Stress corrosion testing of
ASTM A615 -Grade 60 steel in simulated underground coal mine atmosphere

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Abstract
Corrosion of roof support systems in underground mine environments could be a serious concern to ground control as it’s directly related to work force safety and possible production related issues. From Australian studies and also studies around the globe, stress corrosion cracking of roof bolts was found to be a problem in underground coal mines resulting in premature bolt failures (B.K. Hebblewhite, 2004). This has not been seriously studied or looked into in U.S. underground coal mines. As a preliminary study, the most common roof bolt material, ASTM A 615 steel that is used in the mines is studied under stress corrosion conditions. ASTM G30 standard, where the U-bend specimens are prepared from the material of interest and tested in the simulated coal mine conditions in a custom designed humidity chamber. The u-bend sample were tested for 15, 30, 45, 90, 180 and 330 days respectively in the corrosion simulated chamber and were analyzed for SCC at the respective time periods. Samples from 6 months showed signs of SCC. Based on these results alternative materials may be needed under certain conditions to avoid the bolt failures as a result of SCC, and more research on this subject is considered necessary in the U.S.

Key Words: stress corrosion cracking, strain induced corrosion cracking, roof bolts, underground mining, ground control, carbon steel.

Introduction
Roof support systems or rock anchors installed in underground mines are important considering work force safety first and then productivity. The purpose of the roof support systems is to control the ground for a safe and reliable opening over the design period. Stress corrosion cracking (SCC) is defined as the interaction of corrosion and mechanical stresses resulting in crack and failure of the material. The magnitude of the combined effect is a measure of the susceptibility of the material to stress corrosion. It is evidenced from
several studies around the world, that the metal strength loss resulting from the combined stress and corrosion is greater than the effects of stress and corrosion individually ("Stress Corrosion Cracking," 2000; Z. Jian, 2007). Though corrosion may initiate and proceed without any assistance of stress just with corrosive agents, stress nevertheless plays a major role in accelerating the things in terms of material cracking and growth (P.H. Hieu, 2010). One of the main concerns with SCC is its latency without any signs of warning before failure (Jones, 1992).

Several preliminary findings on rock bolt corrosion has been published in the mining journals by Spearing and their research group at SIUC (A.J.S. Spearing, K. Mondal, G. Bylapudi., 2010). One of the major problems with this rock bolt corrosion is the stresses they undergo in the environment (G. Bylapudi, 2015) and after careful consideration preliminary experiments on stress corrosion of this steel has been studied as part of this research, and the results are published in this paper.

The research on ground control systems materials is limited in U.S.A even though one of its major industries is mining. According to NIOSH report, close to 68 million roof bolts were used by the mining industry in the year 2005 and the same was assumed as per other reports during 2014 (A.J.S. Spearing, G. Bylapudi, K. Mondal, A.W. Bhagwat., 2014; S.C. Tadolini, 2006). The results from this study will be of greatest interest to the mining industry ground control professionals. The project results will lead to understand the subject material’s ability to withstand SCC in the underground mine environment else suggest an alternate material to use.

**Material and Environment**

One of the common support systems used in the underground coal mines is steel rebars installed in the mine roof to avoid roof falls. The most commonly used material as the rebars are ASTM A 615 steel and hence considered to investigate. The primary objective of this study is to evaluate the ASTM A 615 Grade 60 steel’s characteristics with respect to stress corrosion in the underground coal mine simulated environment from the ground water records collected from several mines (ASTM G30 is applied to achieve this objective). The ground water samples are simulated and introduced into the chamber on a circulating basis where the pH is being monitored on a regular basis and controlled to stay in the set range of 8.5, which is an average pH based on underground samples collected from 13 mines (G. Bylapudi, 2014). In addition to the pH, several chemicals were added to the water to simulate the underground aqueous atmosphere based on the records and considering the maximum assuming the worst case scenario, and the chemical concentration can be seen in the Table 1. The metallurgical data of the steel under investigation can be seen in Table 2. The secondary objective was to determine the material loss of the steel due to corrosion in the simulated environment after 15, 30, 45, 90, 180 and 330 days respectively (ASTM G1 is applied). The last objective of this study was to determine if the hypothesis is true or not, i.e. whether stress corrosion cracking is a problem or not underground and at what time period that does this problem initiates in the simulated environment. The testing was conducted in a custom designed corrosion chamber to simulate the corrosive environment. The corrosion chamber designed for this purpose can
be seen in Figure 1 below with the test samples. The main purpose of this custom designed chamber was to add chemicals to simulate the humid environment.

