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A Study on the CO₂ Corrosion Behavior of P110 Steel in High-Density Cement

Huali Zhang¹, Yufei Li¹, Shuliang Wang^{1,2,*}, Jiaji Zhang², Tuo Deng², Xueni Zhang², Zhi Zhang³

¹ Gas Production Engineering Research Institute, PetroChina Southwest Oil and Gas Field Branch, Chengdu, Sichuan 610017, China
² School of New Energy and Materials, Southwest Petroleum University, Xindu Avenue 8#, Sichuan 610500, China
³ School of Petroleum Engineering, Southwest Petroleum University, Xindu Avenue 8#, Sichuan 610500, China
*E-mail: wsliang1465@126.com

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High-density cement slurry is commonly used for cementing deep and ultradeep wells. During service, corrosion problems at the interface between the cement and casing steel seriously affect the safety of oil and gas extraction. In this paper, a specimen of a high-density cement/casing steel system was prepared by using hematite powder as the weighting agent. The electrochemical properties and microstructure were investigated by electrochemical techniques, SEM, Raman spectroscopy and XPS. Samples were corroded in simulated formation water saturated with CO_2 at 90 °C for 7–49 days. The results show that with increasing immersion time, the corrosion rate of casing steel in cement gradually increases and the cement resistance first increases and then decreases. The pressure promotes the corrosion reaction; thus, the corrosion potential is more negative and the corrosion rate is higher. The corrosion product morphology on the surface of casing steel in cement is a distribution of cracks. The corrosion product is FeCO₃, showing that mainly CO_2 corrosion occurs. The microgap at the cement/casing steel interface is only 1.0 μ m and accompanied by FeCO₃ production.

Keywords: High-density cement, P110 Steel, Cementing interface, CO₂ corrosion, Electrochemistry

1. INTRODUCTION

With the development of oil and gas exploration toward deep and ultradeep wells [1], highdensity cement slurry is often used to stabilize the formation and thereby improve the quality of cementing [2]. At present, the density of cement slurry is often increased by adding weighting agents. Hematite powder, as a common weighting agent for high-density cement slurry, requires relatively little water, does not promote serious thickening of cement slurry and has good rheological properties [3]; therefore, it is widely used in cementing projects.

CO₂ corrosion is one of the common forms of corrosion in oil and gas fields. CO₂ will react with cement due to an acid-base reaction (carbonation), causing microgaps at the interface between the cement ring and casing [4]. These microgaps will destroy the integrity of the cement ring and cause some corrosive fluids to come to contact with the casing steel. This contact will result in perforation and puncture exposures and even direct scraping, resulting in very large economic losses. At present, some scholars have conducted studies on the corrosion problem at the interface between cement and casing steel [5-8]. However, the corrosion of casing steel and the interface between cement and casing steel in high-density cement have not been covered.

In this paper, hematite powder is chosen as the weighting agent in a high-density cement slurry. The CO_2 corrosion law of the high-density cement and casing steel system is studied to provide a reference for solving corrosion problems faced in actual applications of high-density cement and casing steel.

2. EXPERIMENTAL SECTION

2.1 Sample preparation

The cement/casing steel samples were made of grade G cement and P110 casing steel. The composition of the hematite high-density cement slurry (mg/L) was cement: 400 g, hematite: 600 g, silica: 140 g, microsilica: 8 g, gas channeling-resistant agent: 6 g, filtrate reducer: 23.08 g, dispersant: 46.16 g, NaCl: 36.62 g, defoamer: 5 g, and a water-cement ratio of 0.65. During sample preparation a conductor was welded to the bottom of the casing steel sample. High-temperature-resistant epoxy resin was used to seal the two bottom-end surfaces of the casing steel, leaving only the side of the casing steel as the working surface. The working surface of the sample was polished from 400 to 1200 grit with SiC sandpaper, cleaned with acetone and alcohol and then dried by cold air. The casing steel was placed in the center of a mold, and the prepared cement was poured into the mold and removed after curing at 90 °C for 36 h. Figure 1 shows the specimen diagram. The cement cylinder had a diameter of 30 mm and a length of 70 mm, while the casing steel specimen, which was placed in the center of the cement cylinder, had a diameter of 9.5 mm and a length of 20 mm.



