4-6-2007

Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain Repository — Quarterly technical report No. 11

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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. 11 (submitted during 2nd Quarter of FY 2007)

Start Date of this Quarter, January, 1, 2006 to March 31, 2007

Task No: ORD-FY04-019 and Document No: SIP-UNR-040

Prepared for U.S. DOE/UCCSN Cooperative Agreement Number DE-FC28-04RW12232

-submitted to-

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April 6, 2007
I. 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 quarter, we have performed tests on Mn-24 rock bolts, and 4340 steel for Bernold shields or rock bolts. The importance of these results is that we have used the Yucca Mountain water chemistry (furnished by the DOE) as an electrolyte for the corrosion experiments on rock bolts and other materials in which radioactive waste is stored. During this period, we used potentiodynamic and EIS tests to obtain corrosion rates. We have also performed oxidation and immersion tests of steels as per SIP, but these analyses are not complete and will be reported in quarterly report.

General Statements

Commercial rock bolts are generally made out of High-strength low-alloy (HSLA) steels. They are generally designed for high strength, but perhaps not so much emphasis is made on the corrosion aspects. We have performed corrosion tests on commercial rock bolts Mn-24 Swellex rock bolts, 4340 steel that may be potentially used for rock bolts of different types, and related materials for ground support of the repository. In this report No.11 the corrosion rates have been determined in deaerated (nitrogenated) conditions at different temperatures in simulated 1X, 10X, 100X YM waters for 4340 steel. Some of the experiments were repeated to check reliability etc. Characterization of reacted products by optical microscopy has also been performed during this quarter on other materials. Analyses and more experiments are in progress. Josh Lamb et al..paper on permeation in steel is in print in Met. Trans 2007.

Progress for the Period 1/1/07 to 3/31/07 on rock bolts

Subtask 1: Selection of New High-Strength Low-Alloy Steels, Stainless Steels for Rock Bolts, Steel Sets and Perforated Roof supports. In this report we show 4340 Steel results.

Subtask 2: Electrochemical tests to evaluate corrosion rate and possible corrosion mechanisms on Mn-24 Swellex rock bolts, and 4340 steel.

Subtask 3. Electrochemical Impedance Spectroscopy (EIS) of 4340 Steel

Subtask 6: Thermogravimetric (TGA) recalibration for reliability test at different temperatures (experiments performed but not complete, will be reported later)

Subtask 7: Microstructure Characterization Studies

II. Introduction

This report shows the work done for this period in accordance to cooperative agreement of University of Nevada system for the Task 019 “Subsurface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”, the overall objective of which is to conduct corrosion research and predict the durability of rock-bolts and other underground metallic roof supports. We are performing oxidation tests using Thermogravimetric Analyzer (TGA), and potentiodynamic and immersion tests to determine the corrosion rates of rock bolts.

In this quarter:

- Potentiodynamic tests were performed to determine corrosion rates (CR) of 4340 Steel in 1X, 10X (repeated ) 100X YM waters at several temperatures in deaerated condition.
- EIS studies were performed on the 4340 steel at 10X and 100x YM electrolyte.
- The TGA had to be recalibrated and the anomalies of the modulated data for oxidation studies were performed for determination of accuracy of the data.
- One Met Trans A. Paper is in print 2007 (this was submitted in 2006), One Paper presented at NACE Conference in Florida 2007.

III. RESULTS AND DISCUSSION

3.1 Corrosion Studies on AISI-SAE 4340 Steel in Simulated YM Groundwater
3.1.1 Introduction

Rock bolts and I-beams within the Yucca Mountain Nuclear Waste Repository are important substructure stabilization effort. Low alloys steels have been used in many rock bolts for high strength. In this particular study, potentiodynamic polarization tests and electrochemical impedance spectroscopy (EIS) tests were carried out on AISI 4340 steel in simulated Yucca Mountain (YM) groundwater at one times (1X), ten times (10X) and one hundred times (100X) the normal concentration in both aerated (oxygenated) and deaerated (nitrogenated) conditions. These tests are now complete, except for mechanisms that are currently being planned.

