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Laboratory Testing of Rock Bolt Stress Corrosion Cracking

E. Gamboa
University of Queensland

A. Atrens
University of Queensland

LABORATORY TESTING OF ROCK BOLT STRESS CORROSION CRACKING

Erwin Gamboa [1] and Andrej Atrens [1]

ABSTRACT: The incidence of Stress Corrosion Cracking (SCC) in rock bolts has not been quantified and its magnitude has not been addressed. A laboratory test has been achieved that causes a tensile sample to fail in a manner similar to the failure mode observed from service failures, namely slow SCC followed by fast brittle fracture. The laboratory tests involve subjecting a tensile sam[2]ple to a linearly increasing stress at a slowly applied stress rate whilst the specimen in exposed to a dilute sulphate solution of pH 2.1. Detailed fractography of SCC fracture features from the LT has shown that these fracture surfaces have the same features as fracture surfaces of service failures. An SCC velocity can be calculated from these tests. This SCC velocity can be used to evaluate the benefit provided by a material with a higher fracture toughness. The SCC velocity measured from the laboratory tests indicates that the SCC lifetime is increased only marginally by the use of a rock bolt materials with a higher fracture toughness

Laboratory tests are being used to evaluate the threshold stress for rock bolts of various metallurgies and the environments causing stress corrosion cracking.

A hydrogen embrittlement mechanism for the SCC is indicated by the particular restricted range of conditions for which SCC occurs in the laboratory. In particular, SCC only occurs in the laboratory for the restricted range of environmental conditions corresponding to acid conditions at the open circuit potential (pH of 2.1 or more acid) or at negative applied electrochemical potentials corresponding to copious hydrogen evolution at the steel surface. This is consistent with reports from the USA indicating rock bolt failure due to the presence of H_2S in the mine atmosphere. Similarly, this failure mechanism is consistent with bacterial corrosion of the rock bolt surface during service producing acid conditions leading to SCC.

Water chemistry analyses carried out for a number of Australian mines (including one coal mine) visited during the 2002 suggest that SCC in a coal mine would be caused by bacterial corrosion locally decreasing the mine water pH down to a pH of 2.1.

INTRODUCTION

Any failure of a rock bolt is a potential concern. failures of rock bolts have been reported at a number of Australian mines. The incidence of in rock bolts has not been quantified and its magnitude is not fully understood.

Atrens and Wang (1995) provide a review of SCC which may occur whenever a stressed steel is in the presence of an aggressive environment. The stress corrosion cracks initiate and grow slowly. During this phase which may last months or years there may be no indication of any danger. Fast fracture occurs when the stress corrosion crack reaches a critical length, as determined by the applied stress and the fracture toughness of the steel. Reports indicate that the critical crack length can be of the order of only a few millimetres for rock bolts. The fast fracture is sudden and catastrophic.

Crosky et al (2002) provided a recent review. They analysed approximately 50 different rock bolts, including "AVH, AXR, X, HPC, Threadbar, Wriggle and Tempcored bolts". All the failed bolts examined "contained 0.4% - 0.6 % carbon, but were of a number of different chemical types, including manganese steels, chrome steels and micro-alloyed steels".

[1] *University of Queensland*
[2] *University of Queensland*

The fracture surfaces of rock bolts (composed of Steel *A*) whichfailed in service due to SCC have been described by Gamboa and Atrens (2002a). Subsequent laboratory research by Gamboa and Atrens (2002b) has shown that service failures can be duplicated in the laboratory using the Linearly Increasing Stress Test (LIST). The LIST test involves subjecting a tensile sample exposed to a sulphate pH 2.1 solution to a linearly increasing stress until fracture, with an applied stressing rate of 0.019 MPa/s. The indications were that this test could provide a good method to reproduce service SCC in the laboratory.

Laboratory tests were carried out to study the SCC of rock bolts, to measure the SCC threshold stress for different steel metallurgies and explor the influence of galvanising on rock bolt SCC.

EXPERIMENTAL DETAILS

Materials and Specimens

LIST samples were tensile samples with a gauge section of 20 mm x 3.5 x 2.5 mm. LIST samples were machined from commercial rock bolts for four different steel metallurgies from actual rock bolt samples: Steel *A*, Steel *B*, Steel *C* and Steel *D*. Table 1 gives typical values of the chemical compositions of these steels. Table 2 gives typical values for ASTM grain size (D) and mechanical properties.

Table 1 Typical Chemical Compositions of the Rock Bolt Steels

Steel	C	Si	Mn	P	S	Ni	Cr	Mo	Al	Cu	V	N
A	0.54	0.26	1.63	0.017	0.027	0.09	0.08	0.03	0.004	0.21	0.003	0.008
B	0.37	1.05	1.46	0.013	0.009	0.01	0.02	0.01	0.004	0.02	0.043	0.008
C	0.25	0.36	1.32	0.016	0.027	0.07	0.05	0.01	0.005	0.32	0.210	0.015
D	0.54	0.35	0.90	0.015	0.025	0.35	0.90	0.10		0.15		

Table 2 Typical ASTM Grain Size (D) and Mechanical Properties of the Rock Bolt Steels

Steel	D	YS	UTS	E	RoA	YS/UTS	CVN	CVN	CVN	CVN(Mean)
	μm	MPa	MPa	%	%		J	J	J	J
A	75	622	954	18	38	0.65	6	6	5	6
B	65	635	873	22	50	0.73	17	23	14	18
C	43	689	838	21	52	0.82	33	26	29	29
D		745	890	12						

Steel *A* contains 0.54%C in table 1and 1.63 % Mn in table 1. As the steel is made from scrap steel, it contains residual elements such as Cr, Si, Ni, Cu & Mo. A computer program is used to modify the Mn content of the melt according to the contents of the Cr, Si, Ni, Cu & Mo residuals, so that the steel has a 650 MPa minimum Yield Strength with no heat treatment. The steel has a nearly fully eutectoid microstructure.

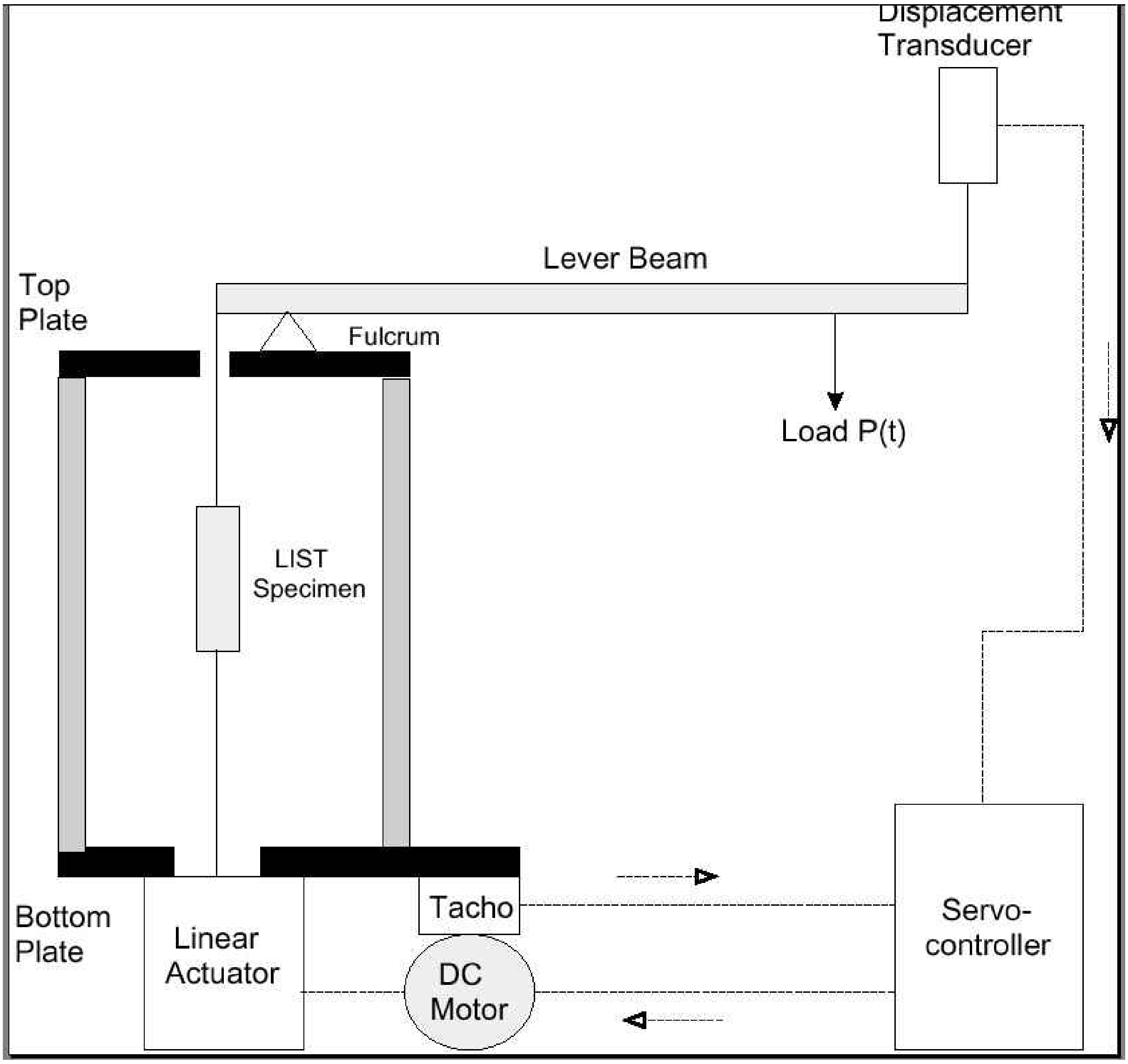
Steels *B* and *C* are micro-alloyed. They contain the carbide former V, which is used to control the austenite grain size during hot working, and are subjected to careful control of the hot working sequence so that they produce a fine grained microstructure of the required mechanical properties. They have lower carbon contents and higher values of Charpy notch toughness (CVN values in Table 2).