Figure 1. Schematic diagram of the cement/casing steel sample

The simulated formation water solution used for the experiments was prepared by adding appropriate chemicals to deionized water according to the formation water composition in Table 1. The solution was stirred with a glass rod to completely dissolve the added chemicals. The solution was completely deoxygenated by continuous bubbling with nitrogen gas for 4 h per 2500 mL of solution and then saturated with CO_2 gas for 4 h.

Table 1. Composition of the simulated formation water (unit: mg/L)

Composition	Na^+	Ca^{2+}	Mg^{2+}	Cl	HCO3 ⁻	SO_4^{2-}	pН	Mineralization
Content	4457.7	204.3	31	6757.8	874.6	76.6	7.31	11964.6

2.2 Corrosion immersion experiment

Corrosion immersion experiments were divided into two parts: high temperature–atmospheric pressure and high temperature–high pressure, and the experimental solutions were simulated groundwater solutions with saturated CO₂. The high-temperature and high-pressure experiments were carried out in a high-temperature and high-pressure reactor at a temperature of 90 °C, total pressure of 10 MPa and CO₂ partial pressure of 3 MPa. High-temperature–atmospheric pressure experiments were carried out in 1000 mL wide-mouth flasks at 90 °C. The prepared cement/casing steel specimens were immersed under the above two conditions for 7–49 days, and the corrosion patterns of the different experiments were measured.

2.3 Electrochemical test

Electrochemical measurements were performed using a Gamry Reference 3000 electrochemical workstation. Electrochemical tests were carried out with a three-electrode system, in which the working electrode was the cement/casing system sample (Figure 1), the auxiliary electrode was a 2 cm \times 2 cm platinum electrode, and the reference electrode was a AgCl electrode. The experimental medium was simulated formation water with saturated CO₂, and the test temperature was 90 °C.

The corrosion potential is the potential of a metal at a stable state without an applied current. The corrosion potential was tested at 90 °C for 1800 s by the electrochemical workstation, and the corrosion potentials after the system was stable were recorded. Electrochemical impedance spectroscopy (EIS) was performed after the system was stable. The test frequency range was 10^{-2} ~ 10^{5} Hz, and the potential amplitude of the sinusoidal AC signal was 10 MV. The scanning potential range of the linear polarization resistance (LPR) test was ±10 mV, and the scanning rate was 0.167 mV/s.

2.4 Analysis of the corrosion products

In this paper, a scanning electron microscope (ZEISS EVO MA15, Carl Zeiss, Germany) was used to analyze the microscopic characteristics of the corrosion products on the cement/casing steel

interface and casing steel surface at different times. The composition of corrosion products was analyzed by energy dispersive spectroscopy (EDS) with the scanning electron microscope. A K-Alpha⁺ X-ray photoelectron diffractometer with an Al target as the X-ray excitation source was used to analyze the elemental composition and elemental valence of the corrosion products on the surface of the casing steel. An ID Raman micro IM-52 Raman spectrometer was used to identify the corrosion products on the surface of the casing steel and to analyze the corrosion product composition.

3. RESULTS AND DISCUSSION

3.1 Electrochemical measurement results

3.1.1 Corrosion potential



Figure 2. Corrosion potential of cement/casing steel under high and normal pressure with different immersion time.

Figure 2 shows the corrosion potential measurements of the cement/casing specimens at different times under both atmospheric and high-pressure conditions. From the figure, it can be seen that the corrosion potential tends to become a straight line under normal and high-pressure conditions and for immersion time ranging from 7 days to 42 days, indicating that the corrosion condition of P110 casing steel in cement does not change much, while the corrosion potential at normal pressure and high pressure show positive change at a immersion time of 49 days. This result is probably because the byproduct of the cement carbonation reaction, namely, CaCO₃, fills into the pores of the cement; thus, the pores shrink, which leads to improved protection of the casing steel. Comparing the results when under atmospheric pressure and high-pressure conditions, the corrosion potential is found to be more negative under high-pressure conditions than under atmospheric-pressure conditions, indicating that the corrosion of casing steel is worse under high-pressure conditions. This result is attributed to the pressure promoting the penetration of corrosive media into the cement. In addition, the corrosion potential measurement results

show that the corrosion potentials under atmospheric and high-pressure conditions are -386 mV~-536 mV and -480 mV~-555 mV, respectively. The corrosion potential under both conditions is less than - 276 mV; therefore, it can be determined that the probability of casing steel corrosion is more than 90%, according to the U.S. ASTMC876 standard [9]. Therefore, the corrosion of casing steel in high-density cement occurs under both atmospheric and high-pressure conditions.

