Research Article

Experimental Study on Chloride Stress Corrosion Cracking Sensitivity of