Steel *D* has a composition similar to that of the Steel *A*. Strengthening is produced by ~ 10% cold work.

The SCC tendency of galvanised rock bolts was evaluated using LIST samples of Steel *A*, that were hot dipped galvanised to have a 100 m zinc layer. Galvanised specimens were subjected to a standard LIST test in the sulphate pH 2.1 solution.

SCC Experiments

Figure 1 shows a schematic of the LIST apparatus from Atrens et al (1993). The apparatus is based on the principle of a lever beam. One side of the lever beam is connected to the specimen whilst the other side has a mass of 14 kg. Movement of the mass away from the fulcrum increases the load on the specimen. The applied engineering stress is calculated from the position of the mass at any time and the original cross section of the specimen.



The standard LIST experimental procedure was as follows. A specimen was inserted into the environmental cell and connected to the loading arms. The environmental cell was filled with the desired electrolyte. The LIST specimens had a free corrosion potential of -450 mV(SHE) in the standard sulphate pH 2.1 solution. The travelling mass was set in motion. Movement of the mass steadily transferred an increasing load onto the LIST sample. The mass was kept travelling until the sample fractured. All samples were loaded at 0.019 MPa/s, as the prior work by Gamboa and Atrens (2002a 2002b) indicated that the LIST test at this rate in the standard sulphate solution reproduced in the laboratory the same type of SCC fracture as observed in service. After the LIST test, the fracture surfaces were examined by Scanning Electron Microscopy (SEM).

The threshold stress was determined using a modified LIST test. The mass was stopped at a predetermined position and the specimen was held at a constant stress for 3 days in the standard sulphate pH 2.1 solution. If the sample did not fail by the end of the three day period, it was removed from the LIST apparatus, cooled to -197°C by immersion in liquid nitrogen, quickly withdrawn clamped in a vice and struck with a hammer, breaking the sample into two pieces at the thinnest part of the test section. The fracture surface was observed with SEM to determine the failure mode of the sample, and in particular whether a stress corrosion crack had formed. If no SCC crack was found, it indicated that the stress was below the threshold stress for SCC.

Test Solutions

All solutions were made using reagent grade chemicals and distilled water. Two standard solutions were used: chloride based and sulphate based. These might be characteristic of two different chemistries that might be found in underground water samples at mine sites. The following provide the details of these two standard solutions:

- Sulphate pH 2.1 solution. This contained 300 ppm sulphate, 100 ppm chloride and 100 ppm carbonate. This solution was made up as follows: 1.6543 g H_2SO_4, 0.3285 g NaCl and 0.5959 g Na_2CO_3 was dissolved in distilled water to make up 1000 mL of solution. The pH of this solution was measured to be 2.1. This solution is designated as "Sulphate pH 2.1".

- Chloride pH 1.8 solution. This contained 1400 ppm chloride, 300 ppm sulphate and 100 ppm carbonate. This solution was made up as follows: 1.6543 g H_2SO_4, 4.6056 g NaCl and 0.5959 g Na_2CO_3 was dissolved in distilled water to make up 1000 mL of solution. The pH of this solution was measured to be 1.8. This solution is designated as "Chloride pH 1.8".

Preparation of Fracture Surfaces

Macro-photographs were typically used to record the macroscopic appearance of the fracture surface of the rock bolt. Then the rock bolts were cut 10 mm below the fracture surface, cleaned using a 5% EDTA (ethylene diamine tetra-acidic acid disodium salt) solution, mounted on an aluminium stub, carbon coated and examined using scanning electron microscopy (SEM). Fracture surfaces of LIST specimens were prepared similarly. The LIST specimens were cut 10 mm below the fracture surface, cleaned using a 5% EDTA solution, mounted on an aluminium stub, carbon coated and examined using SEM.

RESULTS

A detailed comparison was carried out between LIST tests of Steel *A* exposed to the Sulphate pH 2.1 solution and Steel *A* rock bolts failed in service. There were the same fracture modes in both the service and laboratory samples. The fractures involved a small SCC region followed by a large fast fracture (FF) region. Figure 2 provides a typical overview for a service fracture. The images of Figure 2 represent optical images. The SCC region is easily identified by its darker colour due to the presence of corrosion products on the metal surface in the SCC region. This is due to the fact that the stress corrosion cracks grew slowly, allowing a period of time for corrosion to occur. In contrast, the overload region occured essentially instantaneously, so that the surface was bright and shiny as an un-oxidized steel surface. The tear lines radiated away from the fracture origin. These tear lines facilitated the identification of the SCC feature that initiated the final fast fracture event. It was particularly noteworthy that the service stress corrosion cracks often initiated in association with the ribs on the surface of the rock bolt as illustrated in Fig. 2(b).

FIG. 2(a) - Overview Of Service Fracture

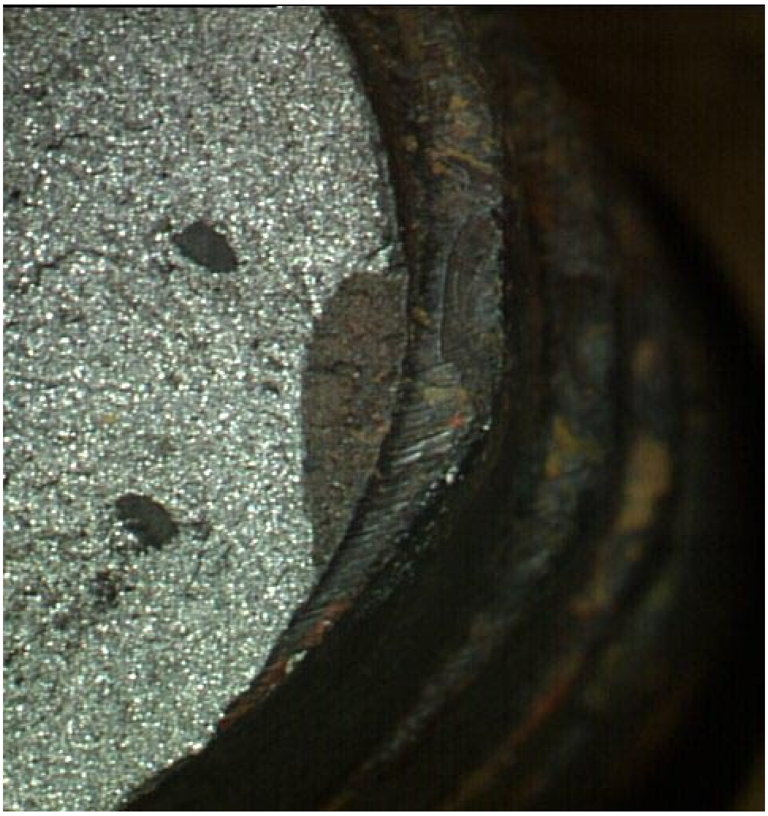


FIG. 2(b) - Service Fracture – Relation Of SCC To Rib

A direct comparison of a service fracture and a LIST fracture is provided in Fig. 3. Again, there was the SCC region and the brittle fast fracture (FF) of the overload region. These regions are identified in the schematics of Fig.s 3(b) and (d). It is worth noting that the fracture surfaces are macroscopically brittle with little indications of any macroscopic ductility.

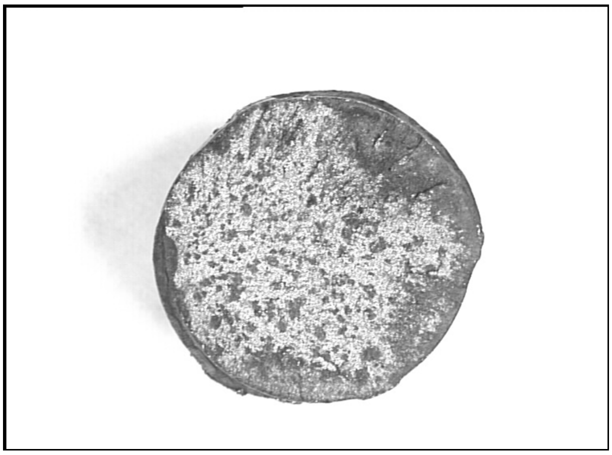


FIG. 3(a) - Service Failure

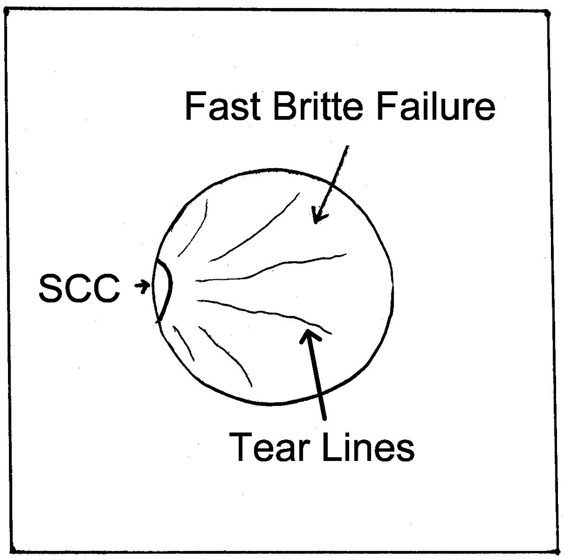


FIG. 3(b) - Schematic of FIG. 3(a)