3.1.2 Electrochemical impedance spectroscopy (EIS)

(1) EIS of the cement/casing system under atmospheric pressure

Figure 3 shows the EIS diagrams under atmospheric-pressure conditions. As Figure 3(a) shows, the EIS diagram has capacitive resistance arcs in both the low- and high-frequency regions. The capacitive resistance arc radius in the low-frequency region decreases with increasing immersion time. In general, the radius of the low-frequency region represents the magnitude of the polarization resistance of the steel in the cement [10], and the capacitive resistance arc in the high-frequency region is related to the nature of the cement [11]. The polarization resistance is inversely related to the corrosion rate, and the corrosion rate of P110 steel in cement increases gradually with increasing immersion time. A magnification of the high-frequency region in the EIS diagram reveals the appearance of Warburg impedance, representing the diffusion characteristics of corrosion over time and suggesting that corrosion is controlled by the diffusion of reactive materials [12]. This result means that the corrosion rate of casing steel mainly relies on the continuous diffusion of the corrosive medium into the cement interior, thereby causing casing steel corrosion. In combination with Figure 3(b), it is found that the EIS diagram for different times of corrosion of the cement/casing steel specimens contains two time constants, representing the carbonation reaction of the cement and the corrosion reaction of the P110 casing steel surface.



Figure 3. Electrochemical impedance spectra with different immersion time under normal pressure: (a) Nyquist plot and (b) Bode plot

The EIS profile was fitted using ZsimpWin software, and the corresponding equivalent circuit diagram [13,14] is shown in Figure 4. R_1 is the solution resistance; R_2 is the cement resistance; Q_1 is a

constant phase angle element representing the cement capacitance C_1 ; R_3 is the bilayer charge transfer resistance on the casing steel surface, representing the polarization resistance; W is the diffusive Warburg impedance; and Q_2 is the electric double-layer capacitance of the casing steel surface. The constant phase angle element can be calculated by the following equation [15]:

$$Z(\omega) = Z_0^{-1} (j\omega)^{-n} \qquad (1)$$

where ω is the angular frequency and $n (0 \le n \le 1)$ is the constant phase angle component parameter, the value of which depends on the roughness of the sample surface. When n=0, Q represents pure resistance. When n=1, Q represents pure capacitance, and when n=0.5, Q represents Warburg impedance.



Figure 4. Equivalent circuit diagram

Table 2 shows the EIS fitting results of the cement/casing steel system specimens under atmospheric-pressure conditions. The cement resistance reaches its maximum value at 28 days and then decreases with increasing immersion time. This result is because the $CaCO_3$ generated by the carbonization reaction at the early stage of cement corrosion covers the cement surface, resulting in decreased cement porosity. After that, the gel material generated by the hydration reaction at the early stage of cement, thereby increasing the cement resistance. After 28 days of corrosion, the hydration reaction disappears. Under the action of CO_2 , the cement undergoes a leaching process and the cement pores increase in size, which decreases the cement resistance.

Table 2. Impedance fitting parameters under normal pressure

Immersion	R ₁	Q1	10 .	R ₂	Q2	10 -	R ₃	W
time	$(R \cdot cm^2)$	(F/cm^2)	11]	$(R \cdot cm^2)$	$(F \cdot cm^{-2})$	112	$(R \cdot cm^2)$	$(S \cdot sce^{0.5}/cm^2)$
7d	303.8	2.558×10 ⁻⁴	0.7453	48.27	2.524×10 ⁻⁴	0.7307	9481	1.67×10 ⁻³
14d	239.4	3.051×10 ⁻⁴	0.223	298.8	4.526×10 ⁻⁴	0.5796	4583	2.773×10 ⁻³
21d	279.2	1.177×10 ⁻⁴	0.2842	334.7	4.987×10 ⁻⁴	0.5808	4493	2.373×10 ⁻³
28d	195.4	7.45×10 ⁻⁵	0.2358	563.3	4.776×10 ⁻⁴	0.5255	3977	2.286×10 ⁻³
35d	137.2	6.126×10 ⁻⁶	0.412	306.3	4.75×10 ⁻⁴	0.3933	2471	6.206×10 ⁻³
42d	119.7	1.655×10 ⁻⁴	0.184	489.7	4.743×10 ⁻⁴	0.483	2368	9.146×10 ⁻³
49d	46.49	1.09×10 ⁻⁴	0.1879	496	6.96×10 ⁻⁴	0.4188	1916	7.871×10 ⁻³

