

# METALLURGICAL ASPECTS OF ROCK BOLT ENVIRONMENT FRACTURE

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## ABSTRACT

The aim of this research was to understand the Stress Corrosion Cracking (SCC) of rock bolts. The laboratory tests have produced fracture surfaces similar to those from service. The experimental study elucidated the environmental condition leading to rock bolt SCC, and was used to determine the threshold stress and the threshold potential. A hydrogen embrittlement mechanism is proposed.

## 1. INTRODUCTION

Stress corrosion cracking (SCC) is used to describe the mode of failure of an engineering material where an environmentally induced crack slowly propagates through the material. SCC requires a susceptible material, a suitable environment (an electrolyte) and a tensile stress. Rock bolting provides strength to the rock mass through a combination of friction and mechanical interlock on the interface between the bolt and the rock strata. For the rock strata to fail and collapse, the clamping action of the rock bolts has to be overcome. Discussions have indicated that SCC is a significant problem in a significant number of Australian mines, Crosky [1] [2] since about 1994. There are indications that the tendency to use rock bolts of higher strength could have led to a higher incidence of SCC. SCC is expected to be manifest as first a period of slow crack growth over a period of time until the crack reaches the critical size (typically 2-3 mm in depth). Subsequently there is a sudden fast fracture across the remaining section of the bolt. Most SCC is reported to occur in the un-encapsulated part of the rock bolt. However, some SCC cases have been reported in the resin-covered area. The presence of H<sub>2</sub>S aggravates the hydrogen embrittlement type of SCC.

Laboratory testing was used to evaluate the SCC tendency of (1) rock bolt steels, and (2) different mine waters. Different steel metallurgies, electrochemical potential and pH values were studied.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 L.I.S.T.

The laboratory testing method, the Linearly Increasing Stress Test (LIST), consisted of applying a linearly increasing engineering stress to a specimen exposed to the environment of interest, Atrens et al [3], Ramamurthy and Atrens [4]. The load on the specimen is increased linearly by means of a lever principle and a moving load on the right hand side of the lever. The lever is maintained horizontal via a linear actuator and servo-controller by means of a displacement signal from the end of the lever arm. One side of the lever beam is connected to the specimen while the other side has a mass of 14 kg, which moves away from the fulcrum. This movement increases the load on the specimen at 0.019MPa s<sup>-1</sup> for the standard test. The applied engineering stress is calculated from the position of the mass at any time and the original cross section of the specimen. After the LIST test, the fracture surfaces were examined by Scanning Electron Microscopy (SEM).

## 2.2 Metallurgy

The chemical composition, mechanical properties, and metallurgies of these steels are presented in Tables 1, 2 & 3 respectively.

Table 1 Chemical composition of the commercial rock bolt steels. (%wt)

GRADE		C	Si	Mn	P	S	Ni	Cr	Mo	Al	V
1355	A	0.54	0.26	1.63	0.017	0.027	0.09	0.08	0.03	0.004	0.003
MA840B	B	0.37	1.05	1.46	0.013	0.009	0.01	0.02	0.01	0.004	0.043
MA810		0.36	1.00	1.40	0.021	0.013	0.01	0.02	0.01	0.005	0.040
MAC	C	0.25	0.36	1.32	0.016	0.027	0.07	0.05	0.01	0.005	0.210
10M30	AH	0.29	0.24	0.72	0.015	0.026	0.14	0.11	0.03	-	0.053
5152CW	D	0.54	0.10	0.90	0.015	0.025	0.35	0.90	0.10	-	-

Table 2 Mechanical properties of the commercial rock bolt steels.

GRADE	D (µm)	YS (MPa)	UTS (MPa)	Elongation (%)	Area reduction (%)	YS / UTS	CVN
1355	75	622	954	18.0	37.9	0.65	6
MA840B	65	635	873	22.2	50.3	0.73	18
MA810	43	689	838	21.4	52.2	0.82	29
MAC	-	-	-	-	-	-	-
10M30	-	400	670	22.0	-	0.60	-
5152CW10D	-	745	890	12.0	-	0.84	-

[ D = grain size ; YS = Yield strength; UTS = Ultimate Tensile strength; CVN = Charpy V-notch impact energy ]

Table 3 Metallurgies of the commercial rock bolt steels.

