Volume 9 Paper 3

The Effect of Surface Films on Cathodic Protection

S.S. Leeds and R.A. Cottis

Corrosion and Protection Centre, School of Materials, University of Manchester, P.O. Box 88, Manchester M60 1QD, UK,
s.leeds@postgrad.manchester.ac.uk, Bob.Cottis@manchester.ac.uk

Abstract

Electrochemical studies, surface chemistry and analytical analysis have been used to establish what is actually happening when steel samples are cathodically protected at a series of potentials in aerated pure 3.5% NaCl solution. It was found that under freely corroding conditions of –671 mV (Ag/AgCl/3.5 % NaCl) at day 30, the corrosion rate was 0.752 mm/yr, whilst a potentiostatic –650 mV and a battery driven CP specimens had corrosion rates of 0.089 and 0.099 mm/yr respectively. This was an 8 times reduction in corrosion rate compared to the freely corroding specimen and shows that the application of even a small amount of Cathodic Protection (CP) produces a significant reduction in corrosion. However it was not enough to halt corrosion completely. Films were observed to form and these significantly affected the current demand and corrosion rate of the test specimens. The specimen with the least corrosion rate and most coherent film was at –1300 mV (Ag/AgCl/3.5% NaCl), with a corrosion rate of 0.00396 mm/yr.

Keywords: Cathodic Protection, electrochemistry, potentiostatic method, weight loss measurement.

Introduction

Corrosion is a significant and undesirable spontaneous electrochemical process that costs billions of pounds each year in terms of replacement and maintenance costs. To mitigate corrosion one process involves the application of Cathodic Protection (CP) to buried or immersed metallic structures. Various international standards/specifications set out the application and control of CP but do not give consideration to the electrochemistry and surface chemistry of the metallic structure/electrolyte interface [1] [2] [3] [4]. The ISO Standard [5] is more definitive, describing CP in terms of what is actually happening electrochemically at the metal/electrolyte interface of a cathodically polarized structure. It defines CP as, "Electrochemical Protection achieved by decreasing the structure potential to a level whereby the corrosion rate of metal is significantly reduced" but still fails to provide information regarding the nature and role of the interface or influence of surface films that form as a direct cause of the cathodic protection process. Furthermore, it ignores the changes in the chemistry of the environment that are induced by the applied cathodic current.

For immersed structures the −800 to −1050 mV (Ag/AgCl) polarised potential is assumed by industry [2] to give protection under aerobic conditions. For buried structures CP criteria such as the −850 mV (Cu/CuSO$_4$) OFF or the −100 mV shift [6] are used. These criteria are based on the assumption that if a certain amount of current is applied to the metallic structure of interest and it meets the protection criteria then it must be protected. However, this is not always the case; for example some pipelines having reportedly met a protection criteria such as −850 mV (Cu/CuSO$_4$) OFF, have been found through metal loss identification to be not fully protected some years later [6]. The conditions for protection are more complex than simply applying more and more current and measuring the resultant potential to achieve a set criterion. What is important is knowing what is happening electrochemically at the metal/electrolyte interface and adjusting the criterion accordingly to achieve an acceptable corrosion rate.

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Electrochemical Analysis of CP

When applying CP for the first time to a structure it is well known that a higher current and less negative structure/electrolyte potential is observed. Over a period of a week or more the current decreases and the voltage becomes more negative until it reaches a stable value determined by the amount of CP applied. Within the CP industry it is stated that the surface has passivated, but does the industry really understand what this means? In order to understand CP and the nature of the "passivity" it is important to know what is actually happening electrochemically at the surface of the metallic structure when CP is applied. The electrode potential/electrolyte pH diagram, the Pourbaix diagram, is used to explain the anodic and cathodic behaviour of steel in electrolytes of different pH and categorizes the domains of corrosion, passivation and immunity, see Figure 1.
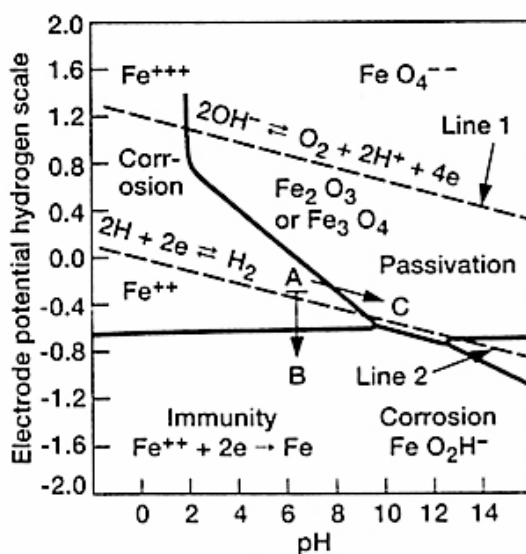

Figure 1: Pourbaix – Potential-pH Equilibrium Diagram for Iron. Lines 1 and 2 Correspond to the Oxygen and Hydrogen Equilibria [7]

As CP is increased and the potential becomes more negative, the surface electrochemistry will move towards the region of immunity, position B on the Pourbaix diagram, where steel is more thermodynamically stable and corrosion does not occur. Over time the

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surface pH will increase as a result of the generation of alkalinity due to the reduction of dissolved oxygen [7] [8] .

$$\frac{1}{2}O_2 + H_2O + 2e^- \rightarrow 2OH^-$$ Cathodic Reaction (1)
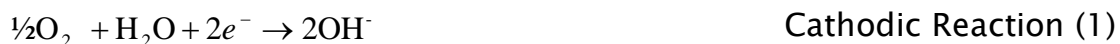
The effect of applying CP is therefore to move the potential in both a negative direction and towards a higher pH to the conditions found in the passive region at position C on the Pourbaix diagram which may be above the potential required for immunity.

