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**Study on stress corrosion behavior of C110 steel by electrochemical hydrogenation**

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**Abstract**

In this study, the effects of hydrogen on the electrochemical behavior and stress corrosion behavior of C110 steel in CO₂ environment was explored by electrochemical hydrogen charging, four-point bending stress loading and electrochemical test. The results show that with the raise of applied stress, the film resistance, transfer resistance of corrosion products decrease, and $I_{corr}$ increases. After hydrogen charging, C110 steel has greater activity, and the combined action of external stress further promotes the corrosion reaction. The application of stress will destroy the formation of corrosion product film and reduce its protection. However, when the stress is further increased to 102% $σ_s$, the larger grains will be broken into smaller grains, so that the surface area of the film will be increased again and the protection of the substrate will be enhanced. The main corrosion products analyzed by XRD are $\alpha$-Fe, Fe₂O₃ and MgFe₂O₄. The applied stress will not change the phase composition of the corrosion products.

**1. Introduction**

As the development environment of oil and gas fields becomes more and more demanding, and the service life of oil well casing steel increases, the problem of corrosion failure becomes more and more prominent, which is likely to cause serious economic losses and environmental pollution accidents [1–3]. Among the various protective measures taken, it is safer to use high-strength acid-resistant pipes with corrosion resistance. Among them, C110 steel has been adopted and widely used in the development of H₂S oil and gas fields around the world [4, 5]. However, in the production process, C110 steel is still subject to complex corrosion of CO₂, hydrogen and many complex stresses, causing corrosion and perforation of the casing and even fracture [6–8]. CO₂ corrosion has always been a problem that cannot be underestimated in the development and transportation of oil and gas industry [9–11]. China’s oilfields are generally acid-based wells dominated by CO₂. Acidic media generated by CO₂ in contact with water can cause fatal corrosion damage to pipes [12–15]. The acidic medium includes hydrogen produced in the environment, and hydrogen permeates into the steel, causing hydrogen damage to the casing steel [16, 17]. More research shows that [18, 19] hydrogen atoms participate in the stress corrosion process, promote the emission, proliferation and movement of deformation dislocations caused by the applied stress, accelerate the stress corrosion behavior, and have a great impact on the corrosion of casing steel in acidic environment, Thereby affecting the service life of the casing in the acid mine. In actual conditions, the synergistic effect of CO₂, hydrogen and stress on the casing steel will cause more damage, but there is currently no comprehensive research on this. Therefore, the electrochemical hydrogenation and four-point bending stress loading experiments were used to study the effects of electrochemical hydrogenation on the electrochemical corrosion behavior and stress corrosion behavior of C110 steel in saturated CO₂ simulated production water environment. It provides a certain reference value for the safe service of C110 steel in actual working conditions.
2. Experimental

2.1. Materials
The experiment used C110 steel material, and its composition and mass fraction (%) were C 0.28, Si 0.31, Mn 0.60, P 0.01, S 0.002, Cr 0.8, Ni 0.59, Mo 0.45. The size of the sample was 106 mm × 15 mm × 3 mm. The sample was sanded to 1200# with water sandpaper, washed with acetone and absolute ethanol, and dried by cold air.

2.2. Electrochemical hydrogenation experiment
In order to investigate the effect of hydrogen on the electrochemical and stress corrosion behavior of C110 steel, the sample was subjected to electrochemical hydrogenation treatment by constant current method. The experimental apparatus is shown in figure 1. The hydrogen-filled sample serves as a cathode and the platinum electrode serves as an anode. Hydrogen charging in a 0.5 mol·l⁻¹ NaOH + 1 g·l⁻¹ thiourea solution for 2 h, the hydrogen charging current density is 15 mA·cm⁻². After the end of hydrogen charging, the sample was washed with deionized water and absolute ethanol, dried and subjected to electrochemical and four-point bending experiments.

2.3. Four-point bending stress loading experiment
The tensile mechanical properties of C110 steel after electrochemical hydrogen charging were tested by SHT4605 microcomputer controlled electro-hydraulic servo universal testing machine. The stress-strain relationship curve obtained is shown in figure 2. The yield strength \(\sigma_s\) of C110 steel after hydrogen charging was 459 MPa and the tensile strength \(\sigma_b\) was 678 MPa.

