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EXPERIMENTAL PROTOCOL FOR STRESS CORROSION CRACKING OF ROCKBOLTS

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ABSTRACT: A new laboratory facility designed and constructed at the University of New South Wales, aims to continue and offer a new approach to researching the phenomenon of the stress corrosion cracking. This new approach includes the use of full sized specimens, a specially designed frame, as well as a new loading regime, known as the Periodically Increasing Stress Test, to closely simulate the loading encountered by bolts in service. Coupled with a detailed water testing program to be undertaken at a number of partner sites, this new approach hopes to further increase understanding of stress corrosion cracking and its causes.

INTRODUCTION

Stress corrosion cracking (SCC) is a failure mode, induced by the combination of an applied stress and an appropriately corrosive environment. SCC will result in the catastrophic, brittle failure of the material, as a result of crack growth, if remediation measures are not taken. SCC is a phenomenon that has caused significant problems for the underground coal industry for the last decade. After first being noted to occur at a BHP mine in the late 1990's (Gray, 1998), SCC of rock bolts has been an active area of research in Australia. Crosky, *et al*. (2002) noted that SCC was an issue in at least three Australian coal mines, although this number has since grown. SCC tends to occur in areas that have clay bands in the bolt horizon, thick coal roofs, corrosive ground water and shearing between the bedding planes (Crosky, *et al.*, 2002). They also note the importance of bolt metallurgy, particularly that of steel toughness, and the possible implication of microbiological action has been noted (Crosky, *et al.*, 2002). To date, no one has been able to effectively re-create underground service conditions to produce SCC failures in the laboratory. Gamboa and Atrens (2003, 2005) produced SCC failure modes in rockbolt steels in the laboratory and concluded that hydrogen embrittlement was the mechanism of failure. However, this work was not carried out in an environment representative of underground conditions. In further work, Villalba and Atrens (2007) concluded that SCC was not linked to any metallurgical factors, which is contrary to the findings of Crosky *et al*, (2002) and anecdotal field experience (Craig, *et al.*, 2010).

An Australian Research Council (ARC) linkage project introduced by Craig *et al*., (2010), aims to build largely on the work carried out by these and other researchers. Overall, the project intends to further classify the cause of SCC of rock bolts by undertaking a forensic analysis of the mine environment and bolts that have failed in service. This analysis will be extended to incorporate the design of a purpose built laboratory facility for simulating the mine environment. This facility will be used to induce SCC failure in specimen bolts so that the failure mechanisms can be identified. The project has the following five industry partners that have a history and/or concerns about SCC:

- Springvale Colliery (Centennial Coal) in the Lithgow seam - Western Coalfields;
- Blakefield Colliery (Xstrata Coal) in the Bulga Complex - Hunter Valley;
- Narrabri Colliery (Whitehaven Coal) in Gunnedah Basin;
- Moranbah North Mine (Anglo Coal Australia) in Bowen Basin, and
- Jennmar Australia.

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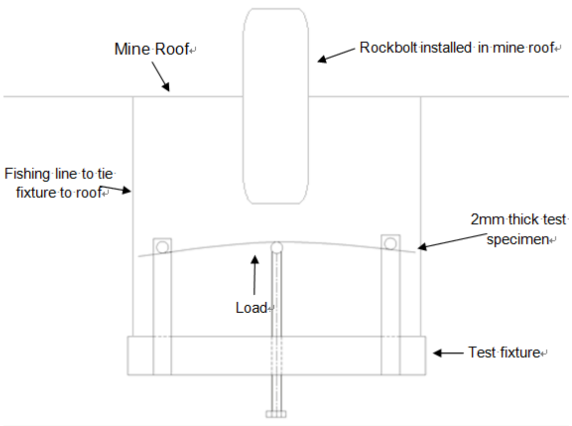
TESTING PROCEDURES

Environments that cause SCC are usually aqueous and can be either condensed layers of moisture or bulk solutions. As stated in the available literature, SCC is alloy/environment specific, that is, it is frequently the result of a specific chemical species in the environment. However, previous studies conducted into the SCC of rock bolts in underground mines have failed to identify/characterise the environment that is responsible for SCC. Consequently, this gives rise to one of the main aims of this project.

