International Journal of ELECTROCHEMICAL SCIENCE www.electrochemsci.org

Study on corrosion behavior of P110S steel in CO₂-H₂Ssaturated solution

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Received: 19 September 2021 / Accepted: 30 October 2021 / Published: 6 December 2021

The corrosion behavior of P110S steel in CO₂-H₂S-saturated completion fluid with pH buffer was studied using immersion tests and electrochemical tests. SEM, EDS, XRD, and potential-pH diagrams were used to analyze the corrosion scale characteristics. P110S steel experienced severe localized corrosion in CO₂-H₂S-saturated completion fluid despite the addition of pH buffer. The main component of the corrosion scale was mackinawite and FeCO₃. The corrosion scale with mackinawite as the main component had an excellent protective effect on the matrix when the scale was intact. However, the corrosion scale was poorly bonded to the matrix and was prone to partial cracking and peeling, which was the cause of severe localized corrosion of P110S steel.

Keywords: P110S, pH buffer, CO₂-H₂S corrosion, Corrosion scale, Electrochemical

1. INTRODUCTION

As the oil and gas wells go deeper, the density and corrosion resistance requirements for completion fluids are increased. Commonly used high-density solid-free completion fluids, such as sodium chloride and sodium bromide, typically have high ionic concentrations that can cause severe corrosion of the casing and tubing once CO_2 -H₂S seep from the threads into the annulus. Corrosion can result in leakage, pollution, high-risk events, and high costs for the wells' workover[1-4]. pH buffer is an economical method of inhibiting CO_2 corrosion[5]. Bicarbonate/carbonate salt is an efficient pH buffer widely used in formate completion fluids[6-10]. The buffers resist pH changes by consuming hydrogen ions (H⁺), as shown in the following equations.

$$CO_3^{2-} + H^+ = HCO_3^-$$
(1)

$$HCO_3^- + H^+ = H_2CO_3$$
 (2)

When there is still CO_3^{2-} in the solution, the pH will remain at 10.2. Even if the CO_3^{2-} is completely consumed, the pH will drop, but it will remain permanently around 6.35[10].

Some studies[5,11-20] have reported the dual effects of bicarbonate/carbonate. Carbon steel can form a dense corrosion scale and be passivated in NaHCO₃ solution[5,11]. A higher concentration of HCO_3^- can promote thicker passivation films to form and accelerate the formation of passivation films.[12]. However, some researchers found that HCO_3^- ions with high concentrations can promote steel pitting at higher pH values. AL-Kharafi[13] found that at high concentrations of $CO_3^{2^-}$ - HCO_3^- buffer solution, a high concentration of Cl⁻ ions can cause significant damage to the passivation film of carbon steel, which may lead to pitting corrosion. Han[20] found that a high pH can allow concentrated HCO_3^- to form, thus aggravating pitting. Zhang[14] found that in a solution with only bicarbonate and no carbonate, the corrosion rate of carbon steel reached 32 mm/a.

In addition, in the actual oil and gas production environment, CO_2 -H₂S usually co-exist. Studies have shown that the formation of mackinawite can result in pitting in alkaline solutions, possibly due to competition between different corrosion products[21~25]. Maxwell[26] found that small sulfide concentrations catalyze steel corrosion in carbonate/bicarbonates solutions. For pH values \leq 9, active prevails. Therefore, using pH buffers as a means of corrosion control in a corrosive environment containing H₂S may be risky. However, most of the current studies about pH buffers focus on a pure CO_2 environment. Studies on the H₂S influence on the corrosion of steels in completion fluid with pH buffer are scarce, and the characteristics of corrosion scale and local corrosion mechanism are still unclear.

In this study, the corrosion behavior and mechanism of P110S steel in high-density completion fluid, containing saturated CO_2 -H₂S and sodium carbonate/sodium bicarbonate pH buffer using a high-temperature and high-pressure autoclave, surface analysis methods, and electrochemical measurements. The research results are of great significance to the corrosion control of oil and gas facilities.

2. EXPERIMENT

2.1. Material and solution

The material used here was P110S steel, sulfur-resistant low alloy steel, with chemical compositions (wt%) detailed in Table 1. The size of the immersion samples is $50 \times 13 \times 3 \text{ mm}^3$, and the size of the electrochemical samples is $10 \times 10 \times 3 \text{ mm}^3$. The samples were polished to 1000 grit and then cleaned with distilled water and alcohol. The composition of the solutions is listed in Table 2. The test solution was deoxidized for 8 h before the experiment with 99.999% N₂.