In this paper, the four-point bending stress loading method is used to quantitatively load the C110 steel after hydrogen charging [20–22]. According to the mechanical properties of C110 steel after hydrogen charging, the different stress states and parameters of loading are shown in table 1. As shown in figure 3, the experimental fixture is used to ensure the loading stress by adjusting the distance of the deflection \(y\) in the device. The formula for calculating the \(y\) value is as shown in equation (1):

\[
y = \frac{\sigma (3H^2 - 4A^2)}{12Et}
\]

Where \(\sigma\)-the maximum tensile stress of the specimen, Pa; \(E\)-the modulus of elasticity of the material, Pa; \(t\)-the thickness of the specimen, m; \(y\)-the maximum deflection between the outer supports, m; \(H\)-the distance between the inner and outer supports, m; \(A\)-the distance between the outer supports, m.

The sample after electrochemical hydrogen charging was loaded into a four point bending fixture and loaded with different stresses, and then immersed in the experimental solution for 120 h. The experimental temperature is 90 \(^°\)C. The experimental solution is the simulated produced water containing saturated CO\(_2\). According to the composition and mass concentration shown in table 2, 2.5 l of the simulated produced water is prepared. After deoxygenation with nitrogen for 4 h, CO\(_2\) gas is introduced to saturation. At the end of the experiment, the samples were taken out from the fixture, washed with deionized water and anhydrous ethanol, dried, and tested by electrochemistry, SEM and XRD at the center of the stress concentration area of the corroded samples.
2.4. Electrochemical experiment

In this paper, CS310 electrochemical workstation in Wuhan Koster was used for the test. Three-electrode system was used. Saturated calomel electrode and platinum electrode were used as reference electrode and auxiliary electrode respectively, and the sample was used as working electrode. The sample to be tested was sealed with paraffin, leaving only 1 cm² of test area. The sample was immersed in 3.5% NaCl solution for 30 min after being connected into the system. After the open circuit potential was stabilized, the electrochemical impedance spectroscopy test and the potentiodynamic polarization curve test were carried out, and the experimental temperature was 25 °C.

![Stress-strain curve of C110 steel after hydrogen charging.](image)

**Table 1.** Load different stress value parameters of C110 steel after hydrogen charging.

| Sample | $\sigma_s$ (MPa) | 0% $\sigma_s$ (MPa) | 40% $\sigma_s$ (MPa) | 60% $\sigma_s$ (MPa) | 80% $\sigma_s$ (MPa) | 102% $\sigma_s$ (MPa) |
|--------|----------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| C110   | 459            | 0                 | 183.6             | 275.4             | 367.2             | 468.18             |

![Constant strain four-point bending stress loading fixture diagram.](image)

**Table 2.** Composition and concentration of simulated produced water.

| Composition | Na₂CO₃ | NaHCO₃ | Na₂SO₄ | CaCl₂ | MgCl₂ | KCl | NaCl | HAc |
|-------------|--------|--------|--------|-------|-------|-----|------|-----|
| Concentration (g·l⁻¹) | 0.0322 | 3.0638 | 1.1097 | 0.5053 | 1.6603 | 0.3553 | 16.6172 | 1 ml |

2.4. Electrochemical experiment

In this paper, CS310 electrochemical workstation in Wuhan Koster was used for the test. Three-electrode system was used. Saturated calomel electrode and platinum electrode were used as reference electrode and auxiliary electrode respectively, and the sample was used as working electrode. The sample to be tested was sealed with paraffin, leaving only 1 cm² of test area. The sample was immersed in 3.5% NaCl solution for 30 min after being connected into the system. After the open circuit potential was stabilized, the electrochemical impedance spectroscopy test and the potentiodynamic polarization curve test were carried out, and the experimental temperature was 25 °C.
The EIS scan frequency range is 100 kHz to 0.01 Hz and the AC signal is a sine wave with amplitude of 10 mV. The scanning range of polarization curve is $-1 \text{ V} \sim +1 \text{ V}$ versus $E_{\text{corr}}$, and the scanning rate is $1 \text{ mV} \cdot \text{s}^{-1}$.