In order to identify the environment that gives rise to SCC, several miniature three-point coupon testing rigs, illustrated in Figure 1 will be placed in an underground coal mine. These test rigs with be hung from the roof of the mine, situated under a rock bolt with a consistent groundwater flow, and the water will be sampled and examined. The specimens that will be used in the test will be cut longitudinally across the top of the rock bolt of known chemical composition. This is so that each test includes the various factors for full-length rock bolts such as the stress concentration caused by the rig profile, mill scale and decarburized layer. Furthermore, the test specimens, which will be used, will have a thickness of 2 mm, which is excluding the rib thickness. This thickness was selected as fracture mechanics states that the thinner the test specimen the shorter the crack length needs to be before specimen failure occurs. In selecting such a small thickness not a lot of force will be needed to bend the specimen and the amount of deformation need to take the specimen to yield will be minimal.

Once the test specimens fail, the fracture surface will be examined using a Scanning Electron Microscope (SEM) in order to determine that the failure mechanism is that which occurs in service. Upon establishing this link, the environment will be sampled and examined so that it may be identified and compared to the result obtained prior to specimen failure. Consequently, it will be possible to see if there was any change any change in the water composition.

Water analysis will be carried out for each mine test site and steps will be taken to ensure that the results obtained from such tests are representative of the *in situ* conditions. It is important to note that the test method complies with both the "NSW Coal Regulations and Consideration to OHS" and the "Standard Methods for the Examination of Water, Waste Water and Ground Water".



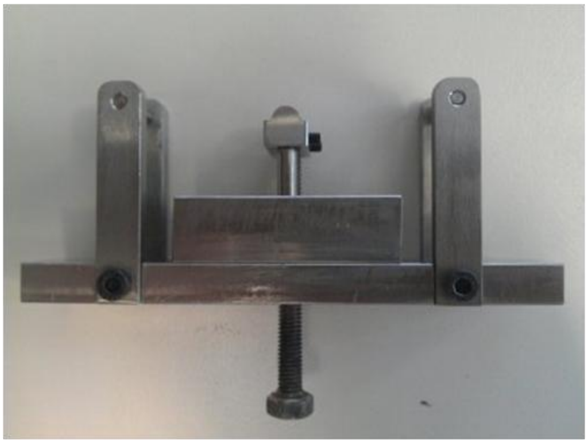


Figure 1 - Miniature three point bend test apparatus. Top shows a schematic of *in situ* test setup whereas, the bottom shows photo of the testing rig which has been constructed

Water sampling

The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled. This objective implies that the relative proportions or concentrations of all pertinent components will be the same in the samples as in the material being sampled, and that the sample will be handled in such a way that no significant changes in composition occur before the tests are made.

The type of sampling that has been chosen to be used during this study is known as the Grab method. Grab samples are single samples collected at a specific spot at a site over a short period of time. The sample bottles, which are being used are predominantly glass, with Teflon® bottles made from

Polytetrafluroethylene (PTFE) being used for samples collected for Inductively Coupled Plasma (ICP) analysis. This is because, this technique measures ionic and cationic species present in the sample and placing these samples in PTFE bottles prevents silica, sodium and boron being leached from the glass thus affecting the results.

Sample storage

Once samples have been collected, they will be packed in crushed or cubed ice before being transported for testing. This is to keep the samples as cool as possible, without freezing, to minimize the potential for volatilisation or biodegradation between sampling and analysis. However, to ensure that the measurements obtained from the analysis are as close as possible to *in situ* measurements, samples will be analysed as quickly as possible on arrival at the laboratory.

To determine the extent of change, if any, within a sample between the point of sampling and the point analysis, several tests will be conducted at various places. pH and dissolved oxygen will be tested at the point of sample collection with electronic probes. Once the samples taken back to the surface, they will be tested again using the same methods. The samples will then not be tested further until they arrive at the laboratory. If there is a significant degree of variation between each of the tests, then a new protocol will be implemented.

