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Passivity breakdown of carbon steel in hot potassium carbonate solutions

S. J. Harjac · A. Atrens · C. J. Moss · V. Linton

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Abstract This research studied passivity breakdown of carbon steel in conditions typical of hot potassium carbonate (HPC) acid gas processing plants. The pitting potential, evaluated from polarisation curves, indicated resistance to pitting increased with increasing equivalent carbonate concentration. The pitting potential was much more positive than the free corrosion potential for uninhibited concentrated carbonate solutions with pH values of 8.5 to 9.5. Therefore pitting is not an issue in these solutions. However, the carbon steel was susceptible to pitting corrosion in the dilute 1.0% bicarbonate solution of pH 8.0 for chloride concentrations in excess of 0.5 wt%. The critical chloride concentration was considerably above the maximum concentration of 0.1 wt% recorded in a typical HPC plant. Consequently chlorides should not usually be of concern to plant integrity.

Introduction

HPC plant overview

Raw natural gas, such as that from the Cooper Basin in central Australia, contains typically 20 mol% CO_2 and up to 20 ppm H_2S. The H_2S and the "acid gas" CO_2 are removed to produce sales quality gas with a CO_2 content below 2 mol% and a H_2S content below 2 ppm. The selective removal of CO_2 can be carried out by a variety of chemical processes, several of which utilise hot alkaline solutions for absorption. One of these is the Benfield process, which is used in more than 600 plants worldwide. The Benfield process is based on an aqueous solution of potassium carbonate and bicarbonate, with potassium vanadate as an anodic inhibitor and with an amine as an accelerant. The typical solution concentration is 27 eqv. wt% K_2CO_3. The carbonate concentration is expressed as equivalent weight percent potassium carbonate, which is calculated via the following expression.

$$C_{\mathrm{eqv\cdot Wt\%K_2CO_3}} = C_{\mathrm{K_2CO_3}} + 0.691 C_{\mathrm{KHCO_3}} \qquad (1)$$

where $C_{K_2CO_3}$ is the concentration of K_2CO_3 [in wt%] and C_{KHCO3} is the concentration of $KHCO_3$ [in wt%]. The H_2S in the natural gas is oxidised to thiosulphate and sulphate so that the solution may accumulate concentrations of 20 wt% thiosulphate and 1.5 wt% sulphate. The HPC plants comprise a closed circuit, consisting of an absorber vessel, a regenerator vessel and associated connecting pipes, pumps and valves. The absorber vessel has typically a pressure of approximately 70 bar. The upward flowing raw natural gas containing CO_2 and H_2S contacts the down flowing potassium carbonate solution in a counter current

S. J. Harjac · A. Atrens (✉)
Materials, School of Engineering, The University of Queensland,
Brisbane, QLD 4072, Australia
e-mail: a.atrens@minmet.uq.edu.au

S. J. Harjac
e-mail: s.harjac@minmet.uq.edu.au

C. J. Moss
Santos Ltd, Santos House, 91 King William Street, Adelaide
5000, South Australia
e-mail: harry.moss@santos.com

V. Linton
School of Mechanical Engineering, The University of Adelaide,
Adelaide 5005, South Australia
e-mail: valerie.linton@adelaide.edu.au

flow. The potassium carbonate absorbs CO_2 and is converted to potassium bicarbonate:

$$K_2CO_3(aq) + H_2O(1) + CO_2(aq) \leftrightarrow 2KHCO_3(aq) \quad (2)$$

The Fraction Conversion, F_C, is expressed as,

$$F_c = 1 - (C_{K_2CO_3}/C_{eqv \cdot Wt\% K_2CO_3}) \quad (3)$$

$F_C = 0.0$ for pure K_2CO_3 and $F_C = 1.0$ for pure $KHCO_3$. Maximum absorption of CO_2 has occurred by the time the solution has reached the bottom of the absorber tower. This solution is called the rich solution and has a typical F_C of 0.9. The rich solution leaving the absorber undergoes a pressure reduction and enters the regenerator tower at close to atmospheric pressure. The CO_2 concentration in solution decreases with decreased partial pressure of CO_2. CO_2 is further stripped from the solution by contact of the solution with steam in the regenerator vessel. The released CO_2 is vented from the top of the regenerator tower to the atmosphere. The regenerated carbonate solution, known as the lean solution, with a typical F_C of 0.4, is recycled back to the top of the absorber vessel. The solution circuit is a closed loop, except for blow down to manage solids loading. Each closed loop is called a train.