In cases where the potential is more negative [6] [9], a second cathodic reaction occurs:

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$ Cathodic Reaction (2)
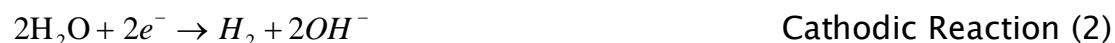
In both cathodic reactions, hydroxyl ions are being formed which bring about an increase in the electrolyte pH adjacent to the metal surface and pushing the surface electrochemistry into the passive domain of the Pourbaix diagram where films are formed.

Whilst the positions of the domain boundaries of the Pourbaix diagram are variable depending upon ion concentration, temperature, pressure etc, an interpretation of the Pourbaix Diagram suggests that the domain potential (line 2, Figure1) for the decomposition of water has a limiting potential for effective CP and that in practice the real structure potential seldom reaches the domain of immunity unless the limiting current density for water decomposition is reached which will never happen in sea water. Increasing the applied potential between the anode and cathode, a common practice to improve the distribution of CP, only leads to more rapid generation of alkali with detrimental effects of more rapid coating decay [10].

Studies of the Effect of Cathodic Protection on Surface Films

The initial cathodic reaction involving the reduction of dissolved oxygen is limited by the fact that only small amounts of oxygen (typically 8ppm) [11] can dissolve in water so for an increase in CP the potential has to become more cathodic leading to the decomposition of water for which under most conditions the limiting current density is never reached. Alkali is generated by both cathodic reactions. In

the process of applying cathodic protection there will be a corrosion reaction that is being progressively suppressed as the potential is made more cathodic. Corrosion product is formed at a slower and slower rate as the CP becomes more effective. What happens to this corrosion product? It is believed that, like the formation of protective calcareous deposits when steel is polarized in seawater [12] [13], there is also a protective film formed from this iron corrosion product. The combined effect is to reduce the area of bare steel exposed to the electrolyte. To date studies of the role of surface films in CP have concentrated on the build up of calcareous deposits in seawater largely ignoring any effects of true passive films.

Previous work by Rodrigo [14] showed that the CP current density for both 3 % NaCl solution and natural seawater was a function of dissolved oxygen concentration. Under conditions of air saturation, approximately 7 mg/L O_2, 50 % less current was required to polarize steel in natural seawater than in 3 % NaCl, he suggested that the calcareous deposits were the main influencing factor.

Hartt et al. [15], studying calcareous deposits, found that extended exposure time is required to achieve steady state, which suggests that potential and current should be treated as individual variables. This can be seen when calcareous deposits are formed under certain conditions promoting different current density decay responses. Hartt et al., like Wolfson [16], found that long term, the current density did not necessarily correlate with the magnitude of polarization. Hartt et al. thought that the magnitude of current density should be proportional to the magnitude of the cathodic polarization and that the current density decay for the initial 10 hour exposure period of their specimens should be the same for all three potentials (–0.78 V, 0.93 V and –1.03 V (SCE)). However, they found that the decay rate for the –1.03 V (SCE) specimen increased, resulting in current densities that after 30 hours were less than for the –0.93 V (SCE) specimen. They suggested that this must be due to some property of the calcareous deposit formed at the more negative potential that upset the expected potential /current density relationship. It was thought that this could be due to a greater resistivity or specific chemistry of the –1.03 V (SCE) deposits/films.

Humble [17] found that calcareous deposits/films formed at lower current densities were more permanent than those formed at higher current densities. Cox [18] further found that no corrosion was apparent when the cathodic current density was in the range of 0.07 to 0.3 mA/cm². Thus, the surface films formed under these conditions provided enough protection even during the time of the experiment when the specimens were out of the water. Further this was argued as being proof that protection was attributed to the chemistry of the calcareous films formed and that the reduced protection of films was attributed to current densities that were greater than 0.3 mA/cm² and were due to the poor coating properties of $Mg(OH)_2$. None of these authors mention the impact of any iron corrosion product films. The observations reported in this paper deal with studies of the iron corrosion product films on mild steel using as electrolyte an aerated pure 3.5 % sodium chloride (NaCl), pH 7 in order to eliminate possible calcareous deposit interference.

EXPERIMENTAL PROCEDURE

Materials

Tests were carried out on bright mild steel specimens manufactured according to BS. 970 Pt3, 1991, having a composition given in Table 1.