Water analysis

It must be noted that a proper characterisation of the environment is required as slight changes in the temperature, degree of aeration and/or concentration of ionic species can render an environment that is harmless into one that can cause SCC. Consequently, this allows evaluation of the environments that both cause SCC and don't cause SCC and assessment of the differences between the two. In doing this, a proper understanding of the environment that causes SCC will be established and appropriate remedial action will be implemented to prevent SCC of rock bolts. Table 1 outlines the different tests that will be used in this research to characterise the underground mine water along with the preservation techniques and container type being used.

The ICP analysis, outlined in Table 2 and Table 3 show different cations and anions which will be measured during this test, respectively.

Table 1 - Water testing regime

<table>
<tr><th colspan="2">Determination</th><th>Container</th><th>Sample Type</th><th>Preservations</th></tr>
<tr><td colspan="2">Acidity</td><td>Glass</td><td>Grab</td><td>Refrigerate</td></tr>
<tr><td colspan="2">Alkalinity</td><td>Glass</td><td>Grab</td><td>Refrigerate</td></tr>
<tr><td colspan="2">Solids</td><td>Glass</td><td>Grab</td><td>Refrigerate</td></tr>
<tr><td colspan="2">Hardness</td><td>Glass</td><td>Grab</td><td>Add HNO_3 or H_2SO_4 to pH <2</td></tr>
<tr><td colspan="2">pH</td><td>Glass</td><td>Grab</td><td>Analyze immediately</td></tr>
<tr><td colspan="2">Salinity</td><td>Glass</td><td>Grab</td><td>Analyze immediately or use wax seal</td></tr>
<tr><td colspan="2">Conductivity</td><td>Glass</td><td>Grab</td><td>Analyze same day, refrigerate</td></tr>
<tr><td colspan="2">Dissolved Oxygen (DO)</td><td>Glass</td><td>Grab</td><td>Refrigerate and ensure there is no head space when the bottle is full</td></tr>
<tr><td colspan="2">Biochemical Oxygen Demand (BOD)</td><td>Glass</td><td>Grab</td><td>Refrigerate</td></tr>
<tr><td colspan="2">Chemical Oxygen Demand (COD)</td><td>Glass</td><td>Grab</td><td>Analyze as soon as possible or add H_2SO_4 to pH <2; refrigerate</td></tr>
<tr><td rowspan="2">ICP</td><td>Anions</td><td>PTFE</td><td>Grab</td><td>Add H_2SO_4 to pH <2; refrigerate</td></tr>
<tr><td>Cations</td><td>PTFE</td><td>Grab</td><td>Refrigerate, keep in dark</td></tr>
</table>

Table 2 - Cations of Interest

Cations	
Sodium (Na)	Potassium (K)
Magnesium (Mg)	Calcium (Ca)
Iron (Fe)	Copper (Cu)
Zinc (Zn)	

Table 3 - Anions of Interest

Anions	
Bromide (Br^-)	Chloride (Cl^-)
Fluorine (F^-)	Nitrite (NO_2^-)
Nitrate (NO_3^-)	Sulfate (SO_4^{2-})
Phosphate (PO_4^{3-})	

Preliminary flow rate measurement of the water trickling off the bolts at Springvale Colliery has already been taken to calibrate the flow rates used in the experiments. Flow rate measurements are important as they provide information on the quantity of water passing over the bolts. Three bolts in the F-Heading at the mine were selected, each representing low, medium and heavy flow. The flow rates were used as design values for the reticulation system and these results can be seen in Table 4.

Table 4 - Flow rate measurements taken at Springvale Colliery

Description	Flow Rate (mL/min)	Flow Rate (L/Week)
Light Dripping	20	201.6
Heavy Dripping	60	604.8
Constant Trickle	100	1008

LABORATORY DESIGN

The core aim behind the design of the laboratory facility was to perform testing on full sized specimens. The work done by Gamboa and Atrens (2003) and Villalbao and Atrens (2007) focused on using small, representative samples for their SCC experiments. While useful in gaining an understanding, an upgrade to full sized specimen testing was needed to examine bolt surface characteristics such as de-carburisation and stress concentrations caused by ribs and surface irregularities.