ZSimpwin 3.30 software was used to fit the original impedance data. CView2.0 software was used to fit the original polarization curve data.

2.5. Surface analysis
Using Zeiss EVO/MA15 scanning electron microscope (SEM), the surface morphology of C110 steel loaded with different stress after electrochemical hydrogen charging was observed after 120 h of corrosion in saturated CO$_2$ simulated produced water.

Using Dandong Haoyuan DX-2700B x-ray diffractometer (XRD), the phase of C110 steel loaded with different stress after electrochemical hydrogen charging was observed after 120 h of corrosion in saturated CO$_2$ simulated produced water. The scanning angle ranged from 10° to 90°.

3. Results

3.1. Effect of electrochemical hydrogenation on electrochemical behavior of C110 steel
3.1.1. Electrochemical impedance spectroscopy
Figure 4 shows the impedance of C110 steel before and after electrochemical hydrogen charging. It can be seen that the shape of Nyquist diagram in figure 4(a) before and after hydrogen charging is roughly the same, which is composed of high frequency capacitive arc and a section of inductive arc with smaller radius in low frequency area, but the radius of capacitive arc after hydrogen charging is obviously reduced. The smaller the radius of capacitive reactance is, the smaller the resistance of electrochemical reaction is, and the more likely the steel is to be corroded [23]. It is precisely because the hydrogen atom enters into the metal grain and defects appear, which
makes the local plastic change of the matrix, resulting in the decrease of the resistance when corrosion occurs in the solution. According to the Bode diagram in figure 4(b), the EIS spectrum before and after hydrogen charging has two time constants, and the curve has no obvious change before and after hydrogen charging. It shows that the electrochemical hydrogen charging reaction is controlled by activation, which does not change the mechanism of electrode reaction process, but greatly promotes the corrosion process and reduces the corrosion resistance of C110 steel [24, 25].

See table 3 for EIS parameter values fitted using the equivalent circuit model of figure 5. Among them, \( R_s \) is solution resistance, \( Q_{dl(1)} \) is corrosion product film capacitance, \( R_t \) is transfer resistance, \( Q_{dl(2)} \) is double electric layer capacitance and \( L \) is inductive reactance. Combined with table 3, it can be seen that \( R_t \) of C110 steel becomes smaller after hydrogen charging, from 1782 \( \Omega \cdot \text{cm}^2 \) to 1196 \( \Omega \cdot \text{cm}^2 \), while \( R_t \) represents the difficulty of charge transfer process. The lower resistance indicates that hydrogen charging accelerates the electrode reaction rate of C110 steel in solution and promotes the reaction [26]. However, the capacitance of the double layer increases from 7.925 \( \times 10^{-5} \Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-1} \) without hydrogen charging to 1.92 \( \times 10^{-4} \Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-1} \) after hydrogen charging for 2 h, indicating that the corrosion resistance of the steel decreases after electrochemical hydrogen charging. This should be due to the presence of hydrogen in the surface grains of the metal, which improves the activity of the steel, reduces the resistance of mass transfer process, accelerates the reaction, and reduces the corrosion resistance of the steel [27, 28].

**3.1.2. Potentiodynamic polarization curves test**

Figure 6 shows the potentiodynamic polarization curve of C110 steel before and after hydrogen charging. It can be seen that after hydrogen charging, the self-corrosion potential is significantly negative shifted, and the cathode and anode slopes of the curve are steeper, which shows that the electrochemical hydrogen charging promotes the dissolution of matrix anode to a certain extent, and reduces the corrosion resistance of C110 steel. This is mainly due to the diffusion of hydrogen atoms into the matrix after hydrogen charging, which will occupy the gap position of the grain or be captured by reversible hydrogen traps in the steel, so that hydrogen is easy to react at the interface of the steel and the solution, resulting in the increase of lattice distortion energy at the grain boundary, the increase of activity on the steel surface, and the decrease of electrode potential, which can promote the whole electrode reaction, and make the metal more vulnerable to corrosion.