Prior to commencement of processing raw natural gas, each train (including the absorber vessel) is subject to a passivation procedure involving circulating an inhibited solution. The duration may vary from 4 h to 5 days. The passivation process aims to provide corrosion protection via the formation of a protective magnetite layer on the steel surface.

The solutions in the absorber towers can be corrosive. In the upper region of the absorber tower, solution pH is expected to be around 9.0, whereas a solution pH of 8.0 is expected in the bottom of the absorber. At such high partial pressures of CO_2, a surface layer of iron sulphide (pyrite) might produce a low corrosion rate if the ratio of $H_2S:CO_2$ is below 1:20 [1]. However magnetite formation is normally the aim of anodic inhibition in HPC plant. Magnetite is thought to offer more reliable protection since it is thin and tightly adherent, as opposed to pyrite, which can be present in multiple layers that are susceptible to spalling and flaking. Nevertheless, magnetite passivity may be vulnerable to process conditions such as loss of inhibitor, flow accelerated corrosion and chemical breakdown in the presence of aggressive ions. Of particular concern is the susceptibility of the magnetite film to pitting corrosion due to aggressive ions like chlorides.

Service Experience

Pitting is typically the form of the degradation of absorber towers [2–6]. Many operators [6] have reported localised and unpredictable corrosion. High corrosion rates have been reported of up to 2,300 mm/year, which are significantly greater than the expected rate of passive dissolution [7]. This service experience suggest that anodic inhibition may be susceptible to adverse operating conditions that shift the operating conditions outside the conditions for passivity [8]. Although considerable empirical knowledge exists based on operational experience, understanding of HPC corrosion under controlled laboratory conditions is needed to provide a technical basis for improved HPC corrosion performance.

HPC plant operators have attributed pitting to inadequate inhibitor concentration at the steel surface, the presence of aggressive ions such as chlorides, erosion due to solution turbulence and high solids loading, mechanical scraping due to loose hardware and the erosive effect of froth bubbles imploding on the passivated surface [2]. One particularly important factor may be the chloride concentration. Chlorides in HPC solutions stem from make-up water. The measured minimum, maximum and nominal plant chloride concentrations in a typical plant have been reported to be 0.004 wt%, 0.08 wt% and 0.015 wt% respectively [9]. Despite the presence of chlorides in HPC plant solutions, and despite the known action of chlorides in inducing pitting, there is no guideline which defines the threshold chloride concentration, above which depassivation may be initiated. It would indeed be useful to know the critical chloride concentration that would lead to depassivation. Similarly, it is important to understand the influence of the other aggressive ions: thiosulphates and sulphates.

Aims

The aim was to understand the threshold chloride concentration in the absorber tower solutions that would cause pitting of the carbon steel. Solution parameters explored included (1) chloride concentration, (2) pH, (3) presence of the other aggressive species, sulphates and thiosulphates and (4) presence of inhibitor. An understanding of the limits of chloride concentration may be beneficial to plant personnel who would be provided a better understanding of HPC plant corrosion. This understanding can be utilised in developing guidelines for maintaining the effectiveness of the anodic inhibition system.

Experimental procedure

This article investigated the influence on pitting susceptibility of carbon steel of chloride concentration, pH and presence of other aggressive ions. The polished steel specimens were immersed in inhibitor free carbonate–

Table 1 Base composition of the test solutions and their measured pH values

Solution	Measured pH	K_2CO_3 [wt%]	$KHCO_3$ [wt%]
A (lean)	9.5	16.0	6.0
B (rich)	8.5	4.0	23.0
C	9.0	4.0	7.0
D	8.0	0.0	1.0

bicarbonate solutions sparged with CO_2, with various pH values and aggressive ion concentration and the free corrosion potential, E_{corr}, was measured. Polarisation curves were measured, at a scan rate of 10 mV/min, over a large potential range, from cathodic to anodic, until the pitting potential, E_{pp}, was exceeded. The measurements used a conventional three-electrode glass cell. The counter electrode was a platinum mesh square of 20 mm × 20 mm. Potentials were measured with respect to a saturated Ag/AgCl reference electrode and are presented with respect to the SHE scale.