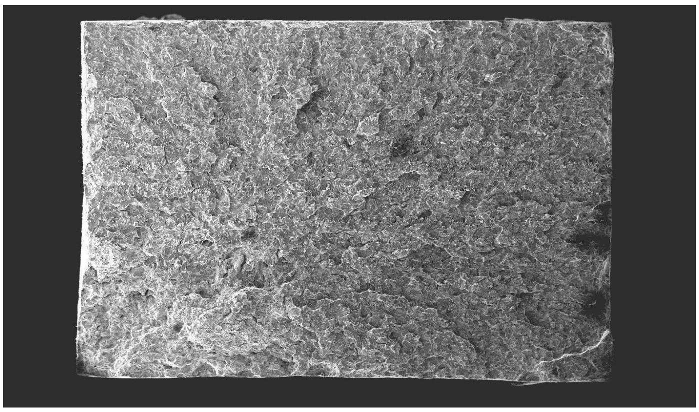


FIG. 3(c) - LIST Sample with SCC

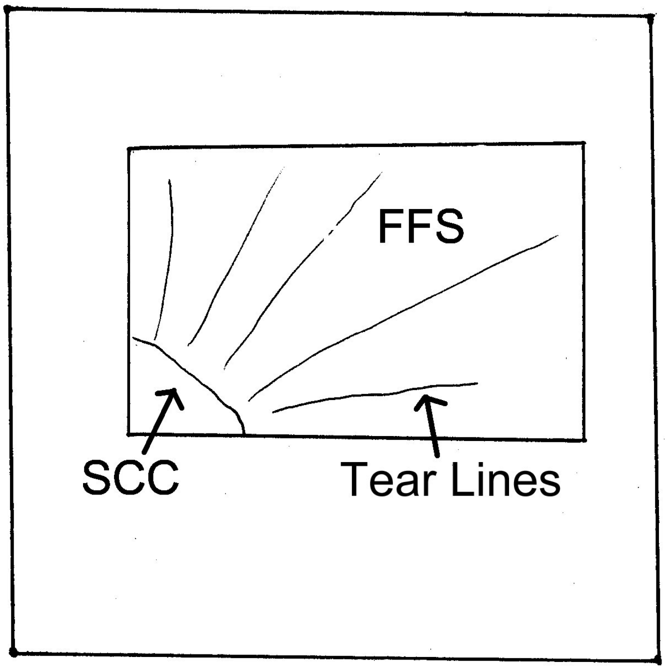


FIG. 3(d) - Schematic of FIG. 3(c)

Within the SCC region there were three different and distinct fracture morphologies: Tearing Topography Surface (TTS), Corrugated Irregular Slopes (CIS), and Micro Void Coalescence (MVC). These are illustrated in Figures 4-6.