With the analysis of the fitting parameter values in table 4, combined with the data analysis, the absolute value of the slope \( b_s \) of the two curves before and after hydrogen filling is greater than the absolute value of \( b_a \), indicating that the reaction process is controlled by the cathode, and the cathode reaction is dominant. The slope of \( b_a \) and \( b_s \) increased after electrochemical hydrogen charging, and the current density of self-corrosion increased with hydrogen charging, while the potential of self-corrosion decreased after hydrogen charging, \( I_{corr} \) increased from 3.3492 \( \mu \text{A} \cdot \text{cm}^{-2} \) to 10.53 \( \mu \text{A} \cdot \text{cm}^{-2} \), corresponding \( E_{corr} \) were −644.78 mV and −902.54 mV, respectively, which showed that hydrogen infiltration promoted the cathodic and anodic reactions, and

**Table 3. EIS fitting parameters of C110 steel before and after electrochemical hydrogen charging.**

| Sample | \( R_s (\Omega \cdot \text{cm}^2) \) | \( Q_{dl(1)} (\Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-1}) \) | \( n \) | \( Q_{dl(2)} (\Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-1}) \) | \( n \) | \( R_t (\Omega) \) | \( L (\text{H} \cdot \text{cm}^{-2}) \) |
|--------|------------------|------------------|------|------------------|------|------------------|------------------|
| Before | 6.281            | 6.545 \( \times 10^{-4} \) | 0.821| 7.925 \( \times 10^{-5} \) | 1    | 1782             | 4.206 \( \times 10^4 \) |
| After  | 5.171            | 7.783 \( \times 10^{-4} \) | 0.771| 1.92 \( \times 10^{-4} \) | 1    | 1196             | 2.052 \( \times 10^4 \) |

**Figure 5. Equivalent circuit diagram of C110 steel before and after electrochemical hydrogen charging.**
hydrogen charging promoted the corrosion of C110 steel. The corrosion resistance of the steel will also decrease with the permeation of hydrogen.

3.2. Effect of electrochemical hydrogenation on stress corrosion

3.2.1. Electrochemical impedance spectroscopy

The EIS spectrum of C110 steel after electrochemical hydrogenation since different stresses is shown in figure 7. Figure 7(a) is Nyquist map. Combining with figure 7(b) Bode map, it can be inferred that the reaction has two time constants. There is only capacitive arc in each curve, and the formation of capacitive arc may be related to the corrosion product film on the surface of metal matrix or to the double electric layer in corrosive medium [29, 30]. From figure 7(a), it can be seen that the radius of capacitive arc loaded with tensile stress is nearly half smaller than that of capacitive arc without stress, and with the increase of stress, the radius is also gradually decreasing.

See table 5 for EIS parameter values fitted using the equivalent circuit model of figure 8. Among them, $R_s$ is solution resistance, $Q_{dl}$ is constant phase angle element, $n$ is constant phase angle parameter, $R_f$ is resistance of corrosion product film and $R_t$ is transfer resistance. With the increase of applied stress, $R_t$ and $R_f$ in the reaction have decreasing trends. When the applied stress increases from 0% $\sigma_s$ to 40% $\sigma_s$, the transfer resistance decreases from 4625 $\Omega \cdot cm^2$ to 2389 $\Omega \cdot cm^2$, and the corresponding corrosion product film resistance decreases from 4814 $\Omega \cdot cm^2$ to 3431 $\Omega \cdot cm^2$. When the stress increases to 102% $\sigma_s$ of the plastic deformation, $R_t$ decreases to 1653 $\Omega \cdot cm^2$, and the corresponding $R_f$ decreases to 2317 $\Omega \cdot cm^2$, which is 64.25% and 51.87% lower than that when no stress is applied. Since the action of stress, the structure of the corrosion product film is destroyed, the protection of the film to the metal is reduced, the transfer resistance and film resistance in the solution reaction system are reduced, the charge transfer becomes easier, the corrosion rate of C110 steel in the corrosion medium is increased, and the further corrosion is promoted.