Table 1: Composition of the Mild Steel used in the Experimental Work

C	Si	Mn	P	S
0.120 %	0.180 %	0.850 %	0.026 %	0.031 %

Each specimen was cylindrical in shape, 0.6 cm diameter and 4 cm in length. The specimens were prepared by abrading with 1200 grit silicon carbide paper, then cleaned and degreased in acetone and preweighed.

The test electrolyte was a solution of 3.5 % w/v Analar grade sodium chloride (NaCl) in deionised water.

Test Cell

To separate anode and cathode reactions and to limit any effects of chlorine from the anodic reaction a test rig was constructed such that it had concentric inner and outer cells. The plastic walled inner cell had holes in the bottom covered with an agar gel made up with 3.5 % NaCl solution to allow for ionic exchange and glass beads to weigh it down. This inner cell held the test specimen (WE), Ag/AgCl/3.5% NaCl reference electrode and air bubbler. The reference electrode was constructed out of a 40 cm, 0.6 cm PVC tube. This was filled with agar, in 3.5 % NaCl and an activated Ag/AgCl electrode, calibrated against a standard Ag/AgCl electrode. The reference electrode was constructed in this way to limit the migration of silver ions onto the steel specimens because Scanning Electron Microscopy (SEM) analysis of earlier films on the steel specimens, found silver deposited when the RE were constructed in a typical Luggin capillary design or if the Ag/AgCl reference electrode was directly in the electrolyte solution. Graphite anodes were arranged in a triangular configuration to allow for an even spread of the current, see Figure 2.
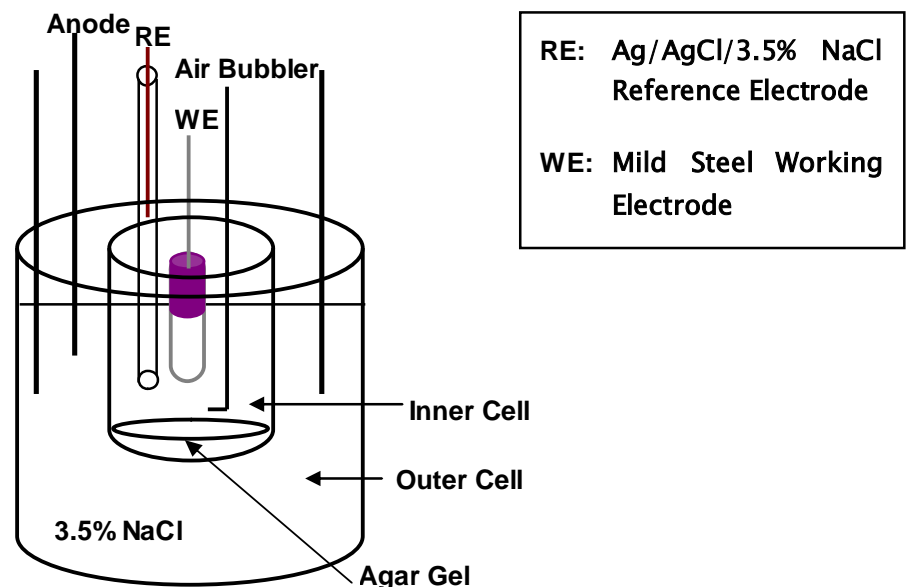

RE:	Ag/AgCl/3.5% NaCl Reference Electrode
WE:	Mild Steel Working Electrode

Figure 2: Test cell configuration

Potentiostatic and Weight Loss Methods

Eight test specimens were investigated and each was connected to a separate potentiostat. The potentials of the specimens were controlled in the range −650 to −1300 mV (Ag/AgCl/3.5% NaCl). A further specimen was exposed to freely corroding conditions (initially −526 mV, moving to approximately −671 mV (Ag/AgCl/3.5% NaCl) over a period of 720 hrs). An additional specimen was connected to a battery and variable resistor and set initially to a potential of −650 mV (Ag/AgCl/3.5% NaCl) with no permanent reference electrode and used primarily as a control to check for silver contamination when carrying out SEM analysis. The specimens were exposed for 30 days in pure 3.5 % aerated NaCl solution. The current for each specimen was recorded automatically every 15 minutes. Specimens were then cleaned according to the ASTM G1 specification [19]. A second unexposed control specimen was also cleaned to correct for any cleaning procedure weight loss and resulted in a correction factor of 0.0002 mm/yr.

Film Preparation for SEM Analysis

At the end of the exposure time sections of the films formed were detached and transferred onto double sided carbon impregnated tape then placed in a vacuum overnight at 10^{-2} Torr. These films were then analyzed in the SEM in order to identify the composition of the films. The films on the metal specimens were also examined directly for comparison with the detached films.

RESULTS

Specimen Morphology

Observations were made to log any changes in specimen morphology. The freely corroding specimen (−671 mV (Ag/AgCl/3.5% NaCl) developed a blue/green deposit by the end of day one, suggesting that this was hydrated magnetite, $Fe_3O_4.H_2O$, an unstable form of rust. This soon formed red brown flaky rust, hydrated ferric oxide. The −650 mV potentiostatic specimen also formed red brown flaky rust, showing that the initial application of CP does not stop the onset of corrosion.