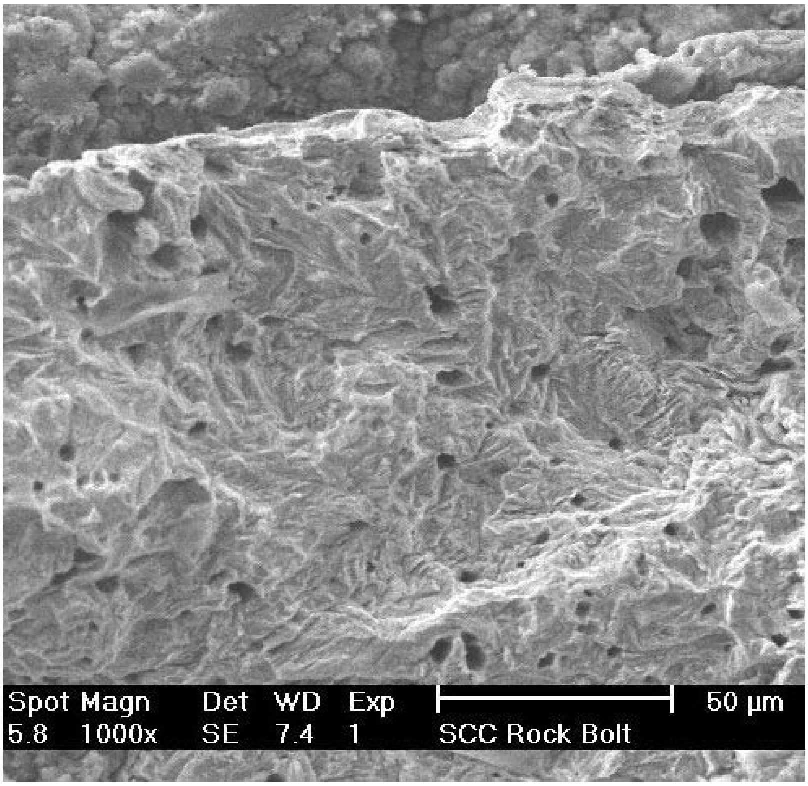


FIG. 4(a) - TTS observed within a rock bolt SCC region

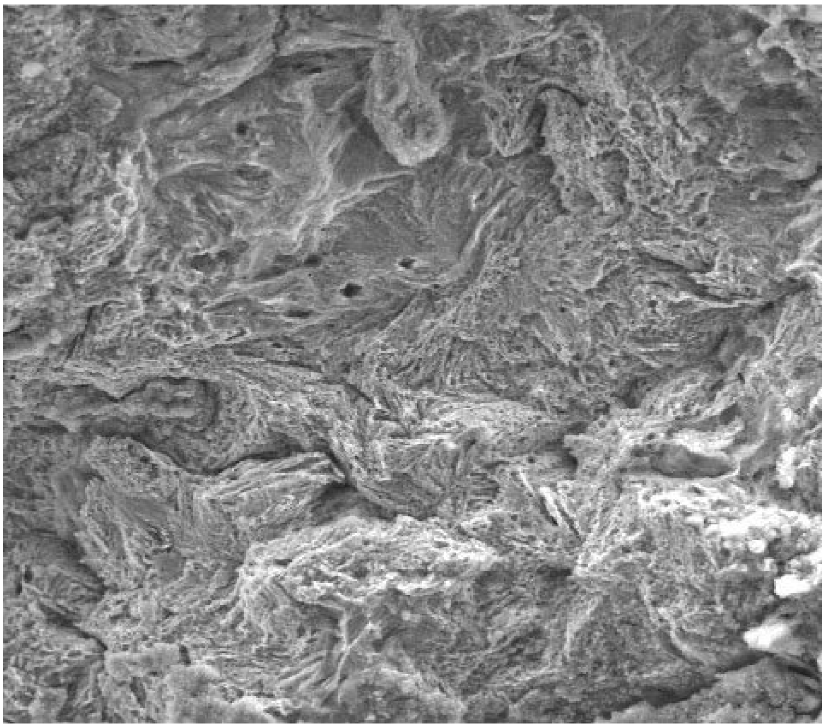


FIG. 4(b) - TTS observed within a rock bolt SCC region

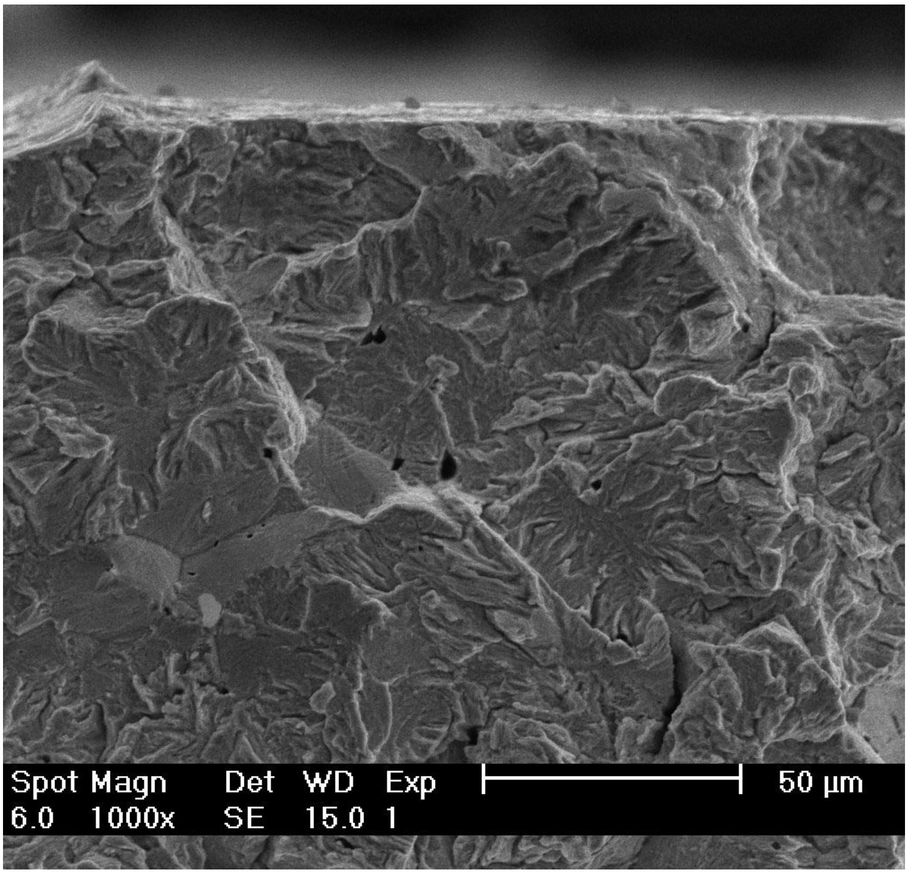


FIG. 4(c) - TTS observed within a LIST SCC region

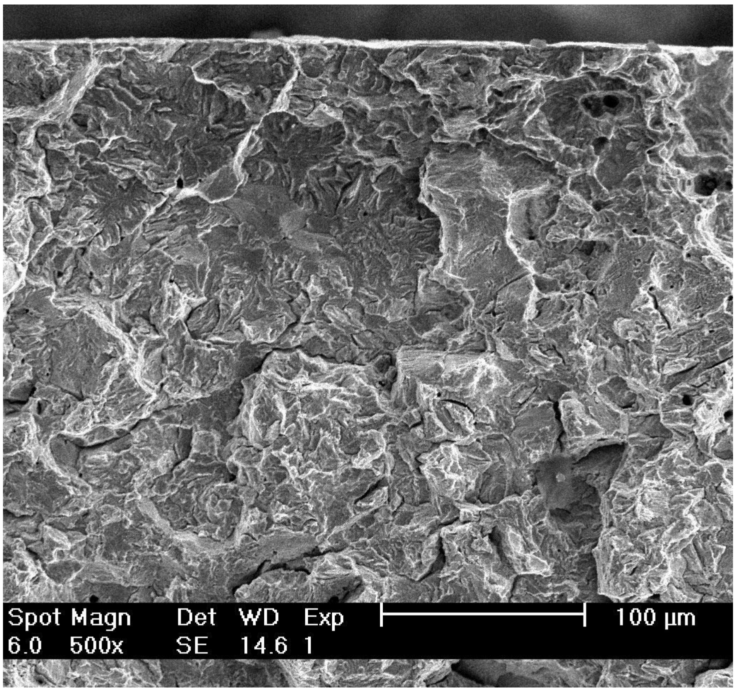


FIG. 4(d) - TTS observed within a LIST SCC region

Typical TTS morphologies for rock bolts are presented in Fig. 4(a) & (b) to allow comparison of typical TTS morphologies from LIST samples in Figures 4(c) & (d). The tearing topography surface was characterised by a flat convoluted surface with tiny ridges apparently oriented randomly. TTS typically occured close to the free surface and consequently was associated with the early stages of SCC. TTS has previously been described by Toribio and Vasseur (1997) as "a characteristic microscopic fracture mode with a kind of ductile tearing appearance, a certain degree of plasticity and a very closely spaced nucleation".

Typical CIS morphologies for rock bolts are presented in Figures 5(a) & (b) to allow comparison of typical morphologies from LIST samples in Figures 5(c) & (d). The CIS surface was characterised by flat plateaus separated by corrugated slopes.

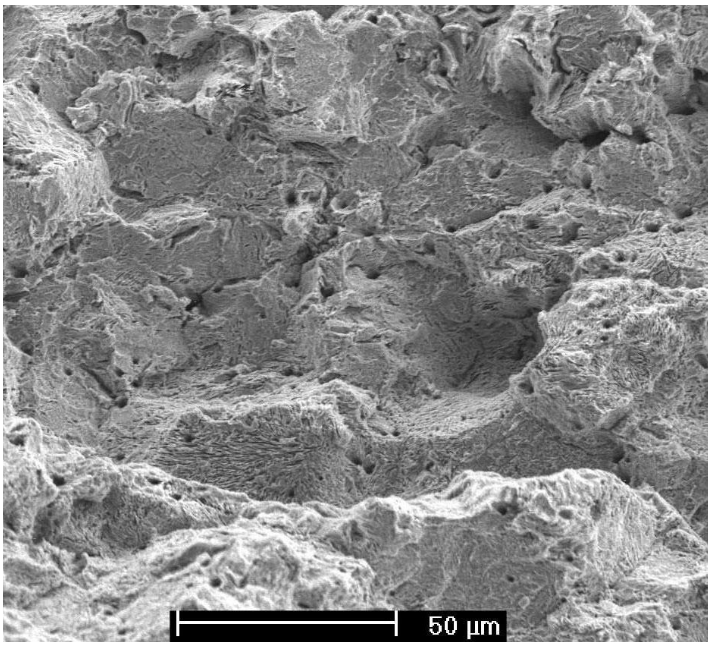


FIG. 5(a) - CIS observed within a rock bolt SCC region

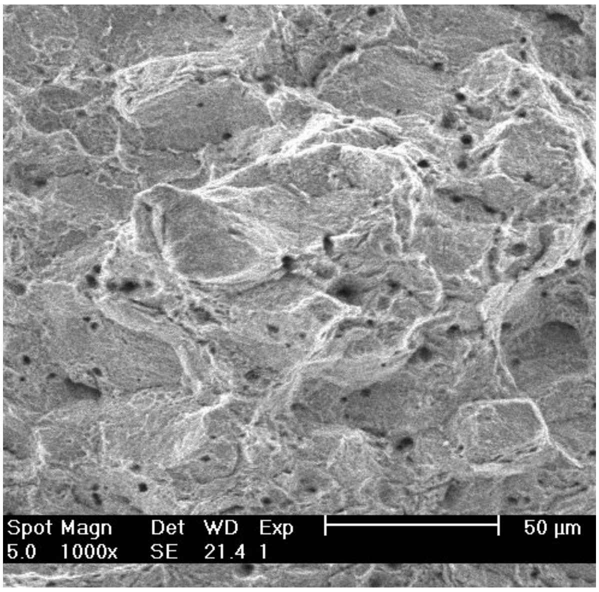


FIG. 5(b) - CIS observed within a rock bolt SCC region

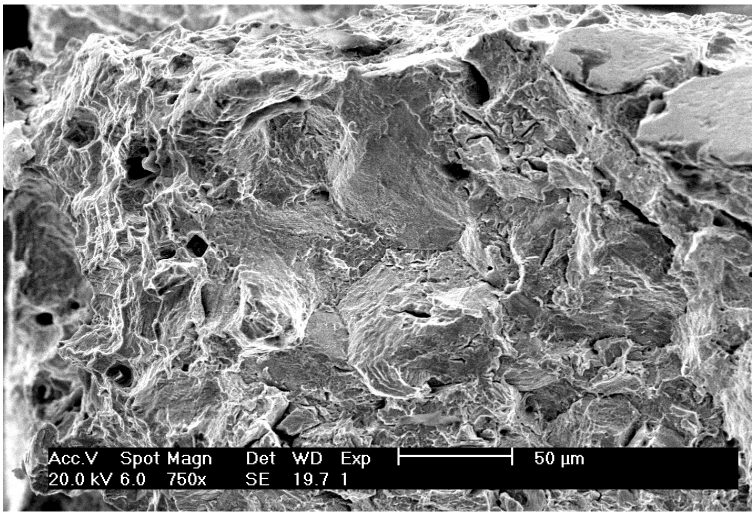


FIG. 5(c) - CIS observed within a LIST SCC region

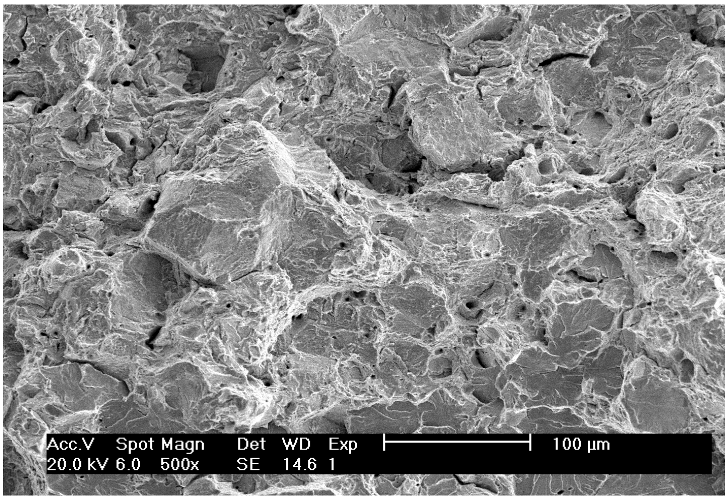


FIG. 5(d) - CIS observed within a LIST SCC region

Typical morphologies for the MVC-FF transition for rock bolts are presented in Figures 6(a) & (b) to allow comparison of typical morphologies from LIST samples in Figures 6(c) & (d). The MVC within the SCC region was significantly flatter that the dimple rupture observed in the overload region of tensile samples without SCC.

