted and is in final print.

1. Introduction
This report encompasses the work done for third quarter in accordance to cooperative agreement of UCCSN for the Task 019 “Subsurface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”, the objective of which was proposed earlier, to conduct corrosion research and predict the durability of rock-bolts and other underground metallic roof supports. We have recently started working on oxidation tests using Thermogravimetric Analyzer (TGA), stress corrosion cracking/hydrogen embrittlement studies on rock bolts, other support materials including bench mark materials.

In this quarterly report we will discuss “Dry” oxidation of steels and other supporting materials that are susceptible to oxidation under the repository conditions. William M.C. rock bolt and Split Sets rock bolt (HSLA steel made by “International Rollforms”) were tested by ramping from 50°C to 500°C with 1°C/min scan rate under O₂ atmosphere by using a Thermogravimetric analysis (TGA). In addition, isothermal oxidation test of Alloy 22 in Pure O₂ atmosphere @ 980°C using TGA was performed. The oxide samples were also examined by SEM.

2. Experimental – Thermogravimetric Analyses

2A. Thermal Analyses

A TA Instruments, Model Q500, Thermogravimetric analyzer has been used for oxidation research. This instrument can be operated in two modes for investigating thermal stability behavior in controlled atmosphere: (1) dynamic, in which the temperature is increased at a linear rate, and (2) isothermal, in which the temperature is kept constant. This instrument
has a continuous weighing capacity of 1.0 g and a sensitivity of 0.1 µg, and a heating rate of 0.1 to 50 °C/min. This TGA was used to conduct the oxidation experiments. (Please see Technical Progress report No. 2 (2005) for details of TGA experiments.

2B. Materials

The materials obtained from the manufacturers, such as William’s Rock bolt, IRF Split sets, and Alloy 22 specimens, are shown Figures 2B.1-2B.3.

Figure 2B.1. William M.C. Rock Bolt

Figure 2B.2. “International Rollforms” - Split Sets Rock bolt - HSLA Steel

Figure 2B.3. Ni-Base Alloy 22
3. Results and Discussion

In this quarter we present Thermogravimetric* and Stress Corrosion Cracking, electrochemical studies (of Alloy 22) results on the following materials:

1. **Rock Bolts**: *William Type rock bolts* (medium carbon steel) and *International Roll Forms (IRF) - Friction type Split Sets* (HSLA steel)
2. **Baseline Materials**: *Alloy 22* (Ni based superalloys) TGA and electro-mechanical studies.

### 3.1 Rock Bolts - Dry Oxidation Ramping Tests

Thermogravimetric tests were performed on *Williams and IRF rock bolts*, using a TA Instruments - TGA Q500, with a heating rate of 1°C/min from 50°C to 500°C, under pure O₂ atmosphere. This study showed there was weight gain during heating for both rock bolts. The weight gain was continuously recorded as a function of temperature and is shown in Figure 3.1.1. The composition of the William M.C. rock bolt is listed in Table 3.1, the IRF split set compositional analyses are in progress.

**Table 3.1. The composition of the William M.C. rock bolt.**

<table>
<thead>
<tr>
<th>Elements</th>
<th>C</th>
<th>Mn</th>
<th>S</th>
<th>Al</th>
<th>Cr</th>
<th>Cu</th>
<th>Mo</th>
<th>N</th>
<th>Ni</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Composition</td>
<td>0.44</td>
<td>1.57</td>
<td>0.31</td>
<td>0.004</td>
<td>0.08</td>
<td>0.19</td>
<td>0.03</td>
<td>0.009</td>
<td>0.06</td>
<td>0.27</td>
<td>Bal</td>
</tr>
</tbody>
</table>

The William’s M.C. rock bolt sample is of a cylindrical shape with a diameter of ~3 mm and ~2.5 mm in height, with a initial weight of 126.076 mg. After the TGA experiments there is a weight gain of 0.0861 wt%. Characterization of these rock bolt surfaces were performed using scanning electron microscopy (SEM) and EDS microanalyses. The oxide layers formed on the Williams rock bolt specimen are shown in the SEM micrograph (Figure 3.1.2). The EDAX spectrum on Williams rock bolt showed major peaks of Fe-Kα, Fe-Kβ, and O- Kα; as expected.