Samples of grade ASTM A53 carbon steel (0.25%C, 0.9%Mn, 0.05%P, 0.06%S) of dimensions 10 mm × 10 mm × 5 mm were polished with silicon carbide paper down to 1,200 grit, washed in acetone in an ultrasonic bath for 2 min and mounted onto a glass working electrode holder, which allowed an electrical contact to the back of the specimen. These specimens were used to measure E_{corr} and the pitting potential, E_{pp}, in the test solutions presented in Table 1 with concentrations of aggressive ions presented in Table 2. These solutions contained no inhibitor. All solutions were made with reagent grade chemicals and deionised water. Test solutions A and B simulated the carbonate–bicarbonate fractions of the rich and lean solutions in an absorber tower, with a total equivalent carbonate content of 20 eqv. wt% K_2CO_3, and a fraction conversion of 0.8 and 0.2 respectively. Solutions C and D had pH values expected within the top and bottom of an absorber tower. All solutions were deaerated by sparging with CO_2 and maintained at a temperature of 95 °C. The pH of each solution was measured with two different pH metres that were rated to 100 °C and that had inbuilt temperature compensation. Both pH metres were calibrated at room temperature with three standard buffer solutions of pH 4.0, 7.0 and 10.0. Both pH metres gave comparable pH readings.

X-ray Photoelectron Spectroscopy (XPS) was used to investigate the passive film for specimens with a standard chemical passivation treatment comparable to that produced in plant. Grade ASTM A53 carbon steel samples of dimensions of 10 mm × 10 mm × 5 mm were prepared as described above and mounted onto the glass working electrode holder. These samples were subjected to the following passivation procedure.

Step 1 (pretreatment). The sample was immersed in 1wt% NaOH solution for 5 min at ambient temperature.

Step 2 (chemical passivation). The sample was immersed in a solution containing 1.0 wt% $KHCO_3$ and 0.8 wt% V_2O_5, aerated by air sparing, at a temperature of 90 °C for 8 h during which time E_{corr} was recorded. The pH of the solution was measured to be between pH 7.5–8.0.

Step 3 (verification of passivity). The sparging gas was changed to nitrogen. The potential of the steel sample was held constant at the previously measured value of E_{corr} for 1 h. The low current densities recorded verified that a good passive film had been developed.

XPS was performed using a Kratos Axis ULTRA X-ray Photoelectron Spectrometer incorporating a 165 mm hemispherical electron energy analyser. Survey scans were undertaken at an analyser pass energy of 160 eV and high resolution scans at 20 eV. Survey scans were carried out over 1,200–0 eV binding energy range with 1.0 eV steps and a dwell time of 100 ms. High-resolution scans were run with 0.1 eV steps and 250 ms dwell time. Base pressure in the analysis chamber was 1.0×10^{-9} Torr and 1.0×10^{-8} Torr during sample analysis. Depth profiling of the passive layer was performed using a differentially pumped Kratos Minibeam III ion gun. About 4 keV Argon ions were used at an ion source extractor current of 630 nA, rastered over an area of ~3 mm × 3 mm and stabilised by a Pfeiffer RVG 050C controller in conjunction with the Pfeiffer UDV 140 leak valve. During profiling the sample analysis chamber pressure was $\sim 5.0 \times 10^{-8}$ Torr.

Table 2 Test matrix showing concentration of aggressive ions

Solution	pH	Aggressive ion concentration (wt %)				
A	pH 9.5			1.0%KCl	10.0%KCl	10.0%KCl + 20.0% thiosulphate + 1.5% sulphate
B	pH 8.5			1.0%KCl	10.0%KCl	10.0%KCl + 20.0% thiosulphate + 1.5% sulphate
C	pH 9.0		0.1%KCl	1.0%KCl	10.0%KCl	10.0 %KCl + 20.0% thiosulphate + 1.5% sulphate
D	pH 8.0	0.05%KCl	0.1%KCl	1.0%KCl	10.0%KCl	10.0%KCl + 20.0% thiosulphate + 1.5% sulphate

Results

Free corrosion potential

The corrosion potential, E_{corr}, was monitored in the four solutions A, B, C and D, and the steady state values were measured after 10 min. Figure 1 shows, as a typical case, E_{corr} for the steel in solution D of pH 8.0 sparged with CO_2. The corrosion potential rapidly (i.e., in less than 30 s) attained a steady state value in the range of –585 to –620 mV for all chloride concentrations. Pitting was not observed on any of the steel surfaces during the immersion time of 2 h, indicating that the induction time required for pitting was longer than 2 h.