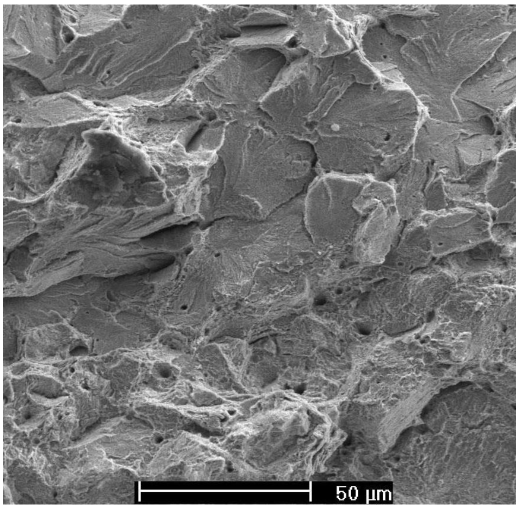


FIG. 6(a) - SCC(MVC)-FF transition observed within a rock bolt fracture surface

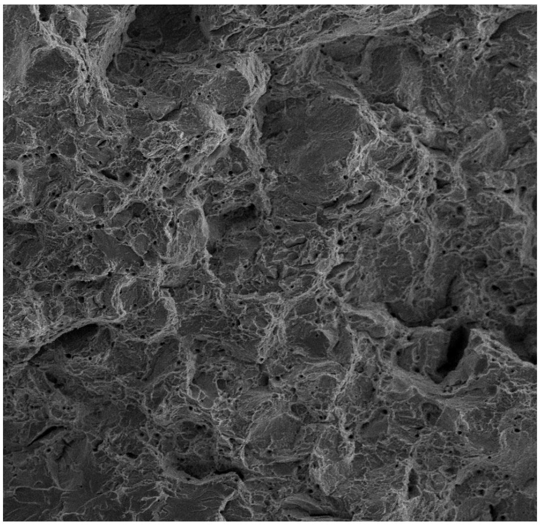


FIG. 6(b)- SCC(MVC)-FF transition observed within a LIST fracture surface

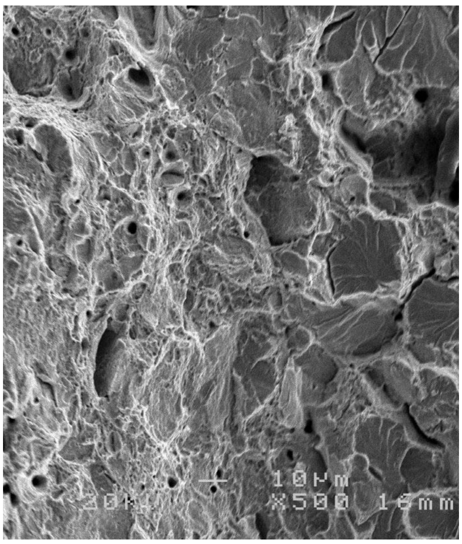


FIG. 6(c) - SCC(MVC)-FF transition observed within a LIST fracture surface

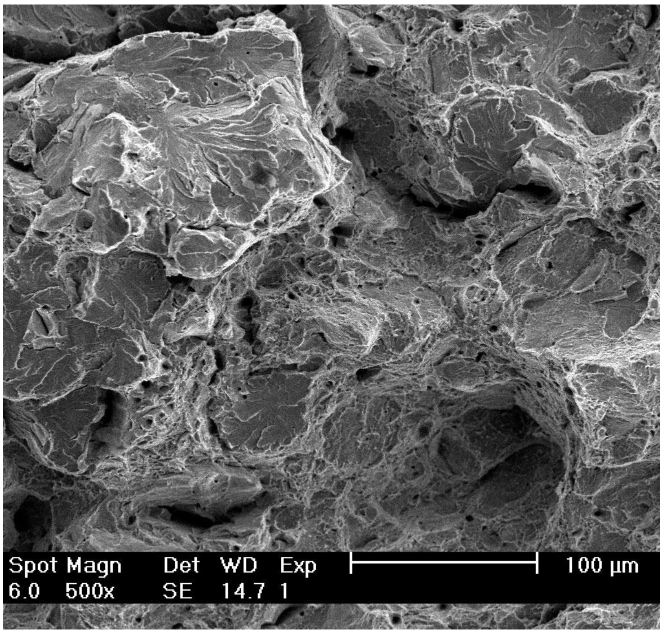
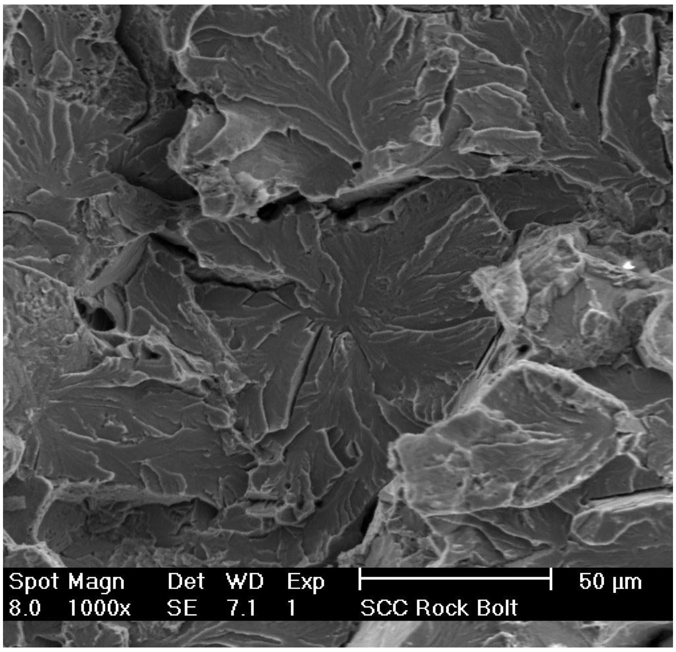


Figure 6(d) SCC(MVC)-FF transition observed within a LIST fracture surface

Typical brittle fast fracture (FF) morphologies for rock bolts are presented in Figures 7(a) & (b) to allow comparison of typical FF morphologies from LIST samples in Figures 7(c) & (d). The FF morphology was typical of cleavage fracture.



IG. 7(a) - FF (cleavage) observed within a rock bolt fracture surface

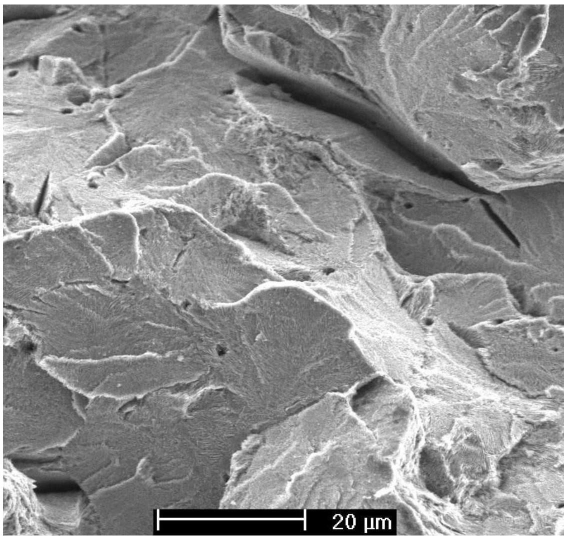


FIG. 7(b) - FF (cleavage) observed within a rock bolt fracture surface

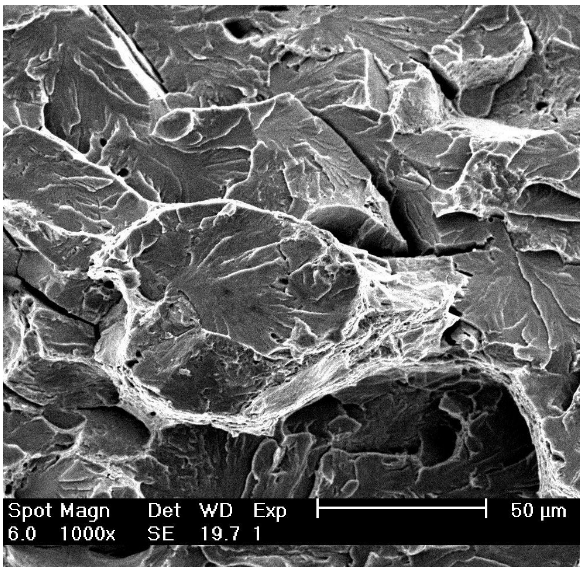


FIG. 7(c) - FF (cleavage) observed within a LIST fracture surface

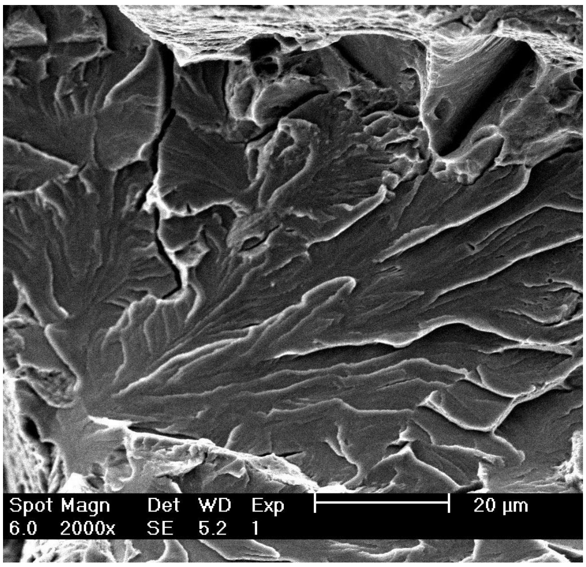


FIG. 7(d) - FF (cleavage) observed within a LIST fracture surface

Figure 8 proves an overview of the SCC region of a LIST fracture surface, with the schematic of Figure 8(b) identifying the various fracture micro-mechanisms.