Table 1 Chemical composition of P110S steel (wt%)

Materials	С	Р	Si	S	Cr	Mn	Cu	Mo	Ni	Fe
P110S	0.30	0.006	0.38	0.0015	0.54	0.43	0.051	0.89	0.046	Bal.

Composition	NaCl	NaHCO ₃	Na ₂ CO ₃	MgCl ₂ ·6H ₂ O	CaCl ₂	Na ₂ SO ₄	Na ₂ S	pH (80°C, 0.1 MPa CO ₂)
Immersion/ Electrochemical test	150000	7700	11000	13.1	4.5	16.6	0	7.56
Electrochemical	150000	7700	11000	13.1	4.5	16.6	500	7.58
test	150000	7700	11000	13.1	4.5	16.6	5000	7.70

Table 2 Composition of the immersion and electrochemical tests solution $(mg \cdot L^{-1})$

2.1 Immersion test

The corrosion simulations were performed in a 3 L autoclave. After immersion for 120 h, the corroded samples removed from the autoclave were immediately cleaned with deionized water and dried. Then the samples were soaked in an acid washing solution for 5 min to remove the corrosion scale, then rinsed, dried, and weighed. The corrosion rate (CR, mm/a) was obtained as follows:

$$CR = \frac{876000(W_0 - W_1)}{t\rho A}$$
(3)

Where W_0 is the sample weight before the experiment, g; W_1 is the sample weight after the experiment, g; ρ is the sample density, g/cm³; A is the exposed area, cm²; and t is the immersion time, h.



Figure 1. High-temperature and high-pressure autoclave scheme

The corrosion scale's morphology and energy dispersive X-ray spectroscopy (EDS) analysis were investigated using a Zeiss SUPRA55 SEM. X-ray diffraction (XRD, RIGAKU Ultima IV) was used to analyze the phase and crystal structure of the corrosion scale.

The immersion tests were performed at 80 °C and 120 °C. The CO_2 partial pressure is 2 bar, and the H₂S partial pressure is 0.5 bar.

2.3 Electrochemical tests

The electrochemical tests were conducted in a 1L glass cell with a three-electrode system using a Gamry Interface 1000 electrochemical workstation. The working electrode had an exposed area of 1 cm². A 20×20 mm² platinum sheet served as a counter electrode. A saturated calomel electrode (SCE) served as a reference electrode. The electrochemical impedance spectroscopy (EIS) tests were conducted at the open circuit potential (OCP) using a 10 mV alternating current perturbing signal in the frequency range from 100 kHz to 10 mHz. After immersion for 1 h in a solution with different concentrations of Na₂S (0, 0.5, and 5 g/L), the potentiodynamic polarization curves were measured at a voltage ranging from -300 to 1500 mV vs. OCP using a scan rate of 0.5 mV/s.

The electrochemical measurements were conducted at atmospheric pressure with CO₂ gas continuously bubbled into the solution at 80 $^{\circ}$ C.

3. RESULTS AND DISCUSSION

3.1 Corrosion rate

As illustrated in Fig.2, the average corrosion rate of P110S steel is only 0.20 mm/a and 0.14 mm/a at 80 °C and 120 °C, respectively, which is significantly lower than that measured in the solution without pH buffer[1,27], indicating that the pH buffer is highly effective in controlling the general corrosion. However, the maximum local corrosion rate can reach 7.30 mm/a and 10.95 mm/a at 80 °C and 120 °C, respectively.



Figure 2. Corrosion rate of P110S steel after immersion for 120 h at 80 °C and 120 °C

3.2 Macro morphology

Fig.3 displays the macroscopic morphology of P110S steel samples before and after removing the corrosion scale and the three-dimensional morphology of the steel surface without corrosion scale. Most surface areas were covered by intact corrosion scales (Fig,3a and Fig.3e). The three-dimensional morphology shows almost no corrosion occurred in these areas (Fig,3d and Fig.3h). However, some

areas on the sample surface were barely covered by corrosion scales, and the corrosion was severe. The three-dimensional morphologies (Fig.3c and Fig.3g) of the samples show that the maximum pitting depths were 100 μ m and 150 μ m at 80 °C and 120 °C, respectively.