3.1.2 Experimental

Electrochemical Tests

The electrochemical tests were performed using (Quality assured) commercially-available 4340 steel. The chemical composition was analyzed by LTI1 and is given in the (last) 10th quarterly report. Disc-shaped test specimens approximately 0.25” were professionally cut and machined locally. The test specimen were mounted in epoxy with ~1.2 cm² surface area. An electrical connection wire was spot welded to the back of the steel specimen. Before the experiment, the sample was polished with 240 grit and 600 grit SiC emery papers and washed with deionized/distilled water before inserting them into the cell for electrochemical testing.

All the electrochemical experiments were conducted in a typical 1 liter Pyrex glass flask covered with a polytetrafluoroethylene lid (please see Quarterly report No. 5 for the photos and description of the apparatus). The reference electrode was a saturated silver/silver chloride (Ag/AgCl) electrode, which has a potential of 199mV more positive than the standard hydrogen potential. Continuously purged gas (nitrogen or oxygen) in the sealed cell maintained constant pressure above the solution. A fritted glass capillary was used for continuous aeration/deaeration of the solution throughout the experiment at the rate of ~ 20 standard cubic centimeters per minute (scm) by using a flow meter. An electrically-heated water bath surrounded the test cell and maintained the temperature of the solution. The specimen was inserted into the electrolyte-filled cell and oxygen/nitrogen was continuously purged for minimum of 60 minutes, or until the steady-state open circuit potential was reached. Electrochemical impedance spectroscopy (EIS) tests were carried out between 10 kHz and 10 mHz at an ac voltage of 100 mV rms. The potentiodynamic tests were carried out at a scan rate of 0.2mV/sec. Both tests were conducted using a commercially available potentiostat.

The electrolyte was prepared by mixing different kinds of salts in distilled, de-ionized water at ~ 40°C. After adding the salts to the de-ionized water, the solution was stirred on a hot plate for 30 minutes without heat, and then allowed to cool down for approximately one hour. Upon cooling, the excess or undissolved salts precipitated out from the solution at the bottom of the flask. The solution was filtered – once for 1X and twice for 10X & 100X – before using the solution as the electrolyte. Also, the pH of the solution was measured before and after each experimental run.²

The corrosion rate was calculated by following ASTM G59-97³ and ASTM G102-89⁴ procedures. During calculations, the polarization resistance ($R_p$) of the solution was neglected because it was shown by Yilmaz⁵ that the electrolyte concentration was negligible. Tafel constants for both anodic reaction and cathodic reactions are assumed to be constant (0.12V/decade).⁶ From the polarization resistance ($R_p$) value, the corrosion current density was calculated using equation 3.1, where $K_i = 3.27 \times 10^{-3}$ mm·g/µA·cm·year, $I_{corr} = \mu A/cm^2$, $\rho = 7.87$ g/cm³ and $EW = 27.95$ gm/equivalent.⁴

$$\frac{CR}{K_i} = \frac{I_{corr}}{\rho} \cdot EW$$

(3.1)

3.1.3 Results & Discussion

In this section we present the results of the potentiodynamic and impedance spectroscopy studies of 4340 steel in 1X, 10X & 100X Yucca Mountain solutions.
Corrosion Studies in Deaerated (Nitrogenated) YM Water Electrolytes

Potentiodynamic and corrosion rate graphs as a function of temperature, are shown in Figures 3.1.1 – 3.1.8. As can be seen in Figures 3.1.1 – 3.1.3, the $E_{corr}$ hovers around or slightly below -700 mV for 25°C, 45°C, 65°C and 85°C. The potentiodynamic scans for 75°C and 90°C were left out to make the graphs less congested. However, these scans show similar behavior relative to 85°C.