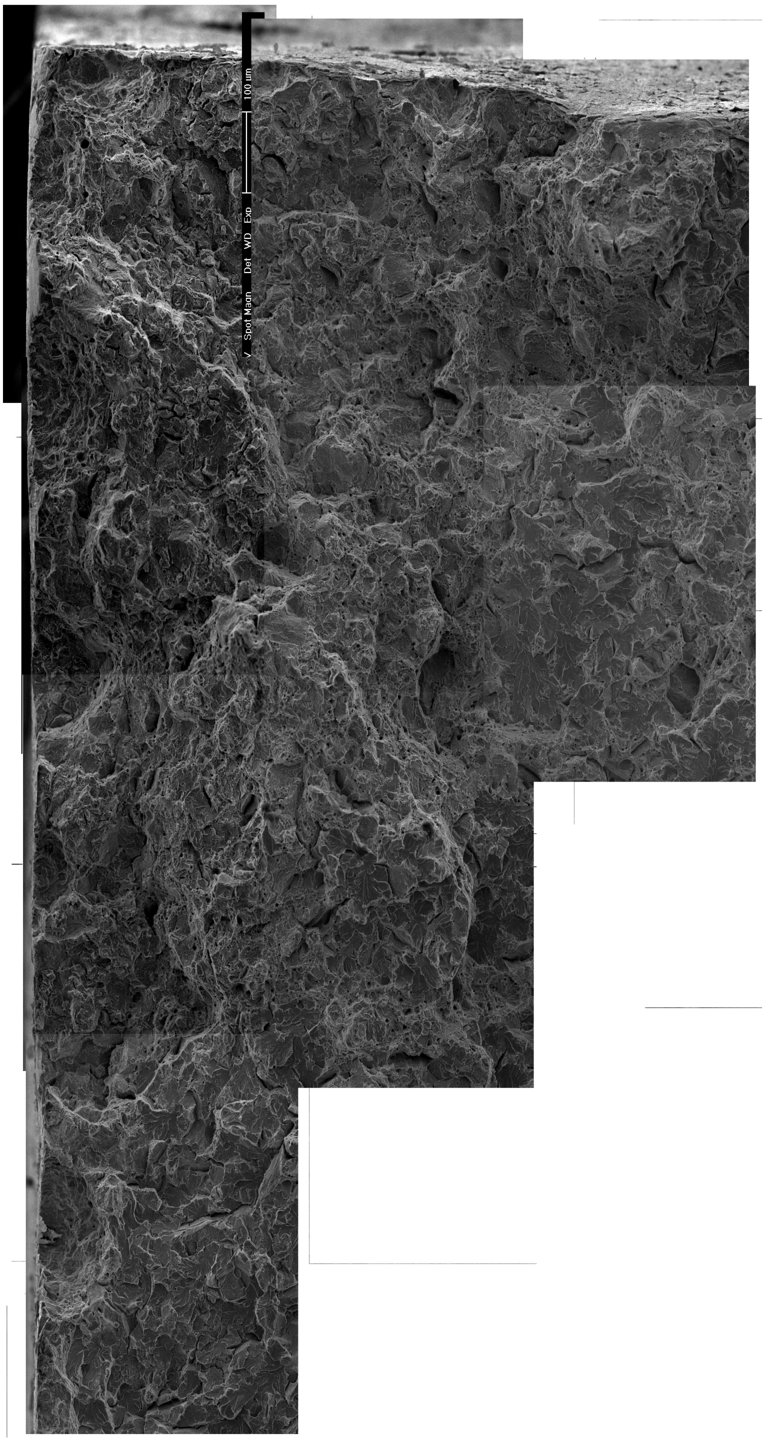


FIG. 8(a) - Mosaic of the SCC region of a LIST fracture surface

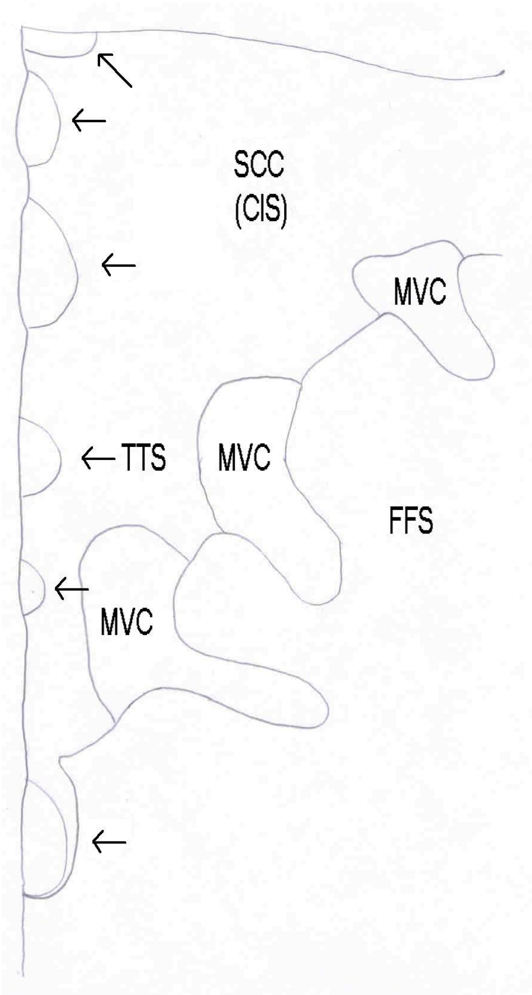


FIG. 8(b) - Schematic of FIG. 8(a) - indicating the various fracture modes

Environments Causing SCC

The results of the SCC tests in the various experiments are summarised in Table 3 where S indicates a sulphate solution and C indicates a chloride solution. In the first tests only one environmental factor was changed (either the pH, the concentration or the corrosion potential), whereas in subsequent tests two factors were modified in order to study interactions, for example the pH and the corrosion potential. The data indicates that SCC was controlled by the combination of the applied potential and the pH. Furthermore, the data indicates that the solution concentration was not an important issue.

Table 3 Results of the standard LIST test for Steel *A* in various environments

Sample	Environment	SCC
LIST24	S pH 2.1	Yes
LIST41	S pH 2.1 and CO_2	Yes
LIST 38	S pH 2.1 and coal, aerated	Yes
LIST 44	S pH 2.0, x10 conc	Yes
LIST 43	S pH 2.0, x100 conc	Yes
LIST 42	S pH 2.0, x1000 conc	Yes
LIST 28	S pH 4.2	No
LIST 29	S pH 6.3	No
LIST 34	S pH 9.4	No
LIST 47	S pH 2.1 Ecorr	Yes
LIST 48	S pH 2.1 Ecorr+100 mV	No
LIST 50	S pH 2.1 Ecorr+150 mV	No
LIST 49	S pH 2.1 Ecorr-100 mV	Yes
LIST 51	S pH 2.1 Ecorr-300 mV	Yes
LIST 52	S pH 6.2 Ecorr	No
LIST 53	S pH 7.27 Ecorr-300 mV	No
LIST 54	S pH 7.46 Ecorr-570 mV	Yes
LIST 45	S pH 6.16, x100 conc, Ecorr	No
LIST 64	S pH 6.6, x100 conc, Ecorr-300 mV	No
LIST 66	S pH 6.6, x100 conc, Ecorr-500 mV	Yes
LIST 85	S pH 1.2, Ecorr-150 mV	Yes
LIST 87	S pH 1.2, Ecorr+100 mV	Yes
LIST 88	S pH 11.8, Ecorr-850 mV	Yes
LIST 89	S pH 11.8, Ecorr-500 mV	No
LIST 26	C pH 1.8	Yes
LIST 39	C pH 1.8 and CO_2	Yes
LIST 40	C pH 1.8 and coal, aerated	Yes
LIST 46	C pH 1.8, x100 conc	Yes
LIST 31	C pH 3.1	No
LIST 37	C pH 7.7	No
LIST 36	C pH 10.7	No

The data has been displayed on an E-pH diagram in Figure 9 allowing identification of the conditions leading to SCC for LIST testing of Steel *A* samples. Sulphate solutions are represented by circles and chloride solutions are represented by squares. A full symbol indicates fracture by SCC, whereas an empty circle or square means that no SCC was detected. Symbols with a flag represent experiments performed with an applied potential.

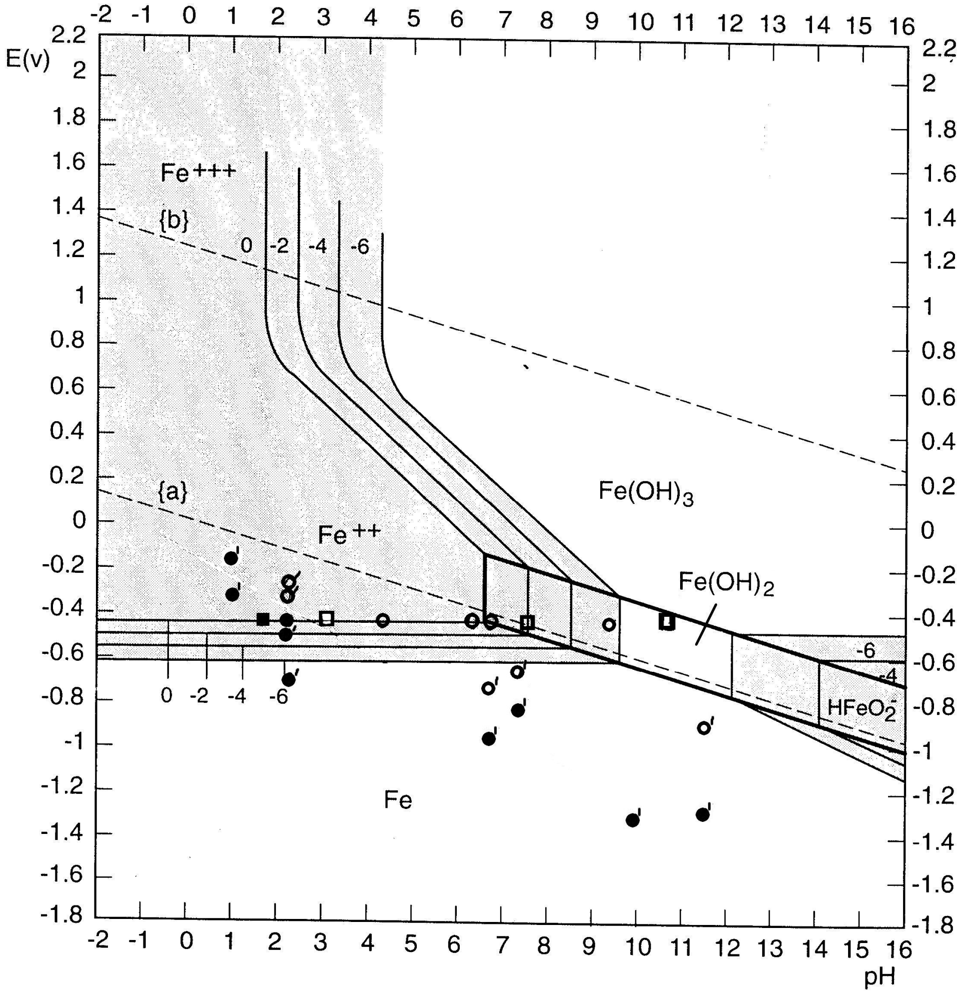


FIG. 9 - LIST test results superimposed on an E-pH diagram. Full symbols indicated SCC, open symbols indicated ductile failure in the LIST test. Circles represented the sulphate solutions. Squares represented the chloride solutions. Symbols with flags indicated tests under potential control

SCC only occurred in these laboratory tests for the restricted range of environmental conditions corresponding to acid conditions at the open circuit potential (pH ~ 2.1 or more acid) or at very negative applied electrochemical potentials corresponding to copious hydrogen evolution at the steel surface. This indicated that a hydrogen embrittlement mechanism was responsible for the SCC. This is consistent with reports from the USA indicating rock bolt failure due to the presence of H_2S in the mine atmosphere. Similarly, this failure mechanism is consistent with bacterial corrosion on the rock bolt surface during service producing acid conditions leading to SCC.