The specimens with potentials ranging from –700, –800, –850, –900, –950, –1000 and –1300 mV had progressively varying degrees of a grey film forming slowly on the metal through out the 30 days, with the more negative potentials forming a black film.

Analysis of Films

Samples of the films were collected and analyzed under SEM. It was very difficult to take samples of the film as they are very thin and adherent. Three or four different regions of the film were analyzed to get an overall view of the composition. The films on the metal specimens were also examined directly to observe any differences between the sections of film taken and the film directly on the specimens. Table 2 details the elemental composition of the films. Figures 3 to 8 show the film structure. In each case sections of the film and the film on the specimen are viewed. It can be seen that the more negative the potential then the more uniform the film that is formed, which is in agreement with the observations made during the experiment. From the freely corroding to the –800 mV specimen the films formed are looser and less coherent. From –850 mV to –1300 mV the films were more uniform. It can be seen that the films form a compact structure, with a crystalline appearance. It is thought that the film is composed of magnetite and probably accounts for the reduction in corrosion as the specimen potentials are made more negative.

Table 2: SEM Analysis

Freely Corroding to -1300mV Specimens

	Specimen Potential									
	Freely Corroding	Battery	-650 mV	-700 mV	-800 mV	-850 mV	-900 mV	-950 mV	-1000 mV	-1300 mV
Element	A	A	A	A	A	A	A	A	A	A
	%	%	%	%	%	%	%	%	%	%
C	15.64	16.79	44.72	-	-	29.36	16.0	-	46.27	55.35
O	26.81	35.92	15.67	34.13	45.37	35.51	54.05	5.03	19.32	9.67
Na	14.37	9.48	13.93	22.85	9.57	5.97	5.45	8.22	4.79	5.43
Si	0.30	0.93	0.93	4.07	6.79	4.23	1.04	4.58	0.12	0.25
S	1.60	0.21	0.30	0.42	1.12	0.69	0.09	-	7.02	0.49
Cl	13.27	7.83	15.79	21.5	6.68	4.17	1.31	4.12	4.95	2.44
Cr	0.46*	0.29	-	-	0.28*	0.18*	0.04*	-	1.16	0.05*
Mn	1.98	0.61	-	0.37	1.02	0.66	0.56	1.38	6.75	0.16
Fe	26.30	28.25	8.46	14.76	28.94	19.03	39.3	30.54	24.56	21.78
Ca	-	-	0.20	0.83	0.26	0.17	-	0.85	-	-
K	-	-	0.093	0.25*	-	-	-	-	-	-
P	-	-		0.69	-	-	0.01	-	0.17	0.25
Zn	-	-		1.33	-	-	-	-	-	-
Cu	-	-		-	-	0.17*	--	-	-	-

A: % Atomic, * : <2 Sigma, Spectrum taken at 15kV, System resolution is 62

SEM Micrographs

Micrographs for each specimen were taken to view the morphology of the films formed at a particular potential (Figure 3 to 8).
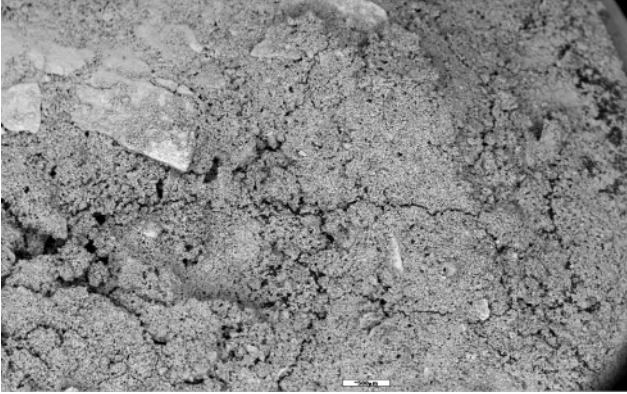

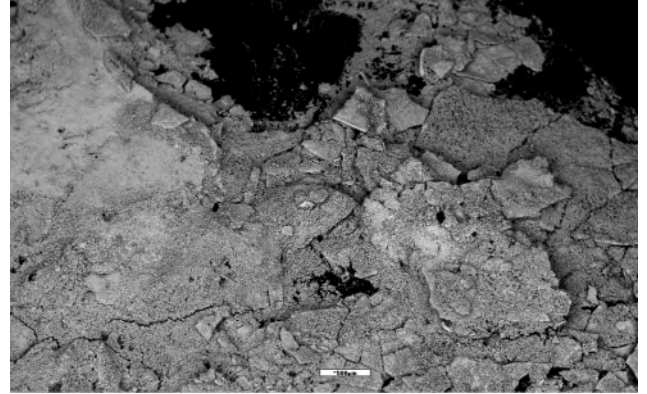**Figure 3: SEM (BSE) of the Freely Corroding Specimen (size bar 500 μm)**