GRADE		C	Si	Mn	Ni	Cr	V	
1355	C-Mn eutectoid	A	0.54	0.26	1.63	0.09	0.08	0.003
MA840B	Microalloyed	B	0.37	1.05	1.46	0.01	0.02	0.043
MA810	Microalloyed		0.36	1.00	1.40	0.01	0.02	0.040
MAC	Microalloyed	C	0.25	0.36	1.32	0.07	0.05	0.210
10M30	Plain carbon	AH	0.29	0.24	0.72	0.14	0.11	0.053
5152CW10D	Cr+Ni + 10% CW	D	0.54	0.10	0.90	0.35	0.90	-

## 2.3 Constant Stress Test (Threshold Stress Determinations)

The threshold stress was determined using a modified LIST test. The mass was stopped at a predetermined position and the sample was held at a constant stress for three days in the sulphate pH 2.1 solution. If the sample did not fail at the end of the three days period, it was removed and cooled down to  $-197^{\circ}\text{C}$  and then struck with a hammer to break it in two pieces. The fracture surface was observed with SEM to determine the failure mode and whether there was SCC. If there was no SCC, it indicated that the stress was below the threshold stress for SCC.

## 2.4 Electrolyte

The standard sulphate-base pH 2.1 solution was made using reagent grade chemicals and distilled water to simulate what might be found in underground water samples at mines as follows. 1.6543 g  $\text{H}_2\text{SO}_4$ , 0.3285 g NaCl and 0.5959 g  $\text{Na}_2\text{CO}_3$  was dissolved to make 1000 ml solution.

## 2.5 Preparation of Fracture Surface

Rock bolts were photographed to record the as-received condition. Then the rock bolt was cut 10 mm below the fracture surface and cleaned with a solution of 5% Ethylenediaminetetra Acetic Disodium Salt solution (EDTA) for 3 min. The samples were mounted on aluminium stubs for analysis using Scanning Electron Microscopy (SEM). The same procedure was used for the LIST samples.

### 3. RESULTS & DISCUSSION

#### 3.1 Fractography and mechanism

A fractography analysis using SEM on rock bolts that failed in service in various Australian mines determined that SCC was the responsible fracture mechanism, Gamboa and Atrens [5-10]. Also, fractography analysis using SEM was made on the LIST samples produced in the laboratory. Comparison was made between the service and laboratory surfaces, Gamboa and Atrens [6-10] and both were shown to be similar, establishing the *Linear Increasing Stress Test* (LIST) as a valid laboratory test for SCC. The SCC surfaces contained the following different surfaces:

1. *Tearing Topography Surface (TTS)*, characterised by a ridge pattern independent of the pearlite microstructure, but having spacing only slightly coarser than the pearlite spacing.
2. *Corrugated Irregular Surface (CIS)*, characterised as porous irregular corrugated surface joined by rough slopes.
3. *quasi Micro Void Coalescence (qMVC)* in the rock bolts was different to that in samples failed in a purely ductile manner. The qMVC in rock bolts was flatter and more regular than the pure MVC, being attributed to hydrogen embrittling the ductile material near the crack tip.

The interface between the different fracture surfaces revealed no evidence of any additional mechanism involved in the transition between the fracture mechanisms. The microstructure had no effect on the diffusion of hydrogen nor on the fracture mechanisms.

The following mechanisms described the fracture process: hydrogen diffused into the material until it reached a critical level. The material embrittled allowing a crack to propagate through the brittle region. Once the crack propagated outside the brittle region it arrested. The corrosion reaction produced hydrogen that diffused into the material along the direction of the greatest triaxial stress, directly into the material perpendicular to the applied axial stress. Subcritical crack growth propagated once the material ahead the crack tip had reached a critical hydrogen concentration. Once the critical size crack had been reached the rest of the material failed by fast brittle failure.

#### 3.2 Environmental Influences and Influence of Metallurgy

A series of experiments were undertaken using 1355 AXRC to study the environment effect, Gamboa and Atrens [9]. SCC only occurred for environmental conditions, which produced hydrogen on the sample surface, leading to hydrogen embrittlement and SCC. The fracture surface of a LIST sample failed by SCC displayed the same fracture regions as fracture surfaces of rock bolts failed in service by SCC. The results indicated that SCC was controlled by a combination of the applied potential and the pH, but also indicated that the solution concentration had no effect. SCC occurred in the laboratory for a restricted range of condition: (a) acid conditions and (b) negative applied potential. These conditions were such that abundant hydrogen was produced on the surface of the steel, suggesting a hydrogen embrittlement mechanism.

A series of samples of 10M80 were tested, because this steel was perceived by the industry not to suffer from SCC. The sample subjected to the standard LIST test (sulphate pH 2.1 solution & loaded at 0.019 MPa/s) showed ductile fracture. However, the sample subjected to an extreme test condition (sulphate pH 2.1 solution and an imposed potential of  $-1100\text{mV}$  vs SHE) showed SCC (TTS) and ductile overload regions. It did not present CIS or qMVC regions attributed to a low effective diffusion velocity of hydrogen through the material although a considerable amount of hydrogen was evolved on the surface.