The Split Sets (HSLA) TGA specimen (a cube of ~3x3x3 mm), with an initial weight of 159.297 mg. showed a mass gain of 0.06606 wt.% with temperature ramping from 50 to 500°C (Figure 3.1.1). The SEM characterization showed a slightly different morphology, as compared to the Williams medium carbon steel rock bolts (Figure 3.1.2). The EDAX spectrum on Split set rock bolt showed also major peaks of Fe-Kα, Fe-Kβ, and O- Kα (Figure 3.1.3).

![Figure 3.1.1. TGA ramping curves of William M.C. rock bolt and Split Sets rock bolt at a scan rate 1 °C/min from 50°C to 500°C show a decrease of 0.066-0.086 range.](image-url)
3.2 Baseline Material Alloy 22 - Dry Oxidation Isothermal Tests

Isothermal Thermogravimetric analyses of Alloy 22 is shown in Figure 3.2.1. Three different Alloy 22 samples were run on the TGA with different initial masses. The percent weight change vs. temperature curves for these three samples are shown in Figure 3.2.1. These have been corrected for buoyancy effects resulting from sample pan
position changes mainly due to flow of oxygen gas. Blank sample pans were run under exactly same conditions and then subtracted from the curves obtained from the samples. These corrections are to the complete scan ranges.

There are three distinct regions in these thermograms. In the first region, a sharp weight gain is observed due to temperature ramping from room temperature to 980°C. The isothermal (980°C) regions 2 and 3 are of interest for oxidation studies; the holding time is approximately 1380 minutes. We observe a weight loss in region 2, which is attributed to the formation of grain boundary precipitates, such as μ, σ, and P, topological closed packed (TCP) phases. It is suggested that there is a simultaneous formation of an oxide layer; which could possibly be NiO. The grain boundary precipitation is more predominant than the formation of oxides in this region 2. The Alloy 22 has a FCC structure with a lattice parameter of 0.361 nm with a unit cell volume of 0.047 nm³. The exact nature of precipitates, such as μ, σ, and P is not known at this time. However, previous detailed TEM studies on thermal aging of Alloy 22 by Gorhe [Ref. D. D. Gorhe, MS Thesis, UNR August 2004] at UNR showed formation of these TCP phase under slight different conditions. The unit cell volumes of the TCP phases form previous studies, and oxides from this study are shown in Table 3.2.1. It can be observed that the unit cell volumes of the TCP phases are much higher than that of the closed packed parent Alloy 22. Higher volumes which are indicative of lower densities cause the observed decreases in the mass.

Figure 3.2.1. Isothermal Oxidation of Alloy 22 in Pure O₂ atmosphere @ 980°C Using TGA.

A TTT diagram for Alloy 22, taken from Gorhe’s thesis shown in Figure 3.2.2, indicates completion of precipitation after approximately 3 hours at 980°C. Based on this, it concluded that the TCP phases stop forming after three hours.
Table 3.2.1. Structure And Lattice Parameters of Alloy 22 (\(\gamma\)-Phase), Grain Boundary Precipitates and oxides formed during Heating  [Ref. D. D. Gorhe, MS Thesis, UNR August 2004.]

<table>
<thead>
<tr>
<th></th>
<th>Phase</th>
<th>Bravais Lattice</th>
<th>Cell Parameters (nm)</th>
<th>Volume (nm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>a (nm)</td>
<td>b (nm)</td>
</tr>
<tr>
<td>Alloy</td>
<td>(\gamma)</td>
<td>Cubic (FCC)</td>
<td>0.361</td>
<td>0.361</td>
</tr>
<tr>
<td>Grain Boundary</td>
<td>(\mu)</td>
<td>Hexagonal</td>
<td>0.476</td>
<td>0.476</td>
</tr>
<tr>
<td>Precipitates</td>
<td>(\sigma)</td>
<td>Tetragonal</td>
<td>0.908</td>
<td>0.908</td>
</tr>
<tr>
<td>P</td>
<td>Orthorhombic</td>
<td></td>
<td>0.907</td>
<td>1.698</td>
</tr>
<tr>
<td>Oxides Formed</td>
<td>NiCrO(_3)</td>
<td>Rhombohedral</td>
<td>0.496</td>
<td>0.496</td>
</tr>
<tr>
<td>Cr(_2)O(_3)</td>
<td>Rhombohedral</td>
<td></td>
<td>0.496</td>
<td>0.496</td>
</tr>
</tbody>
</table>

Figure 3.2.2. A TTT Diagram of Grain Boundary Precipitates Formed during Heating For Alloy 22  [Ref. D. D. Gorhe, MS Thesis, UNR August 2004]

During the third stage (region 3), the grain boundary precipitate formation is completed and oxide formation takes place. From table 3.2.1, it can seen that the unit cell volume are still much larger than the Alloy 22. The oxide formed have a higher density as compared to the as-received Alloy22, and thus there is weight gain. Details of the oxide films formed are discussed in the next section.