(2) EIS of the cement/casing system under high-pressure conditions

Regarding polarization resistance R_3 , the polarization resistance gradually decreases with increasing immersion time and reaches a minimum value at 49 days of corrosion. The corrosion rate of the casing steel gradually increases with increasing immersion time. The Warburg impedance gradually increases with increases with increases with increase in the corrosion rate of the casing steel.

Figures 5 and 6 show the Nyquist plots and Bode plots, respectively, of the cement/casing steel specimens corroded for different times under high-pressure conditions. In Figure 5, it can be seen that the impedance spectrum consists of two semicircular capacitive resistance arcs. As the immersion time is increased, the radius of the capacitive resistance arc in the low-frequency region gradually decreases, indicating that the corrosion rate of the casing steel gradually increases. According to the magnified high-frequency image in Figure 5(b), Warburg impedance appears during the corrosion test from 7 days to 49 days. This is because in the cement/casing steel system, the corrosive solution cannot directly contact the surface of the casing steel; rather, the corrosive solution can reach only the cement/casing interface. As Figure 6 shows, the corrosion from 7 days to 49 days contains two time constants. The fitting plot of the EIS data for the equivalent circuit shown in Figure 4 demonstrates that the fitted plot overlaps well.



Figure 5. Electrochemical impedance spectra for different immersion times under high-pressure conditions: (a) Nyquist diagram under high-pressure conditions and (b) Nyquist diagram of the high-frequency amplification area

Table 3 shows the EIS fitting results under high-temperature–high-pressure conditions. Table 3 shows that R_2 reaches its maximum value at 28 days and then gradually decreases with increasing immersion time. This corrosion law is consistent with the experimental results under atmospheric conditions, while the resistance R_2 of cement under high-pressure conditions is obviously smaller than that under atmospheric conditions. These results indicate that the corrosion of cement is more severe under high-pressure conditions, thus weakening the protective properties of the cement. In addition, it can be certain that the hydration process of the high-density cement with hematite powder is completely finished after 28 days. After 28 days, the protective properties of the cement gradually deteriorate with increasing immersion time. The polarization resistance R_3 gradually decreases with increasing immersion time, indicating that the corrosion rate gradually increases. This is because under high-pressure conditions, the pressure promotes the penetration of corrosive media, such as CO₂, into the

cement, allowing this media to reach the cement/casing steel interface and cause the casing steel to corrode.



Figure 6. Bode plots with different immersion time under high-pressure conditions

Table 3. Impedance fitting parameters under high-pressure conditions

Corrosion	R ₁	Q1	10	R ₂	Q2	1 0 -	R ₃	W
time	$(R \cdot cm^2)$	(F/cm^2)	11]	$(R \cdot cm^2)$	$(F \bullet cm^{-2})$	112	$(R \cdot cm^2)$	$(S \cdot sce^{0.5}/cm^2)$
7 d	72.78	3.5×10 ⁻³	0.2861	100.2	6.079×10 ⁻⁴	0.6616	5849	2.888×10 ⁻³
14 d	119.8	4.768×10 ⁻⁵	0.277	211.3	3.108×10 ⁻⁴	0.6613	3825	1.26×10 ⁻²
21 d	233.8	1.226×10 ⁻⁶	0.5786	164.6	4.699×10 ⁻⁴	0.5962	2790	3.725×10 ⁻³
28 d	248.6	4.213×10 ⁻⁶	0.4399	335.7	2.691×10 ⁻⁴	0.6035	2733	6.379×10 ⁻³
35 d	181.8	2.093×10 ⁻⁶	0.5202	236.6	5.969×10 ⁻⁴	0.62	1380	3.68×10 ⁻³
42 d	181.5	2.767×10 ⁻⁶	0.5277	161.4	8.539×10 ⁻⁴	0.5116	454.6	9.565×10 ⁻³
49 d	177.1	3.566×10 ⁻⁶	0.5344	150.5	1.175×10 ⁻³	0.5269	327.5	7.387×10 ⁻³