Steel 5152CW10D can suffer from SCC (as shown by service failures), but the condition causing SCC in the laboratory was the most severe condition of all the studied metallurgies, Table 4. 5152 steels suffered from SCC at a condition with less hydrogen production than those required for SCC in 5152CW10D steel, indicating that cold work helps increase the resistance to SCC of steel. Table 4 showed the threshold potential for different metallurgies tested in the standard pH 2.1 solution.

Table 4 threshold potential for different metallurgies tested in the standard pH 2.1 solution

GRADE			pH 2.1	
			No Scc	SCC
1355	C-Mn eutectoid	A	-250	-350
MA840B	Microalloyed	B		-350
MAC	Microalloyed	C		-350
10M30	Plain carbon	AH	-650	-800
5152	Cr+Ni + 0% CW		-800	-1000
5152CW10D	Cr+Ni + 10% CW	D	-1000	-1200
5152CW55	Cr+Ni + 55% CW		-1200	

Table 5 Determination of the SCC thresholds in the sulphate pH 2.1 solution.

Metallurgy	Stress not causing SCC (MPa)	Stress causing SCC (MPa)	Threshold Stress (MPa)
1355 AXRC	770 <sub>(NP)</sub> ; 861 <sub>(NP)</sub> ; 885 <sub>(NP)</sub>	922	900
MAC	700 <sub>(P after 1.3h)</sub> ; 800 <sub>(P* after 50h)</sub>	830	815
MA840B	700 <sub>(P after 27h)</sub> ; 800 <sub>(P* after 30h)</sub>	850	850
5152CW10D	-	-	> 960

- “P” indicates pitting causing ductile failure after the specified period of load application.
- “NP” indicates NO Pitting.
- “P\*” indicates a sample failed by pitting and stress corrosion cracks were found.

Table 6 Determination of SCC velocity for standard test.

Metallurgy	Sample	LIST test duration (s)	SCC crack size (mm)	Velocity (m/s)
1355 AXRC	LIST 24	53398	1.3	$2.4 \times 10^{-8}$
1355 AXRC	LIST 23	54000	1.1	$2.1 \times 10^{-8}$
MAC	LIST 70	44280	1.0	$2.2 \times 10^{-8}$
MA840B	LIST 69	50400	1.0	$1.9 \times 10^{-8}$

### 3.3 Threshold stress

Experiment were carried out to determine the threshold stress of various bolt metallurgies (900 MPa for 1355 AXRC, and 800 MPa for MAC & MA840B steels). The high value of threshold stress suggests that SCC begins in rock bolts when they are sheared by moving the rock strata. Table 5 summarises the results of the testing program to determine the SCC thresholds for the various metallurgies. A typical crack velocity was measured to be  $2.5 \times 10^{-8}$  m/s, indicating that there is little benefit for rock bolt to have steels with higher fracture toughness. Table 6 showed the determination of SCC velocity for the standard test. For the standard test, the time was taken to be the time to complete the experiment and assumed the scenario of a crack initiation and propagation as soon as the material was under any stress).

## 4. CONCLUSIONS

- Rock bolts failed in service had failed by SCC.
- The fracture surface was characterised by a thumbnail region of SCC and a brittle overload region.
- The fracture surface within the SCC region was characterised as Tearing Topography Surface (TTS), Corrugated Irregular Surface (CIS) and quasi Micro Void Coalescence (qMVC).
- A Fracture mechanism was proposed. A crack propagates as a front outwardly from the crack initiation site, perpendicular to the free surface of the material. The corrosion reaction

produced hydrogen that diffused into the material along the direction of the greatest triaxial stress, directly into the material perpendicular to the applied axial stress. Subcritical crack growth occurred once the material ahead the crack tip had reached a critical hydrogen concentration. Once the critical size crack had been reached the rest of the material failed by fast brittle failure.

- Laboratory work produced the same SCC fractography as the service failures, confirming the Linearly Increasing Stress Test (LIST) in the standard sulphate pH 2.1 solution provided a laboratory test for rock bolt SCC.
- Conditions leading to SCC were found to be associated with abundant hydrogen evolution (acid or at a negative potential), identifying Hydrogen Embrittlement as the likely mechanism.
- 1355 AXRC, MAC & MA840B metallurgies displayed SCC when loaded at 0.019 MPa/s in the sulphate pH 2.1 solutions at the free corrosion potential.
- 5152CW10D rock bolts had the best SCC resistance.
- Cold work increased the resistance to SCC.

## 5. REFERENCES

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