3.3. X-ray Diffraction and SEM Characterization of Oxide Layer of Alloy 22

X-ray diffraction patterns of Alloy 22, before and after TGA oxygen treatment are shown in Figure 3.3.1. The pattern of Alloy 22 sample, before TGA oxygen treatment, matched the cubic structure, space group Fm3m, [JCPDS Card No. 35-1489] referred to as the \(\gamma\) phase in the XRD pattern of Alloy 22. The XRD pattern for the oxidized samples after TGA O\(_2\) treatment show presence of \(\gamma\) phase and the oxides formed on the surface, identified as Cr\(_2\)O\(_3\) (\(\alpha\) phase) and NiCrO\(_3\) (\(\beta\) phase). Both \(\alpha\) and \(\beta\) phases are rhombohedral structure (space group R-3c) and the JCPDS card numbers are 38-1479 and 22-0748, respectively. The Bragg peaks of the as received \(\gamma\) phase showed...
much broader Bragg peak than after heat treatment. We anticipated this as the as received Alloy 22 is in wrought condition.

Microscopic evaluation of the oxide film by SEM confirmed presence of two phases, which are morphologically different as shown in Figure 3.3.2. These films are formed by holding the temperature at 980°C for 1380 minutes. The EDAX results show the major amount of Cr and O, and a depletion in Ni-content. This result confirms the Cr₂O₃ phase in the XRD result. EDAX on the surrounding oxide layer of Alloy 22 (Figure 3.3.3) show a decreased amount of Cr, and an increased Ni-content as compared to the EDAX results in the crystallite. This suggests the NiCrO₃ phase formed in this oxide layer.

Figure 3.3.1 X-ray Diffraction Patterns of the Alloy 22 (a) before and (b) after Oxidation experiments in TGA. The γ phase is Alloy 22, α phase is Cr₂O₃, β phase is NiCrO₃.

Figure 3.3.2. SEM Micrographics of the oxide layer of Alloy 22 sample after TGA Isothermal at 980°C for 1400 minutes.
Figure 3.3.3. (a) EDAX spectrum for a crystallite in the oxide layer of Alloy 22 show the major amount of Cr and O, and a depletion in Ni-content. (b) EDAX spectrum on the surrounding oxide layer of Alloy 22 shows a decreased amount of Cr, and an increased Ni-content compared to (a).

To summarize the Thermogravimetric studies on carbon and Low Alloy Steels Rock Bolts (Non Isothermal studies) show comparable weight gain in Williams (MCS) and Split Set (HSLA) rock bolts, ranging between 0.06 to 0.08 wt.% in the temperature range of 50 to 500°C. Both curves show increased slopes toward the end (after 450°C). Further studies beyond 500°C may be interesting. Base line for Alloy 22 (Isothermal studies) showed an initial weight loss due to GB precipitate formation - higher unit cell volumes and lower densities. When GB precipitate formation is complete then oxide formation becomes predominant. Cr₂O₃ and NiCrO₃ form (From XRD/SEM results). Weight gain is due to increase in density of oxides relative to the parent alloy.

3.3. Electrochemical and Stress Corrosion Studies on Baseline material (Alloy 22)

3.3.1 Experimental Procedure for electrochemical and stress corrosion studies

Experimental procedures are listed below in the following sections.

3.3.1.1 Electrolyte Preparation

The test electrolyte solution (YM water) of chemical composition, given in Table 3.3.1, was prepared by mixing the appropriate formulated chemicals with distilled and de-ionized (DD) water, which were initially kept at 35 °C. Concentrated YM waters 100X was prepared using overall increased ionic species 100 times. First the chlorides and sulfates were mixed with DD water, and then the bicarbonates and silicates were added in appropriate amounts. A five-liter Erlenmeyer was used to prepare a three liter solution for each batch. Insoluble precipitates were accumulated in the bottom of the solution flask within two hours after the preparation. The solutions were taken out by siphoning over the precipitates and were filtered by No.52 (Whatman Qualitative) filter paper to remove insoluble solutes obtaining a clear colorless water solution.
Table 3.3.1. Yucca Mountain Water chemical composition; pH:7.5