It was identified from a review of American Society for Testing and Materials (ASTM) testing methods for examining SCC that two relevant tests existed for simulating the *in situ* stresses placed on a bolt. These were the bent beam test (ASTM G39) and the tensile test (ASTM G49).

The bend test applies a lateral side load to a specimen which generates high tensile forces in the outer radius of the specimen. A number of bend test configurations exists, however the four-point method was chosen because it generates a long section of bolt with a maximum stress (ASTM G38), and allows for easy coupling with a chemical cell (described later).

A static load, two point bent beam test was performed by Satola and Aromaa (2003) on two different types of rock bolt steel - Ø6 mm steel rebar and a Ø5 mm stand of king wire from a 15.2 mm cable bolt. Satola and Aromaa (2003) wanted to assess the corrosion difference between galvinised and un-galvinsed steel, SCC was not their primary focus. They stressed the specimens to 85% of the yield strength and left them to sit in a stagnant bath of varying solutions. Their experiments were unable to yield a SCC failure, however transverse cracking was found in some samples tested in low pH water with high Cl^- ion concentrations. No follow up examination of cracks were performed.

The Linearly Increasing Stress Test (LIST) carried out by Gamboa and Atrens (2003, 2005) is a mixture of a direction tension tests and a modified version of the Slow Strain Rate (SSR) testing, outlined in ASTM G49 and ASTM G129 respectively. The LIST experiment places a specimen in a rig that applies a constant loading rate of 0.019 $MPas^{-1}$ until the sample fails, either by SCC or under normal ductile overload. Gamboa and Atrens (2003) tested small, representative samples machined from rock bolt steel and had success in generating stress corrosion cracking and identified hydrogen embrittlement as the SCC mechanism. Close inspection of the fracture surfaces with a SEM indicated that the failure generated in the laboratory had a similar fractography to the failures experienced in service (Gamboa and Atrens, 2005). However the testing medium, a sulphate solution with a pH close to 2.1, could not be considered representative of normal groundwater conditions in Australia's major coal basins.

The laboratory facility can be divided into three key facets: the load frame, the chemical cell and environmental room. The load frame and the chemical cell will all be housed in an environmental room in which the temperature and humidity can be controlled. The room is designed to mimic the atmospheric conditions of an underground coal mine. Temperature will be controlled with the use of an air conditioning unit and will maintain air temperature between 16^{o}C and 24^{o}C. The temperature can be set and held with an accuracy of $\pm$1^{o}C. Humidity will be controlled with a steam humidifier mounted in the wall to keep a constant humidity within the chamber.

The load frame

The load frame design is based on several of the ASTM standards for conducting SCC testing. The frame will allow for both bend and tensile SCC testing to be carried out without altering the frame between tensile and bending tests. The frame is designed to load a single bolt at a time. As such, an array of frames will be used to test a number of bolts simultaneously.

Stress is applied to the bolt in the bend test by means of a torque multiplier and a screw. As the screw is turned, it moves a loading jig, which imparts a lateral load to the bolt. This lateral loading can be seen in Figure 2. The ends of the bolt are held in a fixed position in the frame. The displacement of the jig is measured to determine the amount of bend in the bolt, which allows the stress in the outer fibers of the bolt to be inferred analytically by equation 1, found in ASTM G39. However, this equation only applies within the plastic limit. The four point system also allows for a large area of the bolt to be held at a constant stress, allowing for accurate stress measurements in the bolt to be made. These results will be checked by attaching a strain gauge to the bolt and through numerical modelling.

$$\sigma = \frac{12Ety}{(3H^2-4A^2)} \qquad (1)$$

Where, E is Young's modulus, t is the steel coupon thickness, y is the amount of deflection, H is the distance between the outer supports and A is the distance between the outer support and the inner support.

The tensile tests are performed with the use of a hydraulic nut. This hydraulic nut is attached to one end of the bolt, while the other end is restrained with the use of a standard nut. The bolts used in the tensile testing are custom made to have threads at each end, which allows for this simple load mechanism. A load cell is incorporated in the system to measure the load on the bolt.