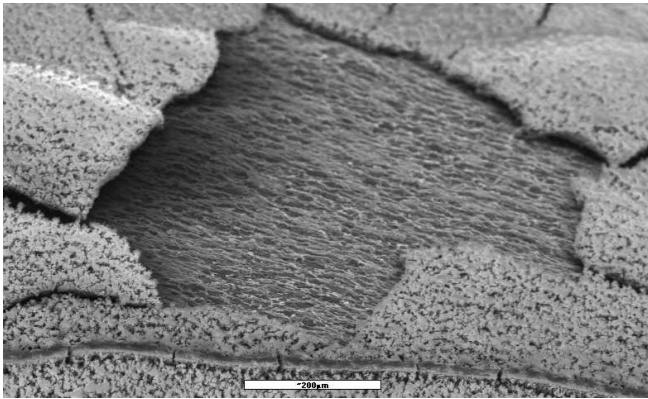**Figure 4: SEM (BSE) of the Battery Specimen (size bar 500 μm)**
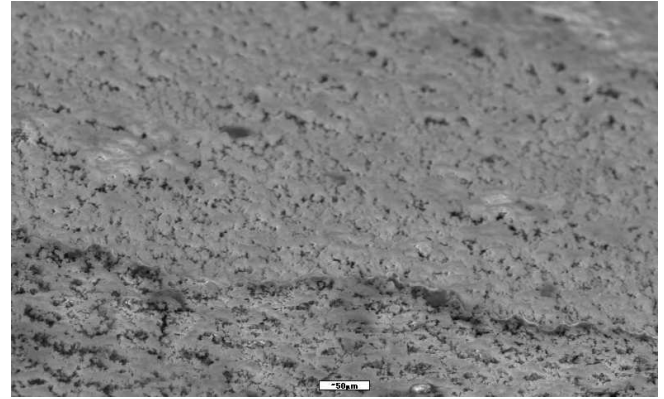

Figure 5: SEM (SE) of the −800 mV Specimen (size bar 200 μm)

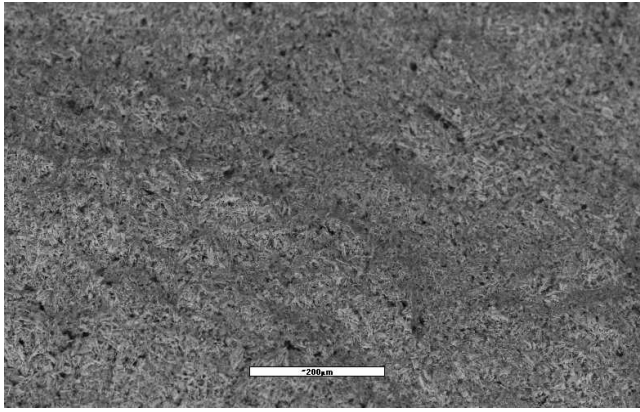**Figure 6: SEM (SE) of the −850 mV Specimen (size bar 50 μm)**

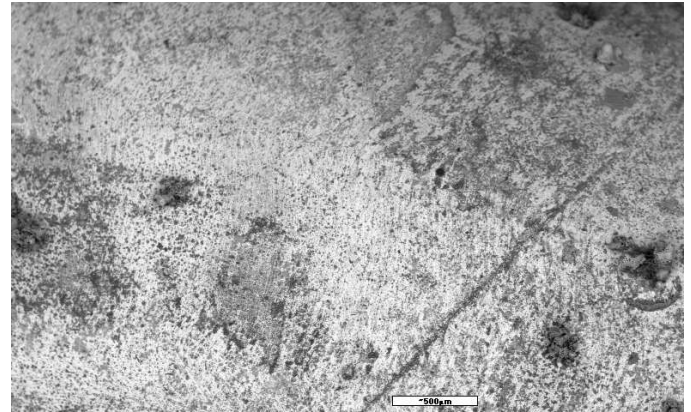**Figure 7: SEM (BSE) of the –1000 mV Specimen (size bar 200 μm)**   **Figure 8: SEM (SE) of the –1300 mV Specimen (size bar 200 μm)**

Potentiostatic Weight Loss Results

In order to establish the optimum CP potential, namely the potential showing the lowest corrosion rate, potentiostatic weight loss measurements were made. The weight loss for each specimen was recorded and converted into the mean corrosion rate, which is expressed as mm per year (mm/yr), refer to Table 3. It was found that under freely corroding conditions, (not connected to any CP), with a potential ranging from –526 mV to –671 mV (Ag/AgCl/3.5% NaCl) there was a corrosion rate of0.752 mm/yr. The –650 mV CP specimen and the battery sample had corrosion rates of 0.089 and 0.099 mm/yr respectively, an 8 times reduction in corrosion rate compared to the freely corroding specimen. The application of a small amount of CP produces a significant reduction in corrosion but not enough to achieve industry accepted levels. As the potential was made more negative the corrosion rate increases between –650 mV and –800 mV (Ag/AgCl/3.5% NaCl) then decreases significantly. At over protection values of –1300 mV (Ag/AgCl/3.5% NaCl) the corrosion rate was found to be 0.00395 mm/yr, suggesting that beyond a certain potential the more negative the potential the lower the corrosion rate. Note that this contrasts with the work of Batt [20], which suggested that the corrosion rate increased at very negative potentials.