![Figure 1. Custom designed humidity chamber used for the testing the U-Bend steel samples](image)

Table 1. Chemical concentration of the water in the chamber

<table>
<thead>
<tr>
<th>Chemical</th>
<th>grams</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe(NO$_3$)$_3$.9H$_2$O</td>
<td>1.08</td>
</tr>
<tr>
<td>NaCl</td>
<td>52.22</td>
</tr>
<tr>
<td>Na$_2$SO$_4$</td>
<td>61.86</td>
</tr>
<tr>
<td>Water Sample in Liters</td>
<td>34 liters</td>
</tr>
</tbody>
</table>

Table 2. Chemical composition of the steel

<table>
<thead>
<tr>
<th>C %</th>
<th>Mn %</th>
<th>P %</th>
<th>S %</th>
<th>Si %</th>
<th>Cu %</th>
<th>Ni %</th>
<th>Cr %</th>
<th>Mo %</th>
<th>Sn %</th>
<th>V %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.35</td>
<td>1.08</td>
<td>0.018</td>
<td>0.069</td>
<td>0.21</td>
<td>0.46</td>
<td>0.11</td>
<td>0.11</td>
<td>0.021</td>
<td>0.010</td>
<td>0.027</td>
</tr>
</tbody>
</table>

Note: CEqvA706% = 0.56

Test Protocol

Since, all corrosion tests are site specific, the researcher undertaking these tests needs to spend the majority of the time in analyzing the corrosive atmosphere from field studies before simulating them in the lab.

ASTM grade 60 steel rebar was taken and cut and then machined into several rectangular strips as per the ASTM G30 standard. The machined test samples were bent close to v shape and then stressed with the nut and bolt mechanism maintaining in a constant strain conditions. To achieve this, two stage method was used. To avoid any galvanic corrosion, teflon insulators were used separating the specimen of interest and nut-bolt configuration. The Figure 2 shows the complete specimens before testing and Figure 3 shows the sample specifications. Once the samples were prepared, in the final stage, surface preparation was performed by degreasing unwanted material or foreign objects.
Figure 2. U-Bend samples fabricated as per the ASTM G30 Standard

Figure 3. Sample specifications

- **D** = 9.25 mm
- **W** = 20 mm
- **L** = 80 mm
- **M** = 52 mm
- **T** = 1.6 mm
- **R** = 6.5 mm
After the samples were prepared to the testing standard, the custom designed humidity chamber was set with the desired atmospheric conditions as to simulate the underground coal mine environment. As part of this testing the temperature was set to 35°C throughout in order to accelerate the test process simulating the worst case scenario. From the historical water sample records that were collected from various coal mines, an average pH of 8.5 is set for the testing and maintained throughout the project. In addition to that, the water was prepared based on the worst case scenario with maximum concentrations as per the records and the details were given in Table 1. All the samples were properly introduced into the chamber as to experience the humid environment similar to the underground. No samples were directly in contact with the water.

The total number of samples that were prepared for this test are 19 in which 3 samples were taken out of the chamber periodically and cleaned as per the procedure below for calculating the weight loss. An extra sample was used as a control sample for calculating the weight loss of the base material when cleaned in the chemicals as per the ASTM G1 standard. Reagent grade chemicals are used during the project from sample preparation, during testing and finally at the time of cleaning corrosion test specimens. For cleaning the corrosion specimens, reagent grade chemicals as mentioned in the C.3.5 from ASTM G1 cleaning procedures table has been applied, and the details can be seen in the Table 3.

### Table 3. Chemical solution used for cleaning corrosion specimens

<table>
<thead>
<tr>
<th>Designation</th>
<th>Material</th>
<th>Solution</th>
<th>Time</th>
<th>Temperature</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>C.3.5</td>
<td>Iron and Steel</td>
<td>500 mL hydrochloric acid (sp. gr 1.19)</td>
<td>10 min</td>
<td>20 to 25°C</td>
<td>Longer times may be required</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3.5 g hexamethylene tetramine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reagent water to make 1000 mL</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

After successful cleaning at each period, the weight loss data was recorded and analyzed. In addition to that, the samples were visually inspected for cracks, pits, etc. Later the samples were cleaned again via sonication using a sonicator for removing any unwanted materials and then inspected under a light microscope and then a scanning electron microscope for identifying the stress corrosion cracks, etc.