<table>
<thead>
<tr>
<th>Ions</th>
<th>Na⁺</th>
<th>SiO₂</th>
<th>Ca²⁺</th>
<th>K⁺</th>
<th>Mg²⁺</th>
<th>HCO₃⁻</th>
<th>Cl⁻</th>
<th>SO₄²⁻</th>
<th>F⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg/l</td>
<td>61.3</td>
<td>70.5</td>
<td>101</td>
<td>8.0</td>
<td>17.0</td>
<td>200</td>
<td>117</td>
<td>116</td>
<td>0.86</td>
</tr>
</tbody>
</table>

3.3.1.2 Sample Preparation for Electrochemical Tests

Mill annealed (MA) Alloy 22 (Heat 2277-3-3119) plate was used for this study. Figure 3.3.1 shows plates of Alloy 22, before it was finally machined to desired dimensions for both the electrochemical tests and SCC tests. The chemical composition of the alloy is given in Table 3.3.2. The mechanical properties of the Alloy are given in Table 3.3.3

![Figure 3.3.1. Alloy 22 before machined to desired dimensions](image)

Table 3.3.2: Chemical composition of alloy 22 [Haynes International]

<table>
<thead>
<tr>
<th>Elements</th>
<th>C</th>
<th>Co</th>
<th>Cr</th>
<th>Fe</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>V</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Composition</td>
<td>0.005</td>
<td>1.05</td>
<td>21.38</td>
<td>3.88</td>
<td>0.24</td>
<td>13.55</td>
<td>Bal</td>
<td>0.006</td>
<td>0.0057</td>
<td>0.028</td>
<td>0.14</td>
<td>3.07</td>
</tr>
</tbody>
</table>

Table 3.3.3. Mechanical Properties of Alloy 22 [Haynes International]

<table>
<thead>
<tr>
<th>Heat</th>
<th>Tensile Strength (MPa)</th>
<th>Yield Stress (MPa)</th>
<th>Elongation (%)</th>
<th>Hardness (RB)</th>
<th>ASTM Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>2277-3-3119</td>
<td>752</td>
<td>321</td>
<td>64</td>
<td>86</td>
<td>3</td>
</tr>
</tbody>
</table>

Cylindrical specimens of 1.27 cm² cross section were machined out of the Alloy 22 plates. Precautions were taken to make sure that heat generation was minimum by using cooling systems incorporated within the machines. Specimens were first soldered to a thick insulated copper wire for electrical connection.

![Figure 3.3.2. Epoxy mounted specimens for linear polarization tests and impedance Spectroscopy, and their electrical connection wires](image)

Then a metallographic type epoxy mount was made with 600 grit finish. The specimen surfaces were washed with water, and degreased with acetone, and rinsed with alcohol and distilled-deionized water. Finally, they were immediately immersed in to the preconditioned, de-aerated electrolyte of YM water (100x) test solution.
3.3.1.4 Sample Preparation for SCC Tests

Cylindrical type tensile test specimens were machined out of alloy 22 within 5/1000 inch tolerance in the diameter and length of the effective gauge (ASTM E-08). The cylindrical tensile specimens and their dimensions are given in Figure 3.3.3. The length of specimen was designed such that it would fit in the environmental cell while the threaded ends were still exposed out of the test cell and could be connected to the universal joints. Figure 3.3.4 shows the actual sample made out of Alloy 22 plate.

![Dimensions of cylindrical environmental test specimen with threaded ends for firm grip to the universal joints of SSRT machine.](image)

The effective gauge length was machined to 22mm, with 4.05mm inch corresponding reduced diameter. Teflon (PTFE, polytetrafluoroethylene) tape wrapped around the specimen only exposed the effective gauge for the current and potential scanning in the test solution. The gauge was manually ground with successive finer SiC emery papers until the 600 grit finish. The specimen surfaces were degreased with acetone, and washed with water, and rinsed with alcohol and distilled-deionized water. Then they were immediately installed in to the test cell and exposed to the test solution, which had already been conditioned by nitrogen gas for at least one hour.