Figures 3.1.4 – 3.1.6 show representative potentiodynamic graphs for 25°C, 65°C and 85°C in 1X, 10X & 100X solution concentrations, along with optical micrographs (showing pitting) at 10X & 100X concentrations. It can be noted that the passivity range increases as the concentration is increased, which makes the pitting potential, $E_{pits}$, more positive. Also, for lower temperatures the passivity range increases and shifts diagonally upward, as in Figure 3.1.2 at 25°C. However, for higher temperatures, it shifts diagonally downward, as shown at 65°C and 85°C, where it virtually disappears. This trend can be easily shown in Figure 3.1.2 at 10X concentration. As can be seen, the passive layers decrease in size from 25°C to 65°C; by 85°C, the passive layer has nearly disappeared. This indicates that for lower temperatures, as the concentration increases the passivity become more noble (more positive) while at the same time, the current density increases. This is because there are more ions in the electrolytes as the concentration increases. Also, it shows that the passive films are more adherent to the steel at these lower temperatures. As the temperature increases, the “passivity region” decreases as the current density increases. Perhaps at higher temperatures the passive films that forms on the surface starts to break down. Additionally, larger pits are formed in 100X solution, relative to 10X solution, such as in Figures 3.1.4 and 3.1.6. Figure 3.1.7 shows the average corrosion rates as a function of temperature for all three solution concentrations. In the deaerated condition, corrosion rates increase with temperature using the 1X and 10X electrolyte. Whereas, using 100X electrolyte, we observe a maximum corrosion rate (CR) of 308 microns/yr. at 45°C. Then the CR decreases steadily from 45°C to 75°C; beyond this there is not much change. Figure 3.1.8 shows a comparison of the average corrosion rates for both 4340 steel and 1144 steel (earlier designated as 1040 steel), respectively. For 100X concentration, the activation energy for 4340 steel is 17.34 kJ/mol, compared to 18 kJ/mol for 1144 steel.

![Figure 3.1.1](image_url)  
Figure 3.1.1. Representative potentiodynamic plots of AISI 4340 steel at normal (1X) Yucca Mountain solution concentration in a deaerated (nitrogenated) environment.
Figure 3.1.2. Representative potentiodynamic plots of AISI 4340 steel at ten-times (10X) the Yucca Mountain solution concentration in a deaerated (nitrogenated) environment.

Figure 3.1.3. Representative potentiodynamic plots of AISI 4340 steel at one hundred-times (100X) the Yucca Mountain solution concentration in a deaerated (nitrogenated) environment.

Figure 3.1.4. Representative potentiodynamic plots of AISI 4340 steel at different Yucca Mountain solution concentrations in a deaerated environment at 25°C.
Figure 3.1.5. Representative potentiodynamic plots of AISI 4340 steel at different Yucca Mountain solution concentrations in a deaerated environment at 45°C.

Figure 3.1.6. Representative potentiodynamic plots of AISI 4340 steel at different Yucca Mountain solution concentrations in a deaerated environment at 65°C.

Figure 3.1.7. Average corrosion rates as a function of temperature and solution concentration for AISI 4340 steel under deaerated (nitrogenated) conditions.
Figure 3.1.8. Comparisons of average corrosion rates as a function of temperature and solution concentration for AISI 4340 and 1144 steels under deaerated (nitrogenated) conditions.

Representative Nyquist & Bode plots are shown in Figures 3.1.9 – 3.1.14. As can be seen from these scans, the general behavior is consistent throughout. The solution resistance decreases to zero at a concentration of 100X, owing to the fact that there is a large amount of electrolytes in the solution. At lower temperatures, the Nyquist curves show a slight Warburg tail, with a more pronounced tail at higher temperatures. This indicates that the electrochemical reaction is nearly all diffusion-controlled. This is consistent with behavior in 1X YM water. The corresponding Bode plots exhibit a single phase angle maxima at nearly all temperatures, but between 65°C and 75°C they begin to exhibit double phase angle maxima. This behavior is unusual, as it is not observed at higher temperatures in the other solution concentrations. This indicates that the passive film formed on the surface at these temperatures is porous, which is consistent with the behavior from the Nyquist plots at these temperatures.