Pitting potential

The pitting potential is defined as a threshold potential above which pit nucleation and pit growth take place. The pitting potential, E_{pp}, of the ASTM A53 carbon steel was evaluated from the polarisation curves, as illustrated in Fig. 2, for the case of solution D (pH 8.0). For a chloride concentration of about 0.5 wt%, the current increased rapidly at a potential of approximately –300 mV, interpreted as indicating the initiation of pitting.

In the solutions free from aggressive ions there was no pitting and all polarisation curves displayed an increase in current above 900–1000 mV attributed to the evolution of oxygen.

The polarisation curves in solutions with aggressive ions indicated that there was no pitting for solution A (pH 9.5, lean), solution B (pH 8.5, rich) and solution C (pH 9.0) with chloride concentrations up to and including 1%. For those solutions, just like in the solutions free from aggressive ions, the polarisation curves displayed a rapid increase in current above 900–1000 mV due to oxygen evolution, indicating that the value of E_{pp} was above 900 mV.

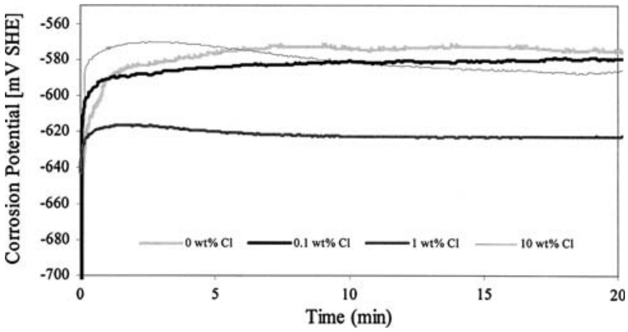

Fig. 1 Corrosion potential, E_{corr}, of ASTM A53 carbon steel immersed in solution D (pH 8.0) with indicated chloride concentration

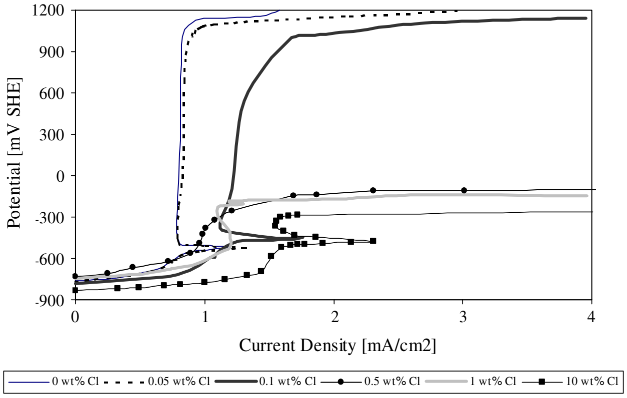

Fig. 2 Polarisation curves for ASTM A53 carbon steel in solution D (pH 8.0) and stated chloride concentrations

The effect of chloride concentration on the pitting potential, E_{pp}, is summarised in Fig. 3. Passivity breakdown may occur if E_{pp} is sufficiently low due to the presence of chlorides and if the solution is sufficiently oxidising (i.e., pitting may occur if $E_{corr} > E_{pp}$). Since the inhibitor influences the oxidising power of the solution and influences E_{corr}, its effect must be incorporated in determining whether pitting is likely to occur. For this reason, Fig. 3 includes the measured values of the corrosion potential, E_{corr}, measured in solution D (pH 8.0), in the presence (0.8 wt% V_2O_5) and in the absence of the anodic inhibitor.