3.3.1.5 Cell Design and Experimental Set-Up for Electrochemical Tests

A one-liter Pyrex brand glass flask is used to house the specimens and other related standard cells for electrochemical experiments as shown in Figure 3.3.5. A disk shape lid constructed out of 2 cm thick Teflon to seal the cell by means of o-ring, was held in place by compression clamps. A tapered Teflon plug with two holes was machined for the middle opening of the lid, in which cylindrical receptacles for specimen and a Luggin probe were placed as shown in the figure. Several other receptacles on the lid sealed the entire cell elements via threaded o-ring fittings. The tapered Teflon stopper constructed for the larger central hole of the lid held both the specimen and Luggin probe in close proximity, as shown in the figure. The distance between the probe tip and the specimen surface was maintained constant using a small Teflon block spacer in between them. It did not block the interface since only the edge of the epoxy mold leaned against the spacer set on the probe. The probe and specimen holder set-up was designed to provide a desired distance of 1 mm in all cases, to eliminate any probe distance effect on the entire potentiodynamic polarization or impedance experiments.
Cylindrically shaped working electrodes (test specimens) were mounted in epoxy with their 1 cm² bottom surface area and electrical connection wire exposed out of it, as shown in Figure 3.3.2. A large (~10 cm²) platinum sheet sealed to a glass capillary was used as a counter electrode to provide good conductivity in the electrolyte. The Luggin probe tip and Ag/AgCl reference electrode were connected via the test solution (YM water) in the probe, without using a salt bridge. Continuously purged gas (nitrogen or oxygen) in the sealed cell maintained constant pressure above the solution, and formed a tall column (8-10cm) of solution bridge inside the probe, which connected the Luggin tip and the reference electrode. Therefore, the reference electrode was able to be placed well above the solution level, outside the cell. Thus, the set-up avoided excessive heat on the reference electrode, by means of the temperature gradient created along the solution bridge in the probe.

3.3.1.6 Cell Design and Experimental Set-Up for SSC Tests

The environmental cell shown in Figure 3.3.6 was designed and glass-blown in 1 liter capacity. It consisted of seven necks and joints at the top, and one joint at the bottom. The joints and necks around the central neck on top of the cell were designed such that the cell elements were easily inserted into the electrolyte. In some cases, the sealing was done only by “stop joints necks” when larger openings were needed. A water cooled reflux condenser fitted into one of the large stop joints minimized the solution loss due to evaporation during high temperature runs. A quartz electric immersion heater (LHP 100, Glo-Quartz Elect. Co), specifically designed for corrosion experiments, was used to heat the electrolytes. A fritted glass end capillary was used for purging gas in to the test solution. De-aeration of the test solution was maintained by continuously purged nitrogen gas at a rate of 150ml/min. A glass blown Luggin probe made from a Pasteur pipette was inserted into the cell via an o-ring fitting, in which the reference electrode (Ag/AgCl) was hold in a fixed position. The tip of the Luggin probe was maintained at a fixed distance from the specimen surface (2 mm). A sealed-end Pasteur pipette thermo-well enclosed thermocouple was inserted into the test solution close to the test specimen. A platinum sheet sealed onto a glass capillary was used as a counter (auxiliary) electrode to supply good current distribution around the specimen in the cell. A PID temperature controller and a quartz immersion electric heater maintained the desired temperatures within an accuracy of ±0.1 °C.

A screw-drive-mechanism universal testing machine (United Calibration Corp., Model STM-10E-S) exclusively designed for slow strain rate testing (SSRT), with 4kN load cell mounted under the moving cross head over the test cell, was used for the entire electromechanical experiments, as shown in Figure 3.3.7. A Pentium based custom made computer (Southern California Computers, Model PAC-1200/PCI-10S) including data processing software and hardware (United Calibration Corp., Datum for Windows, Version III ) was used for stress-strain curve scanning. The universal machine shown in Figure 3.3.7 enabled any desired strain rate between 10⁻⁸/s to 10⁻²/s. A
Gamry model potentiostat (Gamry Instruments, PC4(750) /DC105 II) was used for applying and monitoring potential and current over tensile test specimens in the test cell.

![Gamry model potentiostat](image)

Figure 3.3.6(left) Environmental cell for SCC tests  
Figure 3.3.7(right) Universal Testing Machine Showing the Experimental Setup

### 3.3.2 Results and Discussion for Electrochemical and Stress Corrosion Tests

#### 3.3.2.1 Polarization Curve for Alloy 22

Potentiodynamic polarization curves were obtained for the Alloy 22 at two different temperatures. The specimens were held in simulated 100x YM waters for two hours to reach a stabilized $E_{corr}$ before conducting the experiment. Corrosion films do not form during the two hour period. Corrosion rates of Alloy 22 have been determined for de-aerated 100x YM concentrated-simulated YM waters at the temperatures 25 °C and 85 °C by employing linear polarization methods. General corrosion rates for the de-aerated waters were determined to be between 4.7 μm/year and 5.39 μm/year for 22°C to 85°C. The polarization curves for 100X de-aerated YM water given in Figure 3.3.8.