Figure 3.1.9. Nyquist and Bode plots of AISI 4340 steel at 25°C in deaerated, 100X YM water.

Figure 3.1.10. Nyquist and Bode plots of AISI 4340 steel at 45°C in deaerated, 100X YM water.
Figure 3.1.11. Nyquist and Bode plots of AISI 4340 steel at 65°C in deaerated, 100X YM water.

Figure 3.1.12. Nyquist and Bode plots of AISI 4340 steel at 75°C in deaerated, 100X YM water.

Figure 3.1.13. Nyquist and Bode plots of AISI 4340 steel at 85°C in deaerated, 100X YM water.

Figure 3.1.14. Nyquist and Bode plots of AISI 4340 steel at 90°C in deaerated, 100X YM water.
Aerated YM Water Electrolytes

Potentiodynamic and corrosion rate graphs as a function of temperature are shown in Figures 3.1.15 – 3.1.22; the $E_{corr}$ range is between -500 mV and -400 mV for 25°C, 45°C, 65°C and 85°C. These figures show 4340 steel exhibits active dissolution in an aerated environment in different solution concentrations and temperatures and is more pronounced in the 10X and 100X concentrations. At 25°C, the $E_{corr}$ is more positive (noble) as compared to the other temperatures with the same concentration (Fig. 3.1.16). Also, this 25°C scan shows some passivity, whereas these passive regions are less pronounced at 45°C (in 10X) and 25°C in 100X solution. However, there is no indication of any passivity at higher temperatures, as opposed to the AISI 1144 steel which showed a small amount of passivity at 85°C in 100X solution near this same potential.

Figures 3.1.18 and 3.1.19 show representative potentiodynamic graphs for 45°C and 75°C in 1X, 10X & 100X solution concentrations, along with some optical micrographs at 10X & 100X concentrations. In these optical micrographs, as in the deaerated conditions, larger pits are formed in 100X solution, relative to 10X solution, such as in Figures 3.1.18 and 3.1.19. This can be attributed to the difference in electrolytes required for pit formation and continual attack by existing pits. Figure 3.1.18 and 3.1.19 correlates with Figure 3.1.20, in terms of applied current density versus corrosion rate. However, it is interesting to note that for 1X and 10X concentrations, these temperatures the cathodic reactions are nearly or all diffusion-controlled, especially for lower temperatures like 45°C. This is shown in the cathodic regions of the scan by the near-vertical slope (i.e no change in current density) of the cathodic reaction indicating that electrochemical reactions in aerated conditions are more diffusion-controlled at lower temperatures. However, at higher temperatures the electrochemical reactions are more kinetically controlled. This is more pronounced for 45°C and less pronounced for 75°C. Figure 3.1.21 shows a comparison of the averaged corrosion rates as a function of temperature and solution concentration for 4340 and 1144 steels in Yucca Mountain water. Figure 3.1.22 shows a comparison of the averaged corrosion rates as a function of temperature and solution concentration for 4340 steel in oxidizing and reducing conditions in Yucca Mountain water.

The activation energy for 4340 steel is 21.43 kJ/mol, compared to 30.45 kJ/mol for 1144 steel (10X), and 1.87 kJ/mol, compared to 8.15 kJ/mol for 1144 steel (100X). This is to be expected, as 4340 steel is much harder than 1144 steel, due to the extra alloying elements – nickel, chromium and molybdenum – in the steel.

![Figure 3.1.15. Representative potentiodynamic plots of AISI 4340 steel at normal (1X) Yucca Mountain solution concentration in an aerated (oxygenated) environment.](image)
Figure 3.1.16. Representative potentiodynamic plots of AISI 4340 steel at ten-times (10X) the Yucca Mountain solution concentration in an aerated (oxygenated) environment.