The corrosion rate plotted against specimen potential is given in Figures 9. It is commonly considered by industry that a corrosion rate of 0.001mm/yr [20] [21] represents an acceptable rate of metal loss . On the basis of the data presented it suggests that the potentials in excess of −1300 mV (Ag/AgCl/3.5% NaCl) would be needed to achieve this industry level. However, the very negative potentials can give rise to additional problems dealt with later.

Table 3: Potentiostatic Weight Loss Measurement Data

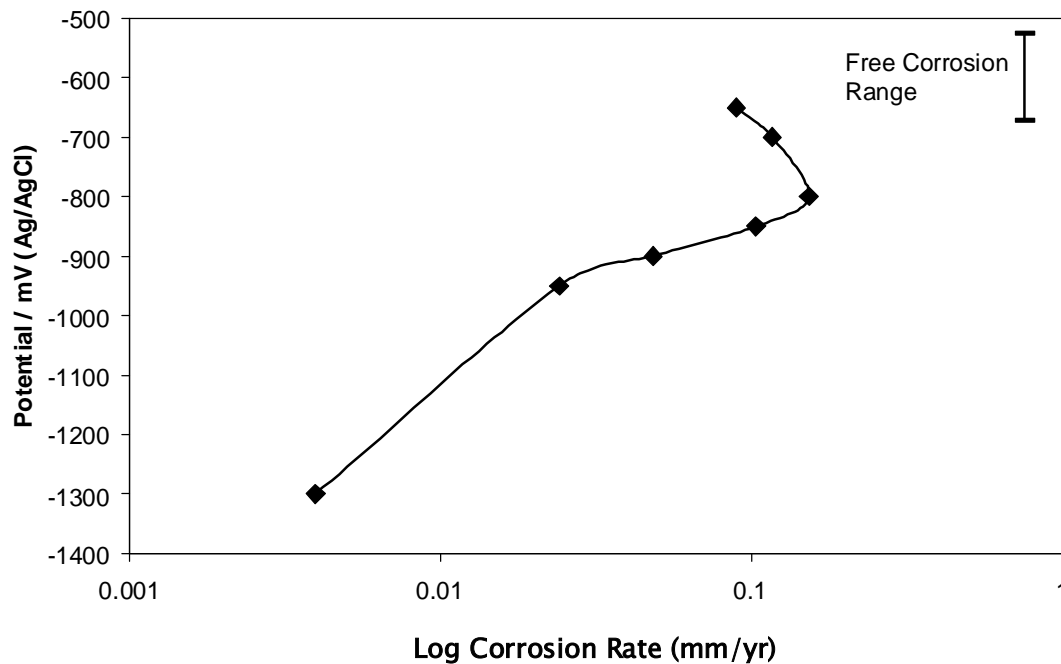| Specimen	Weight Loss (g)	Current Density (mA/cm^2) at Day 30	Corrosion Rate (mm/yr)
Unexposed Control	0.0009	–	0.0002
Freely Corroding	0.38	–	0.752
Battery Controlled	0.05	–	0.099
−650mV	0.045	0.0742	0.089
−700mV	0.059	0.0473	0.116
−800mV	0.077	0.179	0.152
−850mV	0.052	0.193	0.103
−900mV	0.024	0.253	0.048
−950mV	0.012	0.129	0.024
−1300mV	0.002	0.914	0.00396