Figure 3. Morphology of P110S steel after immersion for 120 h: (a)80 °C, with corrosion scale; (b) 80 °C, without corrosion scale; (c) (d) 80 °C, 3D morphology;(e)120 °C, with corrosion scale; (f) 120 °C, without corrosion scale; (g) (h) 120 °C, 3D morphology

The overwhelming view is that the corrosion rate of carbon steel or low-alloy steel can be maintained at an acceptable level and not prone to localized corrosion in the aqueous with pH buffers[5, 28~30]. In contrast, this study found that P110S steel was at risk of localized corrosion in CO_2 -H₂S-saturated solution with pH buffer. This means the pH buffer may have little effect on corrosion control in the CO_2 -H₂S-saturated solution.

3.3 Micromorphology

SEM and EDS analyses of corrosion scales were carried out to investigate the causes of localized corrosion and localized flaking of corrosion scale. Fig.4 displays the SEM and EDS results of P110S steel after immersion for 120 h at 80 °C. As shown in Fig.4a, most areas of the sample surface were covered by the corrosion scale. Although there are small holes in the corrosion scale, the corrosion of the steel matrix was very slight, indicating that the corrosion scale had excellent protection to the matrix. EDS results show that the corrosion scale was rich in S, and the percentage of O atoms was much lower (Fig.4c). It is presumed that the main composition of the corrosion scale was the FeS compound. After the corrosion scale was peeled off, the sample's surface was directly exposed to the aqueous with scattered cubic FeCO₃ grains on the steel surface. Since FeCO₃ did not form a continuous corrosion scale, it provided little protection to the matrix.

Similar results were obtained at 120 °C. As shown in Fig.5a, most areas of the samples were covered by an intact and dense corrosion scale, and unlike 80 °C, no holes were observed on the corrosion scale at 120 °C, which is related to the higher temperature. Generally speaking, the higher the temperature, the denser and more protective the film of corrosion products formed on carbon steel's

surface. As shown in Fig.5b, the same apparent corrosion scale delaminating and peeling off phenomenon was observed near the edge of the local corrosion pits. As shown in Fig.4b and Fig.5b, the surface morphology of the delaminated corrosion scale is identical to the intact one. Accordingly, it can be presumed that there was also a complete corrosion scale cover in the local corrosion areas initially. However, the corrosion scale and the matrix bond were weak, accessible to delamination, and peeling off for some reasons.



Figure 4. SEM image and EDS analysis of corrosion scale of P110S steel after immersion for 120 h at 80 °C

Further, the cross-sectional morphology of the corrosion scale was analyzed. As shown in Fig.6 and Fig.7, the film thickness was $<50 \mu m$ and $<40 \mu m$ at 80 °C and 120 °C, respectively. As shown in Fig.6a, the corrosion scale delaminated significantly, and severe corrosion of the matrix occurred. Also, in Fig.7a and 7b, cracks were observed at the interface and inside the film. The above results indicate that the corrosion scale did have a weaker bond with the matrix and was easy to delaminate. Also, as shown in Fig.6a, FeCO₃ grains were observed under the delaminated corrosion scale, which echoes the FeCO₃ distribution observed in Fig.4d. Because the growth of large FeCO₃ grains takes time, only when the delaminating of the corrosion scale occurs during the experiment can FeCO₃ grains regrow under the original corrosion scale in the subsequent corrosion process. EDS point scan results show that the corrosion scale contained Fe, S, O, and other elements with low content. Presumably, the corrosion products contained FeS compounds and FeCO₃. Among them, the FeS compound was the main

component. In addition, according to the EDS map scan results, it was found that the distribution of Fe, S, O elements in the whole corrosion scale was relatively uniform. This result indicates that the corrosion scale is a monolayer structure, and FeS and FeCO₃ participate in the deposition process of the corrosion scale simultaneously.



Figure 5. SEM image and EDS analysis of corrosion scale of P110S steel after immersion for 120 h at 120 °C



Figure 6. Cross-sectional morphology and EDS analysis results of corrosion scale of P110S steel after immersion for 120 h at 80 °C



Figure 7. Cross-sectional morphology and EDS analysis results of corrosion scale of P110S steel after immersion for 120 h at 120 °C

3.4 Potential-pH diagram

The potential-pH diagram is an essential tool to map out the possible stable species in an aqueous environment at different pH and potential combinations. The potential-pH diagrams of the Fe-CO₂-H₂O system and Fe-H₂S-H₂O system were drawn better to understand the corrosion scale formation and corrosion mechanism. The relationship between potential and pH in the potential-pH diagram was calculated using the Nernst equation. The equations of chemical species used in thermodynamic calculations are listed in Table 3. The thermodynamic data used for the calculations were obtained from the literature[31-34].