Figure 3.1.17. Representative potentiodynamic plots of AISI 4340 steel at one hundred-times (100X) the Yucca Mountain solution concentration in an aerated (oxygenated) environment.

Figure 3.1.18. Representative potentiodynamic plots of AISI 4340 steel at different Yucca Mountain solution concentrations in an aerated environment at 45°C.
Figure 3.1.19. Representative potentiodynamic plots of AISI 4340 steel at different Yucca Mountain solution concentrations in an aerated environment at 75°C.

Figure 3.1.20. Average corrosion rates as a function of temperature and solution concentration for AISI 4340 steel under aerated (oxygenated) conditions.

Figure 3.1.21. Comparisons of average corrosion rates as a function of temperature and solution concentration for AISI 4340 & 1144 steels under aerated (oxygenated) conditions.
Figure 3.1.22. Comparisons of average corrosion rates as a function of temperature and solution concentration for AISI 4340 steel under nitrogenated and oxygenated conditions.

Representative Nyquist & Bode plots are shown in Figures 3.1.23 – 3.1.34. As can be seen from these scans, the general behavior is nearly the same. Both 10X & 100X concentrations show a kinetic/diffusion controlled reaction, as evidenced by the near lack of Warburg tails from both sets of impedance data. Most of the Nyquist plots show a perfect, or near-perfect semicircle, along with single phase maximum curves for the Bode plots. This indicates that the active film on the surface of the sample is not porous.

Figure 3.1.23. Nyquist and Bode plots of AISI 4340 steel at 25°C in aerated, 10X YM water.

Figure 3.1.24. Nyquist and Bode plots of AISI 4340 steel at 45°C in aerated, 10X YM water.
Figure 3.1.25. Nyquist and Bode plots of AISI 4340 steel at 65°C in aerated, 10X YM water.

Figure 3.1.26. Nyquist and Bode plots of AISI 4340 steel at 75°C in aerated, 10X YM water.

Figure 3.1.37. Nyquist and Bode plots of AISI 4340 steel at 85°C in aerated, 10X YM water.

Figure 3.1.28. Nyquist and Bode plots of AISI 4340 steel at 90°C in aerated, 10X YM water.
Figure 3.1.29. Nyquist and Bode plots of AISI 4340 steel at 25°C in aerated, 100X YM water.

Figure 3.1.30. Nyquist and Bode plots of AISI 4340 steel at 45°C in aerated, 100X YM water.

Figure 3.1.31. Nyquist and Bode plots of AISI 4340 steel at 65°C in aerated, 100X YM water.

Figure 3.1.32. Nyquist and Bode plots of AISI 4340 steel at 75°C in aerated, 100X YM water.
Summary of 4340 Steel Corrosion Study
Electrochemical corrosion tests are reported during this quarter for 4340 steel. For these tests, the maximum corrosion rates were 308 microns/yr in deaerated conditions, and 2193 microns/yr in aerated conditions. Summary table showing corrosion rates of 4340 Steel in deaerated and aerated conditions:

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Conc. (X)</th>
<th>Deaerated</th>
<th>Aerated</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>1</td>
<td>29</td>
<td>244</td>
</tr>
<tr>
<td>25</td>
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<td>155</td>
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<td>1078</td>
</tr>
<tr>
<td>75</td>
<td>100</td>
<td>59</td>
<td>871</td>
</tr>
</tbody>
</table>
Comments: We have repeated some of these experiments and this is our final result. Please ignore the previously reported corrosion rates on 4340 steel at these temperatures and compositions.