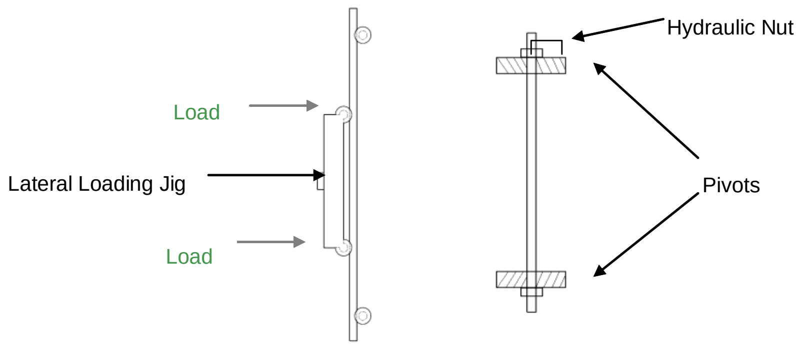


Figure 2 - Schematic of bend and tensile testing in the frame

It was identified from the work done by Gamboa and Atrens (2003, 2005) that a Linearly Increasing Stress Test (LIST) is an acceptable means of accelerating SCC growth. In the LIST carried out by Gamboa and Atrens (2003, 2005), stress in the sample is increased at the very slow rate of 0.019 MPa/s. However, due to the technical constraints associated with scaling this loading method up to a full sized specimen, a modified version of this test has been devised. Figure 3 indicates the design of the new load frame.

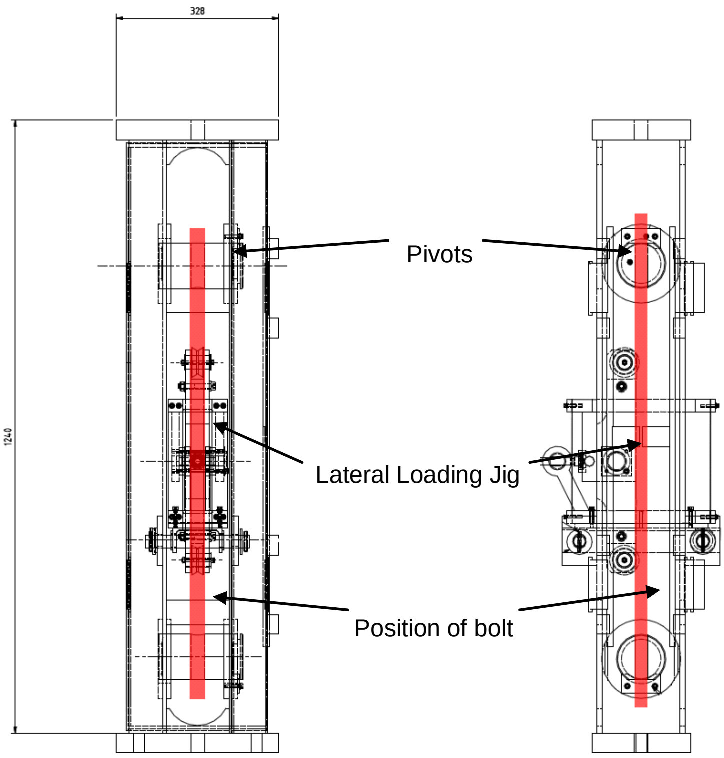


Figure 3 - Design of load frame

The modified loading method will be a Periodically Increasing Stress Test (PIST). This PIST test will work similarly to the LIST, however instead of the test increasing at a constant rate, the stress in the sample will be increased at regular intervals for the duration of the test. These intervals will probably be daily, and the increase in stress to be used will be determined through calibration testing, as outlined in ASTM G129. This loading mechanism is expected to more closely emulate the periodic loading regime experienced by bolts installed in underground coal mines.

The calibration testing described in ASTM G129 is for SSR testing; however it is believed the same principles can be applied for this testing protocol. ASTM G129 requires that the specimens be loaded (in tension) at a chosen rate in an inert environment, and measuring the time taken till failure. The test must then be repeated in the corrosive environment at the same loading rate. These tests must be repeated for a number of loading rates and results plotted on a time vs. loading rate graph to find the optimal loading rate (ASTM G129). The frame has also been designed so that it is easily adaptable to allow for SCC testing of cable bolts in the tensile configuration only.