![Polarization Curve for Alloy 22](image)

Figure 3.3.8. Polarization curve for alloy 22 in 100X YM water
3.3.2.2 Stress Corrosion Cracking Tests

Two tests were conducted in air at room temperature as reference tests. The strain rates were maintained at $1.6 \times 10^{-6}/s$ for all experiments. Table 3.3.4 shows the testing conditions such as temperature, applied potential as well as the test results. Figure 3.3.9 shows the test results of the two specimens at room temperature in air. The results of the two tests are superimposed with the result obtained by Dr. Rebak et al [2] for comparison. Figure 3.3.10 shows the stress-elongation curves for Alloy 22 specimens strained at 400 mV in 100X simulated YM water at room temperature. The UTS value and the time to failure is almost same for the tests conducted in air and 100x YM water. A photograph of the fractured Alloy 22 is shown in Figure 3.3.11.

Table 3.3.4. Tests condition and Results for Alloy 22

<table>
<thead>
<tr>
<th>Sample</th>
<th>Test Environment</th>
<th>Temp. (°C)</th>
<th>Applied Potential</th>
<th>Time to Failure (hrs)</th>
<th>UTS (ksi)</th>
<th>RA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rebak</td>
<td>Air</td>
<td>22</td>
<td>NA</td>
<td>123</td>
<td>786</td>
<td>70</td>
</tr>
<tr>
<td>1</td>
<td>Air</td>
<td>22</td>
<td>NA</td>
<td>96</td>
<td>815</td>
<td>63</td>
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<tr>
<td>2</td>
<td>Air</td>
<td>22</td>
<td>NA</td>
<td>92</td>
<td>791</td>
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</tr>
<tr>
<td>3</td>
<td>100X YM</td>
<td>22</td>
<td>400 mV</td>
<td>89</td>
<td>769</td>
<td>66</td>
</tr>
</tbody>
</table>

Figure 3.3.9 Stress-Elongation curve for alloy 22 in air at room temperature [Kenneth J. King, John C. Estill, Raul B. Rebak, Transportation, Storage and Disposal of Radioactive Materials – 2002, Vol 449].
To summarize (1) general corrosion rates for the de-aerated waters were determined to be between 4.7 µm/year and 5.39 µm/year for 25°C to 85°C. (2) Stress corrosion cracking of Alloy 22 under the YM environment shows that is susceptible to stress corrosion cracking at room temperature and 400 mV. These are just initial studies, and detailed studies are in progress to study the effect of applied potential, temperature and concentration of electrolyte on the susceptibility to stress corrosion cracking.

**Appendix for Stress Corrosion Studies**

The universal testing machine at UNR has pull rods designed to accommodate the SCC test cell glassware. The pull rods are made out of carbon steel which have lower strengths than the Alloy 22. The results obtained show a convoluted effect of the pull rods and the Alloy 22. We compared the data of the manufacturer of the Alloy 22...
(Haynes), as well as Dr. Raul Rebak (LLNL) with our results in air, and found differences in the UTS, and failure strengths. As the extensometer could not be used during the stress corrosion tests, a correlation of stress vs. %elongation was obtained by using Dr. Rebak’s data and to obtain calibration of the machine.

Original Data obtained from SSRT Test

![Graph showing stress vs. % elongation with data points for Sazzad-Org #1, Rebak-Org, and Sazzad-Org #2.]

Plot for Correcting Stress

![Graph showing a linear fit equation: y = -1E-06x^3 + 0.0004x^2 + 1.3557x, with R^2 = 0.9995.]

Plot of SSRT Tests after modifying Stress

![Graph showing modified stress data for Rebak-Org, Sazzad-Mod-Stress #1, and Sazzad-Mod-Stress #2.]
**Plot for Correcting Elongation**

\[ y = -0.0042x^2 + 1.1096x \]

\[ R^2 = 0.9908 \]

**Plot of SSRT after modifying Stress and Elongation**

*Timeline*

For Task ORD-FY04-019 we have started the oxidation tests using TGA, Potentiodynamic tests, stress corrosion studies, x-ray diffraction analyses and scanning electron microscopy as per timeline. The EIS scans could not be performed as schedule the availability of Potentiostats. The immersion experiments have just started, as the specimens were prepared for Swellex rock bolts in expanded state under water pressure at the Company. These delays due to potentiostat availability are not very significant, and we will be able to catch up with the tasks during the next quarter.