The diagonal dashed lines, represented by the letters a and b, are water reduction and water oxidation lines. Due to the absence of oxygen in the aqueous studied in this paper, the primary cathodic reaction is a reduction reaction of hydrogen ions, and attention should be paid to the area below the water reduction line(a). The pH values obtained from the measurements have been marked with dashed lines in the diagrams. As shown in Fig.8, the main stable corrosion product is FeCO₃ in the Fe-CO₂-H₂O system and FeS in the Fe-H₂S-H₂O system at 80 °C and 120 °C. At the same time, we note that the stability area of FeS covers the stability area of FeCO₃ and Fe₃O₄, indicating that it is more inclined to form FeS in the CO₂-H₂S saturated aqueous. This is an important reason for the predominance of FeS in the corrosion proceeds and Fe²⁺ concentration increases, then FeCO₃ is formed by deposition. In addition, the pH at 120°C is higher than that at 80°C, and in a higher temperature, higher pH environment, it is easier to form corrosion scale, which is one of the important reasons for the lower general corrosion rate at 120°C

Table 3. Equations of chemical species used in thermodynamic calculations

No.	Reaction	Equilibrium
1	$2H^+ + 2e = H_2$	$E_{(H^+/H_2)} = E_{(H^+/H_2)}^0 + \frac{RT}{2F} ln\left(\frac{c_{H^+}^2}{P_{H_2}/P^{\theta}}\right)$





Figure 8. Potential-pH diagram: (a)Fe-2bar CO₂-H₂O,80 °C; (b) Fe-2bar CO₂-H₂O,120 °C; (c) Fe-0.5bar H₂S-H₂O,80 °C; (d) Fe-0.5bar H₂S -H₂O,120 °C

3.5 XRD analysis



Figure 9. XRD analysis results of corrosion scale of P110S steel after immersion for 120 h

The XRD results are shown in Fig.9. At 80 °C and 120 °C, the main component of the corrosion products was mackinawite. Furthermore, small amounts of pyrrhotite and FeCO₃ were also detected. The XRD results agree with the predicted results of potential-pH diagrams and EDS results.

3.6 EIS and Potentiodynamic Study

In order to clarify the mechanism of the effect of H_2S on the corrosion scale and localized corrosion, electrochemical experiments were conducted in saturated brine containing different concentrations of S^{2-} .

Fig. 10 shows the EIS test results. All 3 Nyquist curves show 2 time constants. The EIS results were fitted using the equivalent circuit illustrated in Figure 10. The results of the fitting are shown in Table 4. R_s , R_f , and R_{ct} are the resistance of the solution, porous and charge transfer, respectively. CPE_{film} and CPE_{dl} denote the constant phase element (CPE) of the corrosion scale and double layer, respectively.

Both Rct and Rf increased with S^{2-} concentration. It indicates that the steel is more likely to form a very protective film on corrosion products in the solution with S^{2-} ions. The result proved that the corrosion scale with good protection could quickly form on the surface after the sample was immersed into the solution. And the protective properties of the film are mainly related to S^{2-} ions.



Figure 10. EIS curves of P110S steel at different Na₂S concentrations

Table 4. EIS fitting results

Na ₂ S Concentration	R	CPE_{f}		Re	CPE_{dl}		R.,
(g/L)	$(\Omega \cdot cm^2)$	$\begin{array}{c} Y_1 \\ (\times 10^{-4} \Omega^{-1} \cdot \mathrm{cm}^{-2} \cdot \mathrm{s}^{\mathrm{n}}) \end{array}$	n_1	$(\Omega \cdot \mathrm{cm}^{2)}$	$\begin{array}{c} Y_2 \\ (\times 10^{-4} \Omega^{-1} \cdot \mathrm{cm}^{-2} \cdot \mathrm{s}^{\mathrm{n}}) \end{array}$	n_2	$(\Omega \cdot cm^2)$
0	0.79	69.9	0.82	49.61	95.2	0.85	37.33
0.5	0.27	37.9	0.94	721.5	51.7	1.0	236.8
5.0	0.83	0.0569	0.67	16350	43.6	1.0	5186

Fig.11 shows the polarization curves tested in 3 solutions with different S^{2-} concentrations. Corrosion currents obtained from the polarization curves are listed in Table 5. The polarization curves can be divided into 3 zones. In the zone1 and zone2 regions, the higher the S^{2-} ion concentration, the lower the current density at the same potential, and, in the zone2 region, the polarization curves of P110S in the S^{2-} ion-containing solution show a passivation-like phenomenon. The above phenomenon proves that a corrosion scale with good corrosion resistance can form on the steel surface quickly. When the potential rises to the zone3 region, the current density even exceeds that in the solution without S^{2-} ions. In the 5g/L Na₂S solution, the current density fluctuates dramatically, and a phenomenon similar to the metastable pitting sprouting of stainless steel can be observed. The above phenomenon proves that the corrosion scale can be easily destroyed at high polarization potential. The destruction of the corrosion scale can lead to localized corrosion.