### Results

After successfully corrosion testing for the desired intervals of time and cleaning, the following results are obtained. The ideal cleaning procedure of the corrosion samples should remove the corrosion products, with minimum impact on the base metal removal during the chemical cleaning method used in this testing, yet a reference control sample was used for making necessary corrections to avoid base metal loss. The control sample base metal loss after 4 and 8 cycles of chemical cleaning is 0.03 and 0.05 grams respectively. The corrosion test samples from the chamber are first mechanically cleaned by scraping the corrosion products before chemical cleaning. The chemical cleaning procedure was performed on the samples several times and the mass loss was determined and recorded as WL1, WL2 and so on until WL8, where the number represent the cleaning cycle. The mass loss was graphed as a function of the number of cleaning cycles as shown
in the Figure 4. The material loss at a cycle where the data in the plot starts and stays almost parallel to the x-axis will be considered as the corrosion of the metal after removal of corrosion products. For 15, 30, 45, 90 and 180 day samples, fourth cleaning cycle, i.e., data from WL4 was determined to be the mass loss of metal due to corrosion whilst for 330 samples, eight cleaning cycle, data from WL8 was determined to be the mass loss.

The final mass loss data due to corrosion was determined for each sample at respective time period and the results are recorded as shown in the Table 4 for further analysis and for future reference. Mass loss data from sample 1 of 180 days was considered to be an outlier from professional judgement and hence ruled out from the data analysis. The graph from Figure 5 was plotted based on the obtained mass loss data and from the trend line it was evident that the mass loss due to corrosion is incremental with respect to time. The R-squared value of the trend line, which is 0.9464 also proves that it is a good-fit of the line to the obtained data.

Figure 4. Step wise weight loss measurement plots
Table 4. Weight loss data

<table>
<thead>
<tr>
<th>Sample. No</th>
<th>15 days</th>
<th>30 days</th>
<th>45 days</th>
<th>90 days</th>
<th>180 days</th>
<th>330 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.56</td>
<td>0.92</td>
<td>1.14</td>
<td>2.68</td>
<td>1.70</td>
<td>4.35</td>
</tr>
<tr>
<td>2</td>
<td>0.74</td>
<td>0.97</td>
<td>1.08</td>
<td>2.47</td>
<td>3.04</td>
<td>5.62</td>
</tr>
<tr>
<td>3</td>
<td>0.80</td>
<td>1.24</td>
<td>1.32</td>
<td>2.65</td>
<td>2.67</td>
<td>4.36</td>
</tr>
<tr>
<td>Mean</td>
<td>0.70</td>
<td>1.04</td>
<td>1.18</td>
<td>2.60</td>
<td>2.86</td>
<td>4.78</td>
</tr>
</tbody>
</table>

After the samples are treated and the mass loss was recorded, the microstructural changes in the samples were evaluated using scanning electron microscopy (SEM). Scanning electron microscope was used to determine the cracks due to stress corrosion after each time period. Samples from 15, 30, 45, and 90 showed no signs of cracking but there were pits developed at several locations. Samples from 180 days showed signs of SCC that can be seen in the Figure 6. The samples form 330 days are corroded very badly and the sample characterization showed no SCC as the cracks might have propagated into pits with time. Similar work with different grades and coatings in the future will result in more information for solid conclusions. Overall, this preliminary study resulted in some valuable information.

![Figure 5. Weight loss data with respect to time](image)
Conclusions
This stress corrosion testing proves to be a valuable and inexpensive methodology to identify the materials ability to perform in the simulated mine atmospheric condition. Similar tests in the future with different material grades, coatings will lead to better material selection for the specific conditions underground. Since, corrosion tests are site specific, the results vary from one location to other. SCC was not observed in the 15, 30, 45 and 90 days’ test samples. SCC was observed in 180 days’ samples and the samples from 330 days’ test set show advanced pitting resulting from crack enlargements. The weight loss of the test samples increased with respect to time for the designed test period.

References


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The authors are granting ASTM International the non-exclusive right to publish and disseminate the work from this project. The authors also would like to make a note that this work will be used and published in Gopi Bylapudi’s PhD dissertation and will also be published in other major mining related journals as a supportive work with proper acknowledgements and citations wherever necessary.

Conflicts of Interest
The authors wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced the outcome.