3.1.3 Linear polarization resistance (LPR)

Figure 7 shows the LPR fitting results of the high-density cement/casing steel system under highpressure and atmospheric-pressure conditions for different immersion time. The LPR is the slope of the linear polarization curve at i = 0 [16]. From Figure 7, the polarization resistance of casing steel in cement decreases gradually with increasing immersion time under both experimental conditions, which agrees with the electrochemical impedance spectroscopy results. The polarization resistance under hightemperature–atmospheric-pressure conditions reaches a minimum value of 1890.05 $\Omega \cdot cm^2$ at 49 days. whereas the minimum polarization resistance value under high-pressure conditions is 817.29 $\Omega \cdot cm^2$, indicating that the corrosion of casing steel is more severe under high-pressure conditions and that the pressure will make it easier for corrosive media to reach the surface of the casing steel.



Figure 7. Linear polarization resistance with different immersion time under high and normal pressure

The linear polarization resistance is calculated using equation 2 for the self-corrosion current density i_{corr} . In the equation, B is a constant, and for steel in cement in a passivated state, B is 52 mV; in contrast, when the steel is in an active state of corrosion, B is 26 mV [17]. According to the previous corrosion potential measurement results, the casing steel in simulated formation water with saturated CO₂ is corroded. Afterward, the corrosion rate of the casing steel is calculated using equations [17] 3–4, and the results are shown in Figure 8.

$$i_{corr} = \frac{b_a b_c}{2.3(b_a + b_c)} \times \frac{1}{R_p} = \frac{B}{R_p}$$
(2)
$$B = \frac{b_a b_c}{2.3(b_a + b_c)}$$
(3)

where i_{corr} represents the corrosion current, b_a represents the anodic Tafel slope, b_c represents the cathodic Tafel slope, R_p is the polarization resistance, and B is a constant.

$$C_R = \frac{3.27i_{corr}\mu}{n\delta} = \alpha i_{corr} \tag{4}$$

In the above equation, C_R is the corrosion rate in μ m·year⁻¹, μ is the atomic weight, n is the number of atoms gained or lost, δ is the density of the metal material, i_{corr} is the corrosion current density, and α is a constant; for steel, the value of α is 11.6 μ A⁻¹·cm²· μ m·year⁻¹[17].

Figure 8 shows the calculation results. The longer the immersion time is, the greater the corrosion current density (i_{corr}) and corrosion rate (C_R). The corrosion rate under atmospheric conditions can reach a maximum of 159.5 μ m·year⁻¹, while the corrosion rate under high-pressure conditions can reach a maximum of 378.51 μ m·year⁻¹, which is equivalent to 2.4 times that under atmospheric conditions.



Figure 8. Corrosion current density and corrosion rate with different immersion time under high and normal pressure: (a) corrosion current density and (b) corrosion rate

3.2 Microstructural evolution during the test

3.2.1 Microstructure at the cement/casing steel interface

The cement/casing steel interface specimens were obtained by cutting through the middle of the cement casing steel specimen by using a SYJ-160 diamond cutter with a slice thickness of 1 cm. Figure 9(b) shows a schematic diagram of the cutting position. For better scanning electron microscopy (SEM) observation, the cement around the casing steel specimen was removed, and then the cross-section was sandpapered and rinsed with alcohol and dried with cold air. The specimens were obtained as shown in Figure 9.



Figure 9. Specimen for observing the cement/steel interface

Figure 10 shows the microscopic morphology at the cement/casing steel interface. There is no obvious accumulation of corrosion products at the interface, but the demarcation between the cement and casing steel is evident and forms a 1 μ m gap at the interface. In addition, the presence of CaCO₃ crystals is observed at the interface, demonstrating that the carbonation of cement has reached the innermost layer.



Figure 10. Microscopic morphology at the cement/casing steel interface with immersion time of 49 days under high pressure: (a) 500x and (b) 3000x

Figure 11 and Table 4 show the EDS measurement results of corrosion products at the cement/casing steel interface. The corrosion products at the interface mainly contain C, O and Fe. According to the corrosion mechanism of CO_2 on the casing steel, it is presumed that the corrosion products at the interface are mainly FeCO₃.