3.2.2. Potentiodynamic polarization curves test

The potentiodynamic polarization curve of C110 steel after electrochemical hydrogenation since different stresses is shown in figure 9. The trend of the five polarization curves in the figure is almost the same. There is bending on the anode polarization curve, which is not smooth. This feature is more obvious on the curve after applying stress. This may be because the thickness of the corrosion product film produced after corrosion is thinner and less dense, which is not protective to the substrate. Excessive anode over potential produces a large current density, allowing the corrosive medium to penetrate the corrosion product film to reach the surface of the metal substrate, while the surface of the C110 steel substrate after electrochemical hydrogen charging has
Figure 7. Impedance spectrum of C110 steel after hydrogen charging corroded in a saturated CO₂ environment for 120 h since stress.

Table 5. EIS fitting parameters of C110 steel after hydrogen charging corroded in a saturated CO₂ environment for 120 h since stress.

| Tensile stress | \( R_s (\Omega \cdot \text{cm}^2) \) | \( Q_{dl(1)} \) (\( \Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-n} \)) | \( n \) | \( R_t (\Omega \cdot \text{cm}^2) \) | \( Q_{dl(2)} \) (\( \Omega^{-1} \cdot \text{cm}^{-2} \cdot \text{S}^{-n} \)) | \( n \) | \( R_t (\Omega \cdot \text{cm}^2) \) |
|----------------|----------------------------------|----------------------------------|------|----------------------------------|----------------------------------|------|----------------------------------|
| 0%\( \sigma_s \)     | 11.733                           | \( 7.21 \times 10^{-4} \)     | 0.627| 4814                             | \( 8.885 \times 10^{-5} \)     | 0.2254| 4625                             |
| 40%\( \sigma_s \)    | 10.984                           | \( 1.878 \times 10^{-3} \)     | 0.8  | 3431                             | \( 2.143 \times 10^{-4} \)     | 0.5053| 2389                             |
| 60%\( \sigma_s \)    | 9.018                            | \( 1.405 \times 10^{-3} \)     | 0.4124| 3109                             | \( 4.115 \times 10^{-4} \)     | 0.3541| 2185                             |
| 80%\( \sigma_s \)    | 8.201                            | \( 6.704 \times 10^{-2} \)     | 0.6547| 2819                             | \( 3.578 \times 10^{-5} \)     | 0.336 | 1539                             |
| 102%\( \sigma_s \)   | 9.496                            | \( 1.14 \times 10^{-2} \)      | 0.8  | 2317                             | \( 4.908 \times 10^{-4} \)     | 0.5813| 1653                             |

Figure 8. Equivalent circuit diagram of C110 steel after hydrogen charging corroded in a saturated CO₂ environment for 120 h since stress.
greater activity and acts at a higher current density. Since the action of larger current density, the corrosion solution reacts with it more quickly to form new corrosion products which are adsorbed on the steel surface.

With the analysis of the fitting parameter values in table 6, combined with the data analysis, it is found that the absolute value of Tafel slope $b_{bc}$ of C110 steel under stress is greater than that of $b_{ba}$, indicating that the reaction process is controlled by the cathode, and the cathode reaction is dominant. The self-corrosion current density $I_{corr}$ increased from 6.3598 $\mu$A·cm$^{-2}$ when no stress was applied to 24.974 $\mu$A·cm$^{-2}$ when 102%σs tensile stress was applied, and reached the maximum value, an increase of 3.93 times. The larger stress will cause the corrosion product film of C110 steel to crack and produce defects, and the hydrogen atom infiltrated in the electrochemical hydrogen charging process will be captured by the defects, further promoting the corrosion reaction, reducing the corrosion resistance of C110 steel, resulting in the increase of self-corrosion current density.