Determination of Threshold Stress

The results of a testing program to determine the threshold stress for SCC to occur are summarised in Table 4. Some samples did not fail by SCC but displayed pits, which caused ductile overload. Samples that failed in this way have been classified as failing by pitting and a "P" designation has been given. An "NP" designation means that the sample did not have substantial pits, nor was the failure associated with pitting.

Steel *A* samples held at 770, 861 or 885 MPa did not fail in the LIST apparatus during the period of 3 days during which the load was held constant. Furthermore, these samples did not show any evidence of SCC when they were fractured at the temperature of liquid nitrogen. In contrast, the sample held at 922 MPa fractured after eight hours. The fracture surface showed a typical macroscopically brittle appearance typical of SCC causing a fast brittle fracture. Detailed SEM examination was consistent, showing a clear region of SCC followed by a FF region. Furthermore, surface corrosion damage of these Steel *A* samples was limited. There were wide shallow pits all over the gauge surface but there was no evidence of SCC.

Table 4 Determination of the Threshold Stress [MPa] for SCC

Steel	Stress not causing SCC	Stress causing SCC	Threshold Stress
A	770 (NP), 861(NP)	922	900
B	700 (P after 1h), 800 (P* after 50 h)	830	815
C	700 (P after 27 h), 800 (P after 70h)	850	850
D	-	-	> 960

*fractography indicated both pitting & SCC – test is being repeated.

"P" indicates pitting causing sufficient decrease in section to cause ductile overload failure after the specified period of load application.

"NP" indicates no pitting. For all tests of the Steel *A* samples, the samples did not fail during the 3 day exposure period to the specified stress.

The Steel *D* material showed ductile failure for the LIST test completed to fracture in the standard Sulphate pH2.1 solution.

The Steel *B* sample held at 830 MPa failed after 50 hours. The gauge surface displayed extensive surface corrosion and many stress corrosion cracks as shown in Figure 10. Samples held at lower stresses failed fairly rapidly by pitting. It is possible that SCC might have occurred at these lower stresses if failure had not occurred by pitting. A more accurate determination of the SCC threshold stress requires use of a different specimen with a substantially larger cross-section. A larger specimen would tolerate a larger pit size before the section was reduced sufficiently to cause ductile overload fracture. Pits typically decrease in propagation velocity as they grow in size. This means that for a specimen with a larger cross-section area, there would be much longer exosure before failure by pitting, providing more time for SCC initiation and growth.

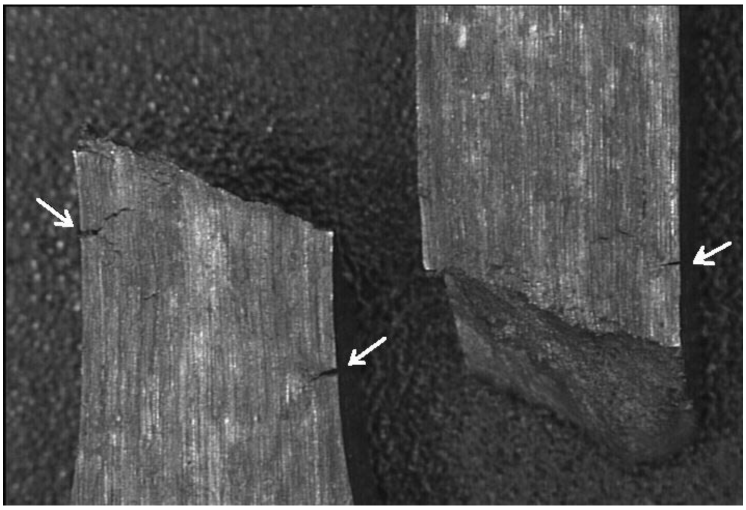


FIG. 10 - SCC along LIST gauge length of Steel *B* sample.

The Steel *C* sample held at 850 MPa failed after 71 hours. The fracture surface displayed both ductile fracture (shown by the lip formed by plastic flow) and also displayed SCC features. Surface damage was extensive all over the gauge surface, with many pits and many secondary SCC illustrated in Figure 11.

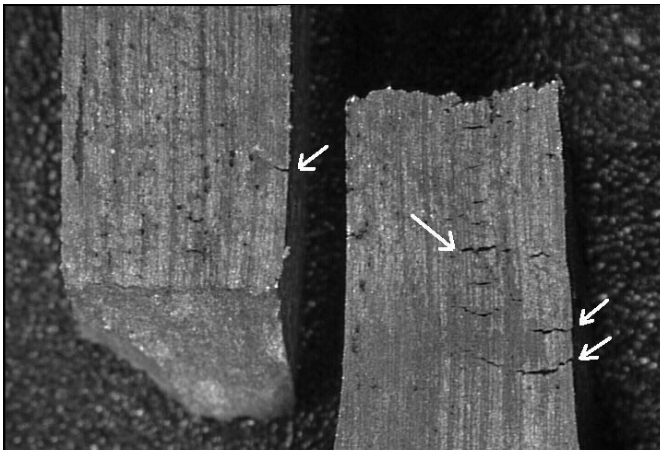


FIG. 11 - SCC along LIST gauge length of Steel *C* sample

The Steel *D* samples failed in a ductile manner, for the LIST test that proceeded until specimen fracture at an applied stress of 960 MPa. The fracture surface was typical of ductile fracture, Figure 12. The ductile failure surface illustrated in Figure 12(a) and the brittle fracture surface illustrated in Figure 3(a) may be compared.

Mine Water Chemistry

In order to compare the laboratory solutions to the mine waters, which represent the service conditions, samples were taken from various Australian underground mines. Table 5 presents the composition of the various mine water samples collected during 2002.

Table 5 Mine Water Compositions

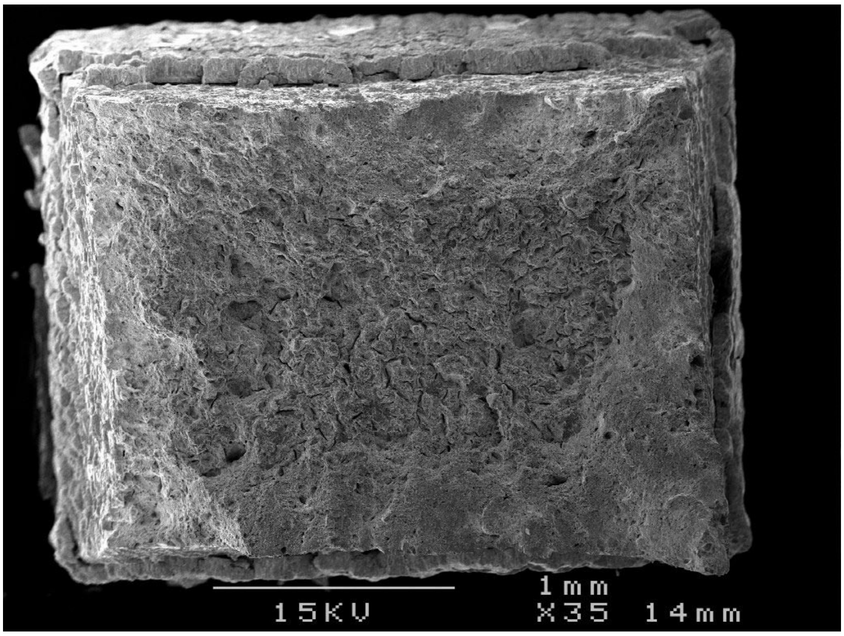
Sample		pH	EC	Ca	K	Mg	Na	S	Cu	TDS	Cl	NO_3	Alkalinity
			mS	mg/L									Mg/L $CaCO_3$
EC1	Dam 1	7.6	50.5	835	99	1188	14839	2090	0.015	47221	23600	2.12	312
EC2	Dam 2	7.2	48.8	809	98	1092	13382	1925	0.014	42161	22600	0.05	347
EC3	2K3	8.1	1.9	155	13	109	117	157	0.017	1508	1082	1.24	396
EC4	2I3	8.3	1.4	91	17	108	96	132	0.018	1183	636	7.95	334
EC5	13DA	6.8	15.3	488	164	4100	831	5854	0.024	30436	83	1.45	211
EC6	13DB	7.1	15.4	489	165	4100	831	5760	0.029	31215	57	7.30	209
EC7	13DC	7.7	16.4	489	189	4420	938	6148	0.004	30480	84	3.09	176
EC8	New	7.9	4.4	113	9	165	750	179	0.018	3006	1736	0.99	968
EC9	Paj	7.7	4.1	423	9	56	546	464	0.035	3322	1434	0.14	182
	Sulphate	2.1	-	0	0	0	388	540	0	-	100	-	100
	Chloride	1.8	-	0	0	0	1942	540	0	-	1400	-	50

All of these samples were taken from metalliferous mines, except for sample EC8 which was taken from a colliery. The composition of EC8 similar to that of the others samples. A comparison of the field samples and the standard sulphate pH 2.1and the chloride pH 1.8 solutions indicated that the chemical composition of the laboratory solutions have similarities to those in the field, particularly to samples EC4, EC8 and EC9. Some of the field samples have much higher concentration of sodium, sulphate and chlorides than the laboratory standard solutions. EC1 and EC2 has more than 4 times the concentration of Na, S and Cl than the laboratory standard solutions. Even though field samples had higher concentrations, none of the mines sampled have reported problems of rock bolt SCC. This is consistent with the present results that show solution concentration was not an important factor causing SCC. The main difference between the field samples and the laboratory standard solutions is that the laboratory solutions have a much lower pH (1.8 and 2.1) compared to those from the field (6.8 to 8.3). It is hypothesised that if micro-organisms were introduced in those regions where the samples were taken the local pH conditions could drop into a region where SCC does occur.