Figure 3 shows that the pitting potential had relatively low values in solution D (pH 8.0) for chloride concentrations above 0.5 wt% and that there was a small decrease in E_{pp} to 570 mV in solution C (pH 9.0) containing 10 wt%

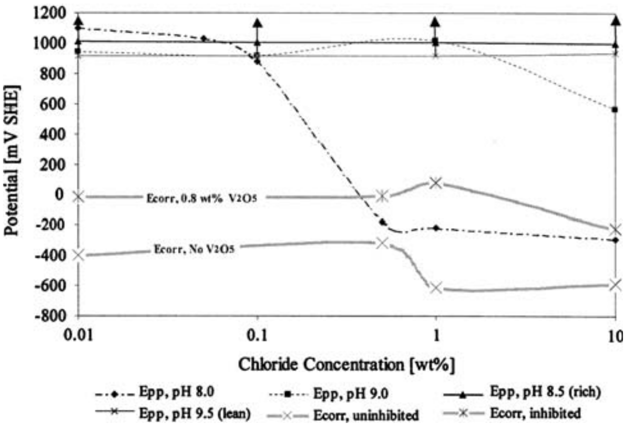

Fig. 3 The values of the pitting potential, E_{pp}, of ASTM A53 carbon steel in solutions A (pH 9.5), B (pH 8.5), C (pH 9.0) and D (pH 8.0) at 95 °C. Also shown are the values of E_{corr} in solution D (pH 8.0) with and without inhibitor

chloride. For all other solutions, the pitting potential was above the value indicated; this is designated by the arrows.

The decrease of E_{pp} for solution C (pH 9.0) and a chloride concentration of 10 wt% is considered to be not significant for HPC plant since it is unlikely that that the plant operates at such highly oxidising conditions (i.e., in excess of 570 mV) and with this high chloride concentration.

However, in solution D (pH of 8.0), which also coincides with the lowest equivalent carbonate concentration, chloride ions have a significant depassivating effect. A significant decrease in pitting potential, to –270 mV, occurred at a critical chloride concentration in the range of 0.1–0.5 wt%. This critical chloride concentration can be interpreted as indicating that at this critical concentration there was sufficient chloride to adsorb at weak sites on the passive layer, compromise passive layer stability and cause pitting. For chloride concentrations in excess of 0.5 wt%, the pitting potential remained constant with increasing chloride concentration suggesting that the amount of chloride ion adsorption was independent of chloride concentration at a chloride concentration greater than 0.5 wt%. This low pitting potential is not expected to cause pitting in the inhibitor free solution as the corrosion potential of the steel in this solution was below the pitting potential over the whole range of chloride concentrations. However, in the presence of inhibitor (0.8 wt% V_2O_5), the corrosion potential was shifted upwards by 500 mV, and the pitting potential was exceeded at a chloride concentration in excess of 0.4 wt%.

Effect of thiosulphate and sulphate Ions

The influence of thiosulphate and sulphate ions on the pitting potential is presented in Fig. 4. For solutions A (pH 9.5), B (pH 8.5) and C (pH 9.0), at a chloride concentration of 1 wt%, the presence of these sulphur containing anions shifted the pitting potential somewhat towards more negative values. For solution D (pH 8.0) there was a minor increase in the pitting potential.

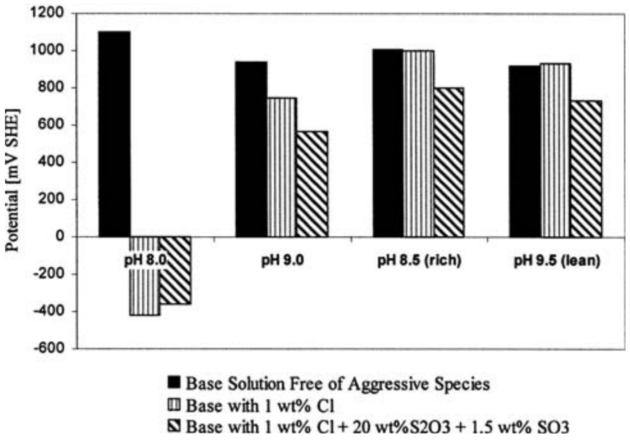

Fig. 4 Effect of thiosulphate and sulphate ions on the pitting potential, E_{pp}

XPS analysis of passive film

XPS and ion sputtering was used to estimate passive film thickness, Fig. 5, which shows the measured relative intensities of the Fe and O peaks. To estimate the thickness it is usual to use the point at half the maximum intensity of either O or Fe as the mid-point of the surface interface. Figure 5 shows that the O midpoint was at ~25 at% which corresponds to $T = 10$ min, whereas the Fe midpoint was ~30 at% which corresponds to $T = 5$ min. The thickness of the film plus interface was reached by a sputtering time of ~20 min, which suggests a passive layer thickness in the range of 5–20 nm (the sputtering rate was 1.0 nm/min) This passive layer thickness was an order of magnitude thinner than the expected thickness of a passive magnetite layer of around 0.1- to 0.3-μm thickness. However, the estimated thickness is not inconsistent with measured thicknesses of passive layers on stainless steels [10–14].