Figure 9: Corrosion Rate Plotted Against Specimen Potential at Day 30

The current density plotted against time to determine the trend in the amount of current required by a CP system operated at specific potentials are shown in Figure 9 to Figure 12, for –650, –950 and –1300 mV specimens respectively. At –650 mV (Ag/AgCl/3.5% NaCl) the specimen showed a continued decrease in current required, but the surface of the specimen was covered with a loose flakey red brown corrosion product (but note that the average corrosion rate over the exposure period was less than that for the free corrosion case, even though the final potential of the latter was more negative than –650 mV (Ag/AgCl/3.5% NaCl)). At –950 mV (Ag/AgCl/3.5% NaCl) after an initial fall in current to day 3 the current demand stayed relatively constant. The fluctuations in current are thought to be due to the break away of some parts of the looser black film easily dislodged by the agitated electrolyte as observed over the course of 30 days. At –1300 mV (Ag/AgCl/3.5% NaCl) after the big initial drop in current up to day 3 the current remained steadier than for the –950 mV specimen. The –1300 mV specimen was covered in a uniform black film (subsequently shown to be magnetite). In practice the current to which the specimens settle represent the current needed to maintain an operational CP system.
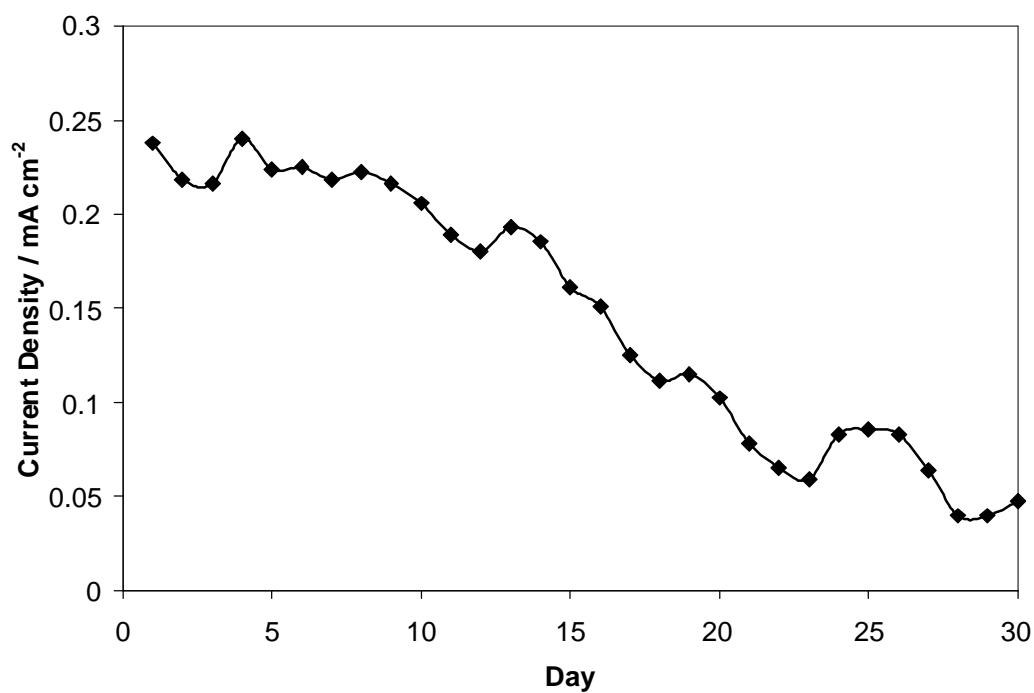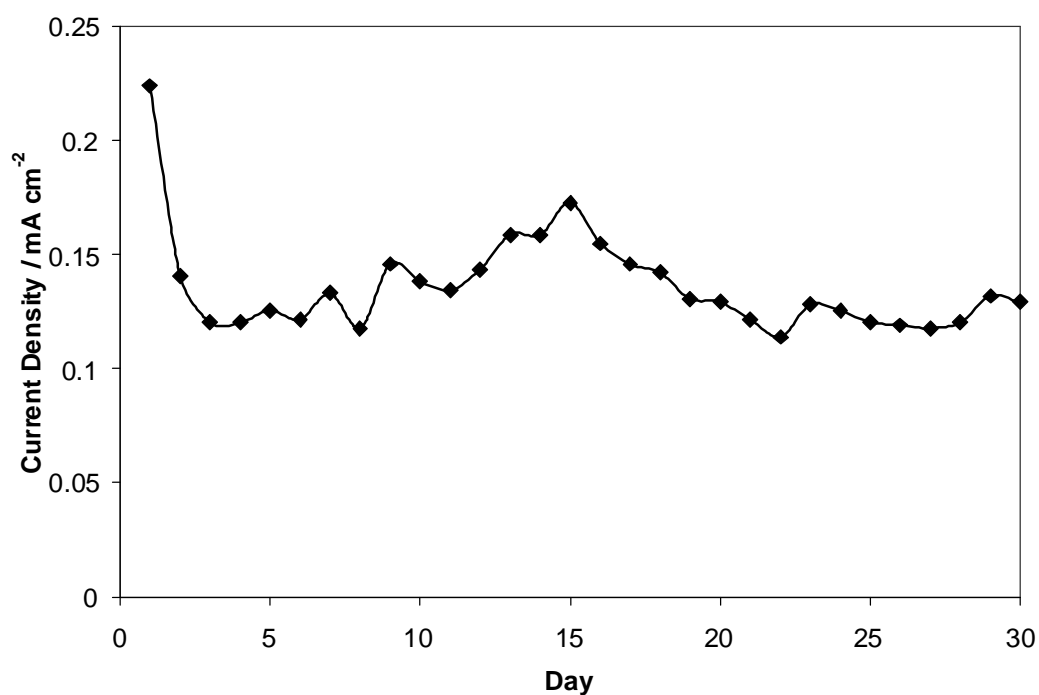
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Figure 10: Current Density versus Time, −650 mV (Ag/AgCl)