References


3.2. Corrosion study of Swellex Rock Bolt (“As –Inflated”) using Potentiodynamic/potentiostatic scans

3.2.1. Introduction: Frictional Rock Stabilizers (FRS) such as swellex™, split set and other types of rock bolts are used in underground rock support applications. These types of rock bolts are used to support large underground tunnels which experience a large amount of stress when installed. The installation of “Swellex” rock bolts is rather unique as these rock bolts, in as-received condition, are in collapsed u-shaped form. These are later inflated on site with hydraulic (water) pressure such that there is circumferential stress developed due to plastic deformation. In the previous reports the corrosion rates of Al* rock bolt in YM water concentrations was reported. From the previous results it was observed that there is a passive region in the anodic region of potentiodynamic scan. In this study, with the aid of potentiostatic scan and Scanning Electron Microscopy (SEM) we investigated the passive region of the Al swellex rock bolt at 25°C and 95°C. The 1x YM water chemistry used for this study is shown in the Table 3.2.1.

<table>
<thead>
<tr>
<th>Chemicals</th>
<th>1x YM (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MgSO4.7H2O</td>
<td>50</td>
</tr>
<tr>
<td>CaSO4.2H2O</td>
<td>210</td>
</tr>
<tr>
<td>MgCl2.6H2O</td>
<td>100</td>
</tr>
<tr>
<td>CaCl2.2H2O</td>
<td>196</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>--------</td>
<td>-----</td>
</tr>
<tr>
<td>KHCO₃</td>
<td>50</td>
</tr>
<tr>
<td>NaHCO₃</td>
<td>200</td>
</tr>
<tr>
<td>NaSiO₃·9H₂O</td>
<td>210</td>
</tr>
<tr>
<td>NaF</td>
<td>2</td>
</tr>
</tbody>
</table>

* In our studies we obtained Swellex rock bolts in “as–inflated” condition and is referred to as “AI”

3.2.2. Experimental Procedures for Corrosion Test:

3.2.2.(a) Specimen and Electrolyte Preparation: AI HSLA (swellex) rock bolt was procured from commercial vendor. Small circular discs of 1.88cm diameter were punched out from AI samples without applying any heat, which can alter the properties of material at the edges. These discs were polished around the diameter to eliminate any cold working effects at the edges during punching. Discs of diameter 1.87cm were soldered to thick copper wire for good electrical conductivity and mounted in epoxy resin. The front side of the disc with area of 2.75cm² is exposed for testing. After molding, the electrical connection between the specimen and the wire were checked with a voltmeter. Before the experiment the specimen was ground manually by using SiC emery papers ranging from coarse (200 grit) to fine (600-grit) followed with disc polishing if necessary. The specimen was degreased with acetone and cleaned with de-ionized water before the experiment.

Salts used for this study are given in Table 3.2.1. The electrolyte preparation (1X) for the experiments was prepared by mixing above mentioned salts in 1 liter of distilled, de-ionized water. These solutions were stirred for half-hour at ~60°C to dissolve the salts. After few hours, the solution was vacuum-filtered twice and stored for experimentation. pH for the solution was maintained in between 7 and 8.

3.2.2.(b) Test cell and Procedures: Electrochemical potentiodynamic scans were performed by using ASTM-G59. These tests were conducted in a typical 1 liter Pyrex glass flask covered with a polytetrafluoroethylene lid with many ports, which contains the working electrode, counter electrode (platinum), gas purge, thermocouple, inlet and outlet for the gas, Luggin probe connected to a silver/silver chloride (Ag/AgCl) reference electrode through salt bridge (agar-agar solution) and gas trap. A heating bath was used to control the temperature and thermocouple for monitoring the temperature. This type of setup was adopted from the D.A. Jones textbook, Principles & Prevention of Corrosion².

All potentiodynamic scans were performed by using a commercially available Potentiostat and controlled by commercial electrochemistry software. The electrolyte was conditioned (de-aerated) by the continuous purging of nitrogen gas for one hour before immersing the specimen. The polished and cleaned specimens were introduced into the cell. A distance of 1 mm was maintained between the specimen and Luggin probe to avoid variation in corrosion current (Icorr). When it reached a steady open circuit potential, the potentiodynamic scans were started by sweeping the voltage from -0.8V to -0.2V at a rate of 0.2mV/sec. Same setup and experimental conditions was used for potentiostatic experiments. Potentiostatic scan was run for 2 hours at constant potential for experiments at 25°C and 90°C.