In summary, it can be deduced from the electrochemical test results that the addition of S^{2-} to the solution will promote the rapid formation of a corrosion scale with good protection on the metal surface. However, this film is unstable and can be easily damaged, leading to severe localized corrosion.



Figure 11. Polarization curves of P110S steel at different Na₂S concentrations

Na ₂ S Concentration, (g/L)	Passive current density, mA·cm ⁻²	Corrosion current density, mA·cm ⁻²	Current density at 0.6V vs. SCE, mA·cm ⁻²
0	/	221	58.8
0.5	132	40.3	336
5.0	30.0	2.56	343

Table 5 Corrosion current obtained from the polarization curves

3.7 Corrosion Mechanism

According to the results of immersion and electrochemical tests, the local corrosion mechanism of P110S steel in CO₂-H₂S saturated solution with pH buffers shown in Fig.12. Usually, when the partial pressure ratio of CO₂ to H₂S is greater than 20, H₂S corrosion dominates[34]. Therefore, in the early stage of corrosion, a layer of corrosion scale with FeS as the main component was rapidly formed on the surface of P110S steel (Fig.12a). According to the SEM results (Fig.4a and Fig.5a), this corrosion scale was dense. According to the three-dimensional morphology of the sample after the removal of the corrosion scale (Fig.2d and Fig.2h) and the EIS results (Fig.10), this layer of corrosion scale has good protective properties to the matrix when it is intact.

However, this corrosion scale is prone to crack and peel off. Research[35] shows that the volume of FeS is 2.56 times larger than that of the iron it replaces at the mackinawite/steel interface, which leads to an increase of the internal compressive stress in the mackinawite scales. As shown in Fig.12b, with the continuous deposition of the corrosion scale, the internal compressive stress gradually increases, and

when it exceeds the mechanical limit of the corrosion scale, the corrosion scale will crack to release the stress. With the development of corrosion, the cracks become larger and larger, eventually leading to the partial peeling off from the surface (Fig.4b and Fig.5b). In short, the corrosion scale is not firmly bonded to the matrix and is prone to crack and peel off. After the destruction of the corrosion scale, the localized corrosion rate in the broken area will be significantly increased (Fig.11).



Figure 12. Schematic diagrams of localized corrosion development

At the same time, the cathodic reaction will proceed according to Equations 4-6[37, 38] in the area still covered with the corrosion scale. Since the anodic area is much smaller than the cathodic area, the galvanic corrosion accelerates the anodic area's corrosion (Equation 7), resulting in severe localized corrosion[39-41].

$$2H^+ + 2e = H_2$$
 (4)

$$2HS^{-} + 2e = H_2 + 2S^{2-}$$
(5)
$$2HCO_{-}^{-} + e = H_2 + CO_{-}^{2-}$$
(6)

$$Fe - 2e = Fe^{2+}$$
(7)

$$Fe - 2e = Fe^{2r}$$
 (7)

5. CONCLUSION

The corrosion behavior and corrosion scale characteristics of P110S steel in CO₂-H₂S-saturated completion fluid with pH buffer were investigated. The main conclusions can be drawn as follows,

1) The localized corrosion rates of P110S steel at 80 °C and 120 °C were as high as 7.30 mm/a and 10.95 mm/a, respectively, which were tens of times higher than the general corrosion rates. Using pH buffer as a corrosion control measure for H₂S-containing environments has a localized corrosion risk.

2) The corrosion scale of P110S steel was monolayer at both 80 $^{\circ}$ C and 120 $^{\circ}$ C. The main component of the corrosion scale is mackinawite, with small amounts of FeCO₃ and pyrrhotite.

3) The corrosion scale with mackinawite as the main component provided excellent protection to the matrix. The corrosion of P110S covered by the corrosion scale is very slight. However, this corrosion scale was poorly bonded to the matrix and was prone to local cracking and peeling, which was the cause of severe localized corrosion of P110S steel in CO_2 -H₂S-saturated completion fluid with pH buffer.

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