Discussion

Pitting

The influence of chloride ions on the pitting tendency of steel exposed to carbonate–bicarbonate solutions was a main factor studied in the present work, with the influence of other aggressive anions such as sulphate and thiosulphate also investigated. The results of Fig. 3 indicate that

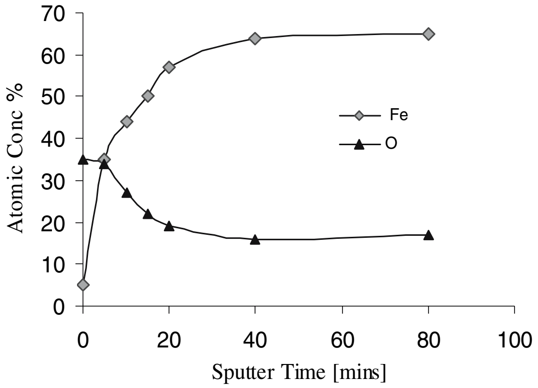

Fig. 5 XPS-sputter profile of ASTM A53 carbon steel passivated for 8 h in 1.0 wt% $KHCO_3$ solution with 0.8 wt% V_2O_5 and 0% Cl^- at 90 °C

chloride ions act as aggressive anions and promote pitting corrosion, as indicated in the shift of the pitting potentials towards more negative values. This effect was most evident in solution D (pH 8.0), the solution that had the lowest pH value of 8.0 and also the lowest equivalent carbonate concentration.

From the HPC plant perspective, the important information is an indication of whether or not chloride induced pitting will occur in the absorber tower, or more specifically, whether pitting will occur at the pH and carbonate concentrations expected in HPC plant solutions. Since it is not possible to duplicate the Benfield solution in a laboratory test at atmospheric pressures, in respect to carbonate composition and pH, these parameters were examined separately, by test solutions A to D. Solution D (pH 8.0), although adequately representing the lowest absorber pH, has a equivalent carbonate concentration lower than that of HPC plant solution. Solutions A (pH 9.5) and B (pH 8.5), that contained an equivalent carbonate concentration comparable to plant HPC solutions, offered good resistance to pitting, but these solutions do not cover the entire pH range expected within the absorber tower, namely pH 8.0 to 9.0. Whilst the results obtained at the pH and equivalent carbonate concentrations best reflecting plant solutions need to be equally considered, the difference in results requires a rationalisation on the importance of pH vs equivalent carbonate concentration on pitting potential in the presence of aggressive ions. Delineating which parameter, pH or equivalent carbonate concentration, is most influential in affecting pitting potential is assisted by a summary of the results presented in Fig 6, which shows an apparent dependence of E_{pp} on equivalent carbonate concentration in 10 wt% chloride solutions. There appears to be less of a relationship between pH and pitting potential. The marked difference in pitting susceptibility of ASTM A53 carbon steel in pH 8.0 and 8.5 solutions seems disproportionate to the small difference in pH.

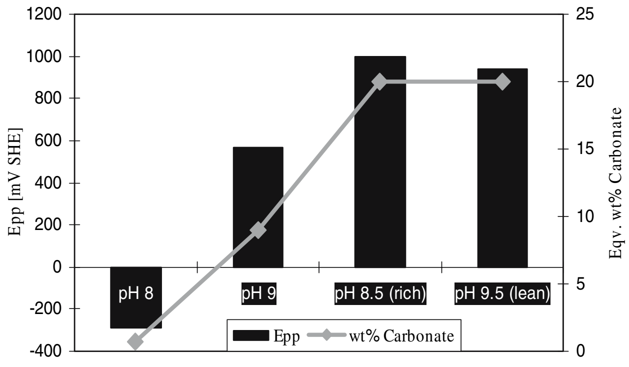

Fig. 6 The effect of pH and equivalent carbonate concentration on pitting potential, E_{pp}, in solutions containing 10 wt% chlorides

The dependence of pitting potential on equivalent carbonate concentration was already advanced by Evans [15]. Evans [15] proposed that OH^- ions form a chemisorbed film, which reinforces the existing passive film and repairs any weak spots. Uhlig and Revie [16] and Nesic et al. [17] suggested that the key issue was the competition between the OH^- and Cl^- ions for adsorption and advanced this explanation for the good pitting resistance of steels in high pH solutions. The literature suggests that chloride ions preferentially adsorb at passive film defect sites [18] and local corrosion is initiated at the defects in the passive film. It is suggested that the high OH^- ion concentration in high pH or concentrated carbonate solutions provides competition for Cl^- adsorption or repairs passive film weak spots thus lowering the pitting tendency [17].