3.2.3 Results and Discussion on Swellex Mn-24 rock bolts

3.2.3.(a) Potentiodynamic scan: Potentiodynamic scan of AI rock bolt in 1x YM water at 25°C from -800mV to -400mV was shown in Figure 3.2.1. This scan covered both cathodic and anodic region. Ecorr (corrosion potential) was observed at round -755mV. Above Ecorr, in the active region the metal (Fe) oxidation was active till around -615mV and then there is passive region from -
615mV to -490mV. After that the passive layer broke down in the trans-passive region. From this kind of behavior in potentiodynamic scan, it is evident that the major contribution of corrosion is in the form of pitting. In the figure 3.2.2 the optical microscope pictures of AI rock bolt before and after the experiments are shown. Form the micrographs after the experiment; it can be observed that there is lot of pits on the surface of the specimen. Also the effect of pits is shown in the figure 3.2.2. Similar kinds of results were obtained at 90°C in 1x YM water.

3.2.3. (b). Potentiostatic scan: To investigate the passive region, potentiostatic scan at -530mV was performed at both 25°C and 90°C. After two hours of scan, it was observed that there is a thick
passive layer formed on the surface of the specimen. Potentiostatic scan at 25°C is shown in the figure 3.2.3. From the potentiostatic scan it can be observed that after few minutes of scan start, the current reached almost to zero and after that it stabilized and maintained constant current flow. The current flow is less than micro amperes, which is negligible. The constant potential (-0.5V) applied is shown in the inset. Similar tendency is observed for the scan at 90°C, but at the end there is little fluctuations observed.

Figure 3.2.3. Potentiostatic scan of Al rock bolt in 1x YM water at 25°C

Figure 3.2.4. EDS profile for Al rock bolt in 1X water at 25°C.
EDS profile for the AI rock bolt is shown in figure 3.2.4. SEM EDS profile showed oxygen peaks of 11.04 weight% and 2.08 weight% of Si and 1.03 weight% of Mn and rest as Iron.

After taking SEM pictures at different locations on the specimen, it is evident that there is a thick passive layer on the surface of the specimen. From the EDS profile the passive layer is conformed to be SiO₂. At higher magnification it was observed that the passive layer is a thick chain of SiO₂ network with average diameter of around 100nm. Passive film of SiO₂ and the average diameter of SiO₂ are shown in figure 3.2.5. From the mapping of the specimen surface it is observed that there is lot of oxygen and silicon on the sample. Passive SiO₂ layer network and the mapping of the AI specimen are shown in Figure 3.2.5. Due to presence of lot of silicate ions in the solutions there is chance of silicon oxide formation on the surface of the specimen.

Figure 3.2.5. The SEM Picture of AI rock bolt at 25°C in 1x YM water: (a)-Passive SiO₂ layer (b)-Thick SiO₂ chains, (c) - Mapping of the AI layer.

After comparison of the both SEM pictures of the AI potentiostatic scans at 25°C and 90°C, it is observed that the passive layer at the 25°C is stable but at 90°C the layer has cracks. From the fluctuations of potentiostatic scan for the experiment at 90°C, it can be said that the passive layer is not stable. The SEM pictures at the both temperatures are shown in figure 3.2.6.

Figure 3.2.6. SEM pictures of AI rock bolt in 1x YM; (a) - at 25°C, (b)-at 90°C.

3.2.4. **Summary:**
- Potentiodynamic scan of AI at 25°C and at 90°C showed passivation.
- Thick SiO₂ passive layer is formed on the surface of the specimen at both 25°C and 90°C.
- Passive layer is stable at 25°C but not stable at 90°C.

3.2.5. **References:**


(a) SEM of passive layer at 20°C for Al-1% Si alloy in potentiostatic scan at 0. (b) SEM of passive layer at 90°C for Al-1% Si alloy in potentiostatic scan at -0.53 V.