The chemical cell

A chemical cell is used to contain the corrosive fluid to which the bolt is exposed. To closely replicate service conditions, the bolt is exposed to constantly flowing water. The cell itself is a length of clear vinyl tubing with a diameter large enough to fit a bolt inside and allow for fluid to flow freely around it. This design can be seen in Figure 4. Vinyl tubing was chosen because of its flexibility which will allow the cell to twist and flex while a load is applied to the bolt. The four-point bend test was chosen partly because it allows an easy arrangement to attach the chemical cell. Using a three point system would have posed problems with potentially rupturing the chemical cell as the load was applied. Figure 4 shows the positioning of the chemical cell in the bend test configuration. The same chemical cell will be used for the both the tensile and bend tests, however the cell use for tensile testing will encase more of the bolt.

The use of stagnant water in other test regimes (Satola and Aromaa, 2003; Spearing, *et al.*, 2010) has shown that the water chemistry can change dramatically during testing. A model similar to that used by Villaescusa, Hassell and Thompson (2008), where the water is kept in a continuous cycle will be used for these experiments.

The water used in the experiment is sourced from the mine sites supporting the project. It is known that stress corrosion cracking is occurring in underground mines (Crosky, *et al*., 2002, 2004). Using water sourced from site gives the greatest chance of capturing the environment that produces SCC increasing the likelihood of generating a SCC failure. Once the load frame has been validated, experiments will be carried out to isolate the water constituents causing SCC.

The water for the experiment is stored in large tanks within the environment chamber and circulated past the bolts, before being collected in a large sump tank. The water quality in the sump is tested frequently to ensure that the water chemistry has not deviated from the planned testing conditions. If the water passes, it is reticulated to the original holding tanks for reuse.

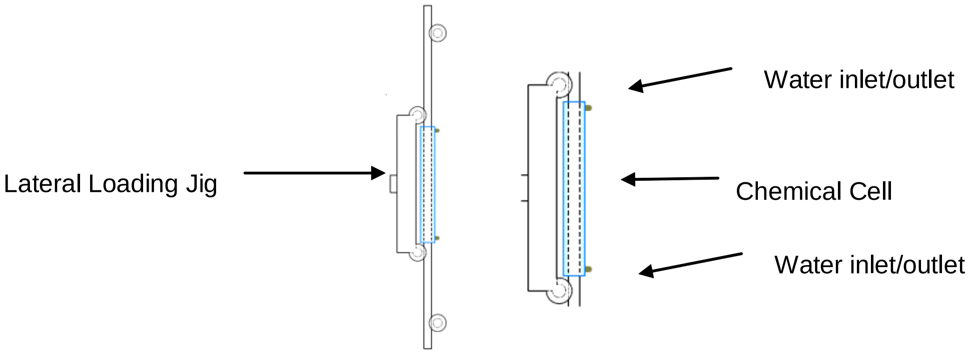


Figure 4 - Bend test schematic showing the position of chemical cell

CONCLUSIONS

SCC continues to be an issue in the underground mining industry. Continued research and experimentation is aimed at answering some questions around SCC and deliver solutions for the industry to use in combating SCC.

The laboratory facility designed at the University of New South Wales offers a new approach to SCC experimentation and research. The new PIST loading strategy is designed to closely emulate the loading experienced by bolts during underground use. The up-scaling to full sized specimens has been done to accommodate the influence of decarburisation and stress concentrations from ribs. By using water collected from mine sites as the corrosive medium in the corrosion chamber, there is a greater chance of capturing the environmental factors leading to SCC.

An extensive and detailed water sampling regime will identify all the potential factors that are causing SCC. The water survey is coupled with XRD analysis to determine the mineralogy of any clay bands that are suspected of playing a role in SCC. The knowledge gained from this environmental analysis is pivotal in identifying the SCC mechanism at play and devising solutions to remedy or prevent the problem.

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