Figure 6 indicated that the pitting potential is high for highly concentrated carbonate solutions. This is supported by Streicher [19] who studied the competitive adsorption of aggressive chloride ions in the presence of inhibitive OH^- ions. He found that the threshold Cl^- concentrations, above which pitting occurred, increased with an increase in the concentration of inhibitor ions. Furthermore, studies by Milosev et al. [20], with passivated copper exposed to bicarbonate solutions containing aggressive sulphate ions, shown the existence of a critical $[HCO_3^-]/[SO_4^{2-}]$ ratio above which the passive film is resistant to breakdown [20]. This is supported by similar studies by Muralidara et al. [21] that showed stable passivity of steel exposed to a sodium hydroxide solution even in the presence of up to 30,000-ppm aggressive ions. The carbonate ions may also assist in hindering pitting through a solution buffering which prevents acidification, and hence prevents stabilization of the pit growth mechanism cavity [22].

Relation to HPC Plant

The critical pitting potential (E_{pp}) was dependent on the concentration of aggressive chloride ions and the equivalent carbonate concentration. There were a number of possibilities for the mechanism responsible for the inhibition by carbonate ions. The presence of carbonate ions during passive film formation may have a positive effect on lessening the defect sites and inhomogenities in the passive film. The carbonate solutions may inhibit pit initiation by the production of OH^- ions which (1) compete with Cl^- ions for adsorption, (2) prevent the establishment of an acidic cavity through solution buffering and (3) may form a chemisorbed film that repairs the passive layer. The threshold chloride concentration in potassium carbonate solutions above which pitting is initiated is approximately 0.5 wt% chloride. This is pertinent to dilute carbonate solutions. In concentrated solutions, the threshold chloride concentration was above 10 wt%. Since the results show that stability towards localised corrosion increases with increasing carbonate concentration, and considering that the HPC plant solutions are concentrated solutions, chlorides are not considered to be a threat to pitting in absorber towers. In these absorbers towers, the maximum chloride concentration has been reported to be approximately 0.08 wt%, which is well below the threshold chloride concentration found in this investigation. However, the susceptibility of pitting in the dilute carbonate solution should be considered in plant operation and included in the guidelines for HPC plant corrosion control. The threshold chloride concentration of 0.5 wt% found in this solution should be considered as a critical operating parameter so that the HPC process should be operated to below this limit at all times.

One condition that is present in HPC plants that was not covered in this research was a concentrated solution with a pH of 8.0, such as the “rich” solution present at the bottom of an absorber tower. Due to experimental limitations, this solution cannot be replicated in a laboratory under atmospheric test conditions. In light of the above findings which have shown the inhibitive properties of concentrated carbonate solutions on pitting, the “rich” HPC plant solution, despite its low pH value of 8.0, is expected to have similar resistance to chloride induced pitting corrosion as the concentrated carbonate solutions of pH 8.5 and 9.5 tested in this investigation. Additional work in an autoclave would provide assurance and allow the important parameters of carbonate concentration and pH of a rich solution to be simultaneously replicated in a single test. The strong correlation of carbonate concentration with pitting potential, together with the apparent ability of carbonate solutions to produce a sufficient amount of OH^- ions to lead to the inhibitive effect seen in this investigation, exclude pitting of carbon steel in HPC plant solutions at the pH levels of 8.0–9.0.

Conclusions

- The passive layer was about 5- to 20-nm thick.
- The pitting potential was dependent on the chloride concentration and the equivalent carbonate concentration.
- In 1 wt% bicarbonate solution containing inhibitor, the threshold chloride concentration above which pitting might be expected is 0.5 wt% chloride.
- It is recommended that HPC plant operation be such that the chloride concentration is maintained below 0.5 wt% chloride.
- Chloride depassivation does not appear to be a cause of HPC corrosion issues.

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