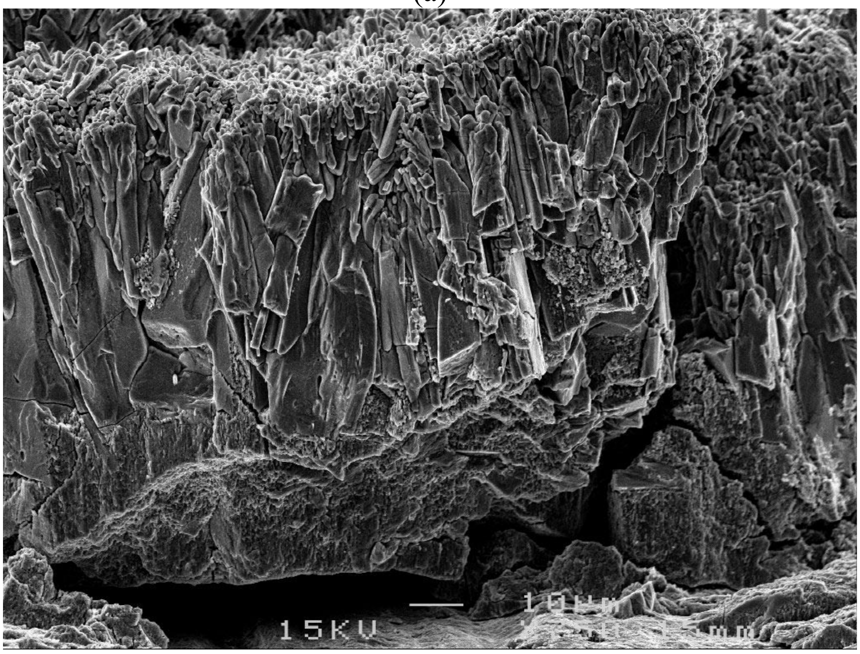
Galvanised Samples

Galvanised samples subjected to the standard LIST test displayed ductile overload, failing at 856 MPa. The surface appeared cracked and the zinc layer had flaked. However the core of the LIST sample (free of zinc) failed in a ductile manner. Ungalvanised samples failed at 950 MPa, indicating that the process of galvanising the LIST samples (i.e. the dipping into molten zinc) had the effect of lowering the strength of the steel. This was attributed to the heating of the steel to the temperature of the molten zinc causing a coarsening of the steel microstructure.

Figure 13 shows the fracture surface of the galvanised LIST sample and a magnified view of the fracture of the zinc layer. Fig. 13(a) shows the cracked zinc layer at the surface of the LIST specimen, whilst there was a ductile failure fracture surface for the steel. Fig. 13(b) presents a higher magnification view of the fracture of the surface zinc rich layer that had cracked and separated as the LIST sample deformed during the LIST test. The zinc layer did not only crack, but it also separated from the steel sample.



(a)



(b)

Fig. 13 Fracture surface of galvanised sample after LIST test
(a) overview. (b) detail of brittle fracture of zinc layer

Figure 14 summarises the controlled potential testing of galvanised samples. This showed that galvanised samples could be made to show SCC when a very negative potential was applied. This observation reinforced the identification of hydrogen embrittlement (HE) as the SCC mechanism.

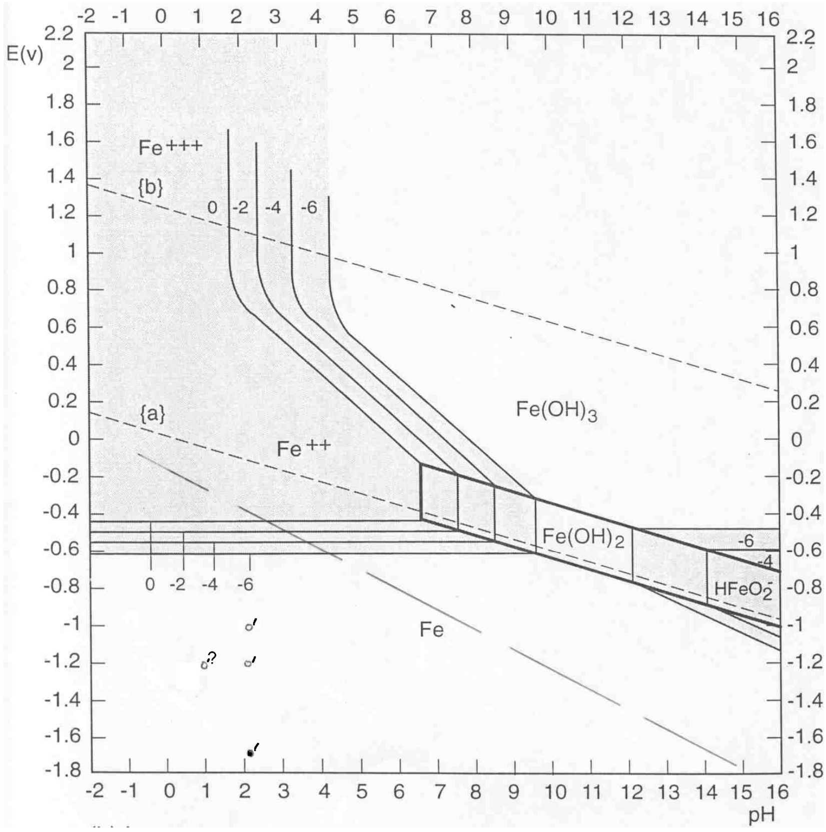


FIG. 14 - LIST test results for galvanised samples superimposed on an E-pH diagram. Full symbols indicated SCC, open symbols indicated ductile failure in the LIST test. Circles represented the sulphate solutions. Squares represented the chloride solutions. Symbols with flags indicated tests under potential control

DISCUSSION

A SCC velocity can be calculated from these tests using $v_{SCC} = d / t$, where d = stress corrosion crack size at the onset of fast fracture, and t = Laboratory Test (LT) duration. Typical values (for Steel *A*) are: $v_1 = 1{,}200 \times 10^{-6}$ m/(50,400 s) = 2.3×10^{-8} m/s; $v_2 = 500 \times 10^{-6}$ m/(19,140 s) = 2.6×10^{-8} m/s; to give an average value of $v_{av} = 2.5 \times 10^{-8}$ m/s. This crack velocity can be used to evaluate benefit provided by a material with a higher toughness. There is an increased lifetime between SCC onset and fast brittle fracture. If the fracture toughness is increased by a factor of two, the critical crack size in service is increased by a factor of $(\text{two})^2 = 4$, e.g. from say 2×10^{-3} m to 8×10^{-3} m. The increased life time $t = d/v = 6 \times 10^{-3} / 2.5 \times 10^{-8}$ = 240,000s = 2.7 days.

The threshold stress for Steel *A* rock bolt samples ~ 900 MPa, indicating that the loading causing SCC in service is due to a combination of the tensile load plus the bending load due to rock shear. This is consistent with the observation that rock bolts are typically bent after failure in service. This bending indicates a stress above the yield stress having been applied to cause the permanent deformation in bending, ie to cause permanent plastic deformation. The bending of the bolt has been attributed to shear in the rock strata. This leads to the issue of whether SCC could be prevented by a rock bolting strata design that prevented shear in the rock strata and thereby maintained the stress in the rock bolt below the threshold stress for SCC initiation.

CONCLUSIONS

- The incidence of SCC in rock bolts has not been quantified and its magnitude is not fully understood.
- A laboratory test has caused a tensile samples to fail in tension in a manner similar to the failure mode observed from service, namely slow SCC followed by fast brittle fracture. The laboratory tests involve subjecting a tensile sample to a linearly increasing stress at a slow applied stress rate whilst the specimen in exposed to a dilute sulphate solution of pH 2.1. Detailed fractography of SCC fracture features from the LT has shown that these fracture surfaces have the same features as fracture surfaces of service failures.
- The crack velocity measured from the laboratory tests indicates that the SCC lifetime is increased only marginally by the use of a rock bolt materials with a higher fracture toughness.
- The threshold stress for Steel *A* rock bolt samples is around 900 MPa, indicating that the loading causing SCC in service is due to a combination of the tensile load plus the bending load due to rock shear.
- The threshold stress for Steel *C* was in the order of 850 MPa and for Steel *B*, 830 MPa.
- Steel *D* bolts have experienced service failures, but Steel *D* rock bolt material did not show SCC in the laboratory test at the free corrosion potential in the standard sulphate solution, pH 2.1. This discrepancy indicates that (1) the susceptibility of Steel *D* should be explored at lower pH values, and (2) the influence of cold work on SCC should be studied.
- A hydrogen embrittlement mechanism for the SCC is indicated by the particular restricted range of conditions for which SCC occurs in the laboratory.
- In particular, SCC only occurs in the laboratory for the restricted range of environmental conditions corresponding to acid conditions at the open circuit potential (pH ~ 2.1 or more acid) or at very negative applied electrochemical potentials corresponding to copious hydrogen evolution at the steel surface. This is consistent with reports from the USA indicating rock bolt failure due to the presence of H_2S in the mine atmosphere. Similarly, this is consistent with bacterial corrosion on the rock bolt surface during service producing acid conditions leading to SCC.
- Water chemistry analyses has been carried out for a number of Australian mines including one coal mines. The water in all cases was neutral, with the pH ranging from 6.84 to 8.32.. The UQ laboratory test indicates that SCC would not occur in any of these neutral mine waters. This does indeed suggest that SCC in a coal mine would be caused by bacterial corrosion locally decreasing the mine water pH down to a pH ~ 2.1.
- Galvanised samples did not show SCC in the laboratory test at the free corrosion potential in the standard sulphate solution at pH 2.1, but SCC could be induced in the pH 2.1 solution at a more negative potential. A galvanised coating has a short life due to general corrosion in the pH 2.1 solution.
- Further research is required.

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