3.2.3. Scanning electron microscopy

The surface corrosion morphology of C110 steel after electrochemical hydrogenation since different stresses is shown in figure 10. It can be seen from the figure that the corrosion product film formed by C110 steel under the condition of no load stress covers the substrate surface completely and evenly. With the increasing of tensile stress, the grain size of the product film gradually decreases. When the applied tensile stress increases from 6.3598 $\mu$A·cm$^{-2}$ when no stress was applied to 24.974 $\mu$A·cm$^{-2}$ when 102%σs tensile stress was applied, and reached the maximum value, an increase of 3.93 times. The larger stress will cause the corrosion product film of C110 steel to crack and produce defects, and the hydrogen atom infiltrated in the electrochemical hydrogen charging process will be captured by the defects, further promoting the corrosion reaction, reducing the corrosion resistance of C110 steel, resulting in the increase of self-corrosion current density.

### Table 6. Polarization curve parameters of C110 steel after hydrogen charging corroded in a saturated CO$_2$ environment for 120 h since stress.

| Tensile stress | $E_{corr}$ (mV) | $I_{corr}$ ($\mu$A·cm$^{-2}$) | $b_{bc}$ (mV-decade$^{-1}$) | $b_{ba}$ (mV-decade$^{-1}$) |
|---------------|----------------|-------------------------------|----------------------------|-----------------------------|
| 0%σs         | −981.33        | 6.3598                        | 150.68                     | −186.74                     |
| 40%σs        | −981.26        | 8.5408                        | 140.46                     | −144.74                     |
| 60%σs        | −982.98        | 12.528                        | 146.41                     | −163.41                     |
| 80%σs        | −983.58        | 23.339                        | 133.48                     | −179.87                     |
| 102%σs       | −987.56        | 24.974                        | 134.96                     | −170.87                     |

Figure 9. Polarization curve of C110 steel after hydrogen charging corroded in a saturated CO$_2$ environment for 120 h since stress.
3.2.4. XRD phase analysis

The XRD pattern of C110 steel after electrochemical hydrogenation since different stresses is shown in figure 11. Through the peak seeking analysis of XRD curve by JADE software, it can be found from figure 11 (a) that the trend of characteristic diffraction peaks of the five curves is relatively similar, indicating that the application of tensile stress will not change the phase composition of corrosion products. However, with the increase of stress, the relative strength of diffraction peak will be slightly smaller than that without stress, which indicates that the tensile stress makes the crystallinity of the grains on the corrosion product film worse and destroys the structure of the product film. According to the comparison in figure 11 (b), it can be seen that the main corrosion products are α-Fe, Fe₂O₃ and MgFe₂O₄.

4. Conclusions

(1) After electrochemical hydrogen charging, the \( R_\text{t} \) of C110 steel decreased and the capacitance of double electric layer increased. \( I_{\text{corr}} \) increased from 3.3492 \( \mu \text{A} \cdot \text{cm}^{-2} \) to 10.53 \( \mu \text{A} \cdot \text{cm}^{-2} \), and the corresponding \( E_{\text{corr}} \) decreased from \(-644.78 \text{ mV}\) to \(-902.54 \text{ mV}\). The permeation of hydrogen atom after hydrogen filling can promote the corrosion of C110 steel and reduce the corrosion resistance of steel.

Figure 10. SEM of C110 steel after hydrogen charging corroded in a saturated CO₂ environment for 120 h since stress.
(2) Since the action of stress, C110 steel after hydrogen charging corroded in a saturated CO$_2$ environment for 120 h. With the increase of applied stress, the $R_f$ and $R_t$ decreased, and $I_{corr}$ increased with the increase of stress. After hydrogen charging, C110 steel has greater activity, and the combined action of external stress further promotes the corrosion reaction.

(3) The application of stress will destroy the formation of corrosion product film and reduce its protection. However, when the stress is further increased to plastic deformation, the larger grains in the material will be broken into smaller grains, so that the surface area of the film will be increased again and the protection of the substrate will be enhanced.

(4) The main corrosion products analyzed by XRD are $\alpha$-Fe, Fe$_2$O$_3$ and MgFe$_2$O$_4$. The applied stress will not change the phase composition of the corrosion products, but will destroy the structure of the corrosion product film and make the crystallinity of its grains worse.

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