Figure 11. Location of the EDS test at the cement/casing steel interface

Element	Wt.%	Atomic%		
С	9.56	29.50		
О	6.03	13.98		
C1	0.13	0.14		
Ca	1.63	1.51		
Fe	82.65	54.88		
Total	100.00	100.00		

Table 4. EDS measurements of the corrosion products at the cement/casing steel interface

3.2.2 Surface morphology of the casing steel

The surface morphology of the casing steel after corrosion is shown in Figure 12. After 28 days of corrosion, the casing steel surface exhibits obvious crack-like corrosion products. In Figure 12(c), after 28 days, $CaCO_3$ crystals are found on the surface of the casing steel, indicating that some of the

cement carbonation reaction products stick to the casing steel surface. When the immersion time is increased to 49 days (Figure 12d), corrosion products are observed on the casing steel surface. The crack width increases significantly, indicating more severe corrosion, which is consistent with the electrochemical test results.



- **Figure 12.** Microscopic morphology of casing steel corrosion for different times under high-pressure conditions:(a) 0 d, (b) 21 d, (c) 28 d, and (d) 49 d
- 3.3 Analysis of the corrosion products
- 3.3.1 EDS analysis



Figure 13. EDS image of the corrosion product film on the casing steel surface

Figure 13 and Table 5 show the EDS results of the corrosion products on the surface of the casing steel. The elements in the corrosion products are mainly C, O and Fe, indicating that the casing steel is mainly subjected to corrosion by CO_2 and that the corrosion product is $FeCO_3$.

Element	Wt.%	Atomic%
С	8.49	25.25
Ο	9.75	21.76
Si	0.63	0.80
Cl	0.62	0.62
Ca	0.39	0.35
Fe	80.12	51.22

Table 5. EDS results of the corrosion products on the casing steel surface

3.3.2 Raman test

Figure 14 shows the Raman spectroscopy results of the corrosion products on the surface of the casing steel. The three Raman peaks at 224 cm⁻¹, 502 cm⁻¹ and 640 cm⁻¹ represent the partial surface oxidation of the FeCO₃ crystal [18]. The peak at 290 cm⁻¹ corresponds to FeCO₃ [19], and the peak at 410 cm⁻¹ represents Fe₂O₃ [20], which is probably due to some of the hematite-containing cement adhering to the surface of the casing steel.



Figure 14. Raman spectroscopy results of the corrosion products on the casing steel surface

3.3.3 X-ray photoelectron spectroscopy (XPS) analysis

Figure 15 shows the XPS analysis results, including the full XPS spectrum and the peak fitting diagram of each element. Figure 15(a) shows that the corrosion products on the surface of the casing steel contain C, Fe, O and small amounts of Ca and Cl. Figure 15(b) shows that the fitted C 1s peak contains two peaks, 284.9 eV for the C atom and 288.9 eV for the carbon in FeCO₃ [21]. The XPS peaks of Fe are shown in Figure 15(c), and the main fitted peaks are at 711.2 eV, 712 eV, 723.7 eV and 724.7 eV. The peaks at 712 eV and 723.7 eV represent the Fe²⁺ in FeCO₃ [22,23], and the peaks at 711.2 eV and 724.7 eV represent the Fe³⁺ in Fe₂O₃ [22]. Figure 15(d) shows that there are two O peaks. The peak at 531.9 eV is the oxygen in $CO_3^{2^-}$, and the peak at 530.2 eV is the O²⁻ in Fe₂O₃ [22]. The Fe₂O₃ in the

corrosion products may come from the hematite powder in the cement adhered to the surface of the casing steel.



Figure 15. Full XPS spectrum of the corrosion products and the fitting results of the elemental spectra: (a) XPS full spectrum, (b) C 1s, (c) Fe 2p, and (d) O 1s

4. CONCLUSION

(1) The electrochemical impedance spectrum results show two time constants. With increasing immersion time, the cement resistance R_2 first increases and then decreases, the casing steel polarization resistance R_3 gradually decreases, and the corrosion rate of casing steel in cement gradually decreases.

(2) The pressure will promote the corrosion reaction; thus, the corrosion potential decreases and the corrosion rate increases.

(3) Cement casing steel is mainly subjected to CO_2 corrosion, the corrosion product is FeCO₃, and the microscopic morphology of corrosion consists of distributed cracks.

(4) A microgap of 1 μ m exists at the cement/casing steel interface after corrosion and is accompanied by the formation of FeCO₃.

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