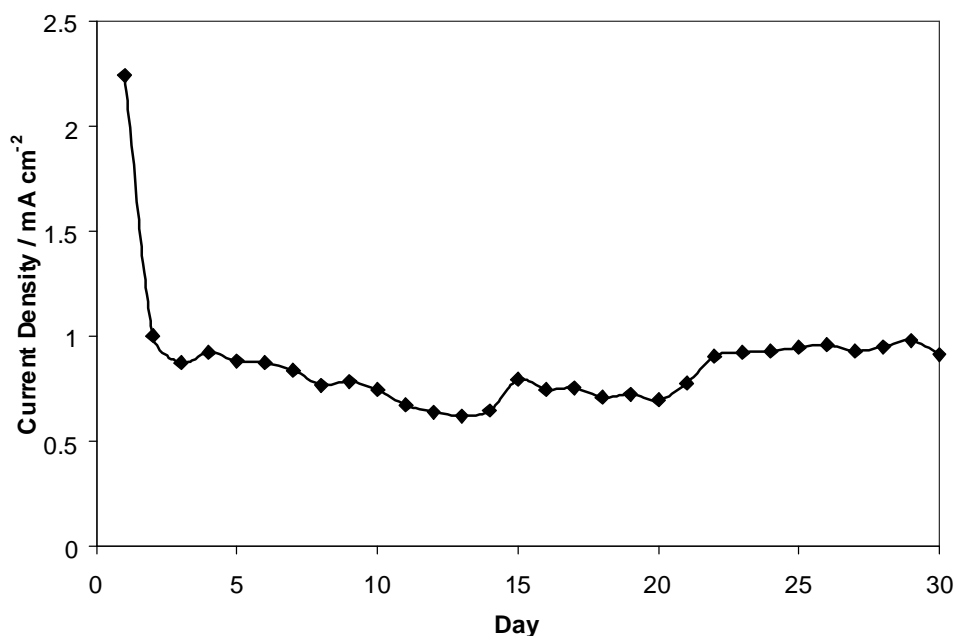Figure 11: Current Density versus Time, −950 mV (Ag/AgCl)

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Figure 12: Current Density versus Time, −1300 mV (Ag/AgCl)

pH Measurements

After 30 days the pH of the solution in the specimen compartment was measured, see Table 4. A general increase in pH was noted as the test potential became more negative.

Table 4: pH of Solution after Cathodically Polarising for 30 Days

Specimen	pH
3.5% NaCl only	7.02
No CP	8.21
Battery Specimen	8.33
−650mV	8.81
−700mV	7.65
−800mV	9.51
−850mV	8.98
−900mV	10.05
−950mV	9.00
−1000mV	9.41
−1300mV	12.74

Interpreting data from Tables 3 and 4 and using the Pourbaix Diagram, it can be found that the freely corroding, the –650 mV and the battery specimens lie on the border between corrosion and immunity, the formation of red brown rust, hydrated $Fe_2O_3.H_2O$ suggesting that these specimens lie more in the corrosion region, allowing corrosion to continue throughout the 30 days, which can be confirmed by the weight loss observed. The –800, –900, –1000 mV specimen solutions are increasing in alkalinity. The –1300 mV specimen, at a pH of 12.7 is on the border between immunity and corrosion, however in this case there is not much corrosion occurring which must be due to the protective film formed at the more negative potentials, as can be confirmed by the limited weight loss observed. The problem with the very negative potentials is the generation of excessive alkali, which will lead to more rapid degradation of organic protective coatings and can also lead to the build up of carbonate/bicarbonate environments, one of the requirements for the development of Stress Corrosion Cracking failures.

DISCUSSION

The experimental work carried out has enabled a better understanding of what is occurring at the metal/electrolyte interface when it is cathodically polarized. The work confirmed that a cathode definitely becomes covered with a protective film which could not be calcareous deposit in these experiments. This film at more negative potentials is thought to be magnetite and is grown only by the effect of the CP on the steel surface. At less negative potentials the magnetite film can become less coherent. The best film was at the more negative potentials where water decomposition is giving rise to alkali and an increase in surface pH, which, for reasons mentioned earlier, is not desirable. Over protection gives an excessively alkaline pH and wastes current and contributes very little to effective CP. The results suggest that some decomposition of water is very important in order to generate the best possible film, but this should be limited to reduce unwanted current.

Corrosion can be halted if an adequate film can be formed on the metal during cathodic polarization and the data suggest that a

minimum of 3 days is required for the film to start influencing what is happening at the steel surface. The work poses a fundamental question, what mechanism is providing the cathodic protection? Is protection primarily the result of lowering the potential into or towards the region of thermodynamic immunity, or is it the result of passivating the steel by the formation of a film in the alkaline environment resulting from the excess cathodic current or is it a combined effect? Further experimentation is planned to interpret better the fundamental mechanisms by which CP works in minimising corrosion of mild steel.

CONCLUSIONS

- Films were progressively formed over a 3 day period as a direct result of cathodically polarizing metal specimens.
- Longer times are needed to form coherent films.
- X-Ray Diffraction has to be carried out to prove what the films are composed of. It is thought that it is magnetite.
- Application of a small amount of CP produced a significant reduction in corrosion, which can be seen by the lower corrosion rate of the -650 mV specimen compared to the freely corroding specimen. However, -650 mV was insufficient to prevent corrosion from still occurring.
- Industry states that to prevent corrosion of metallic structure a potential of -950 mV (Ag/AgCl/seawater) should be used in seawater. In the experimental conditions used in this study, corrosion rates above 0.001mm/yr were still observed.
- Making the potential more negative resulted in a lower corrosion rate, with a potential of -1300 mV meeting an acceptable corrosion rate of 0.001mm/yr. Further work has to be carried out to confirm this result.
- Modelling work is being carried out to try and understand theoretically what is occurring at the electrolyte/metal interface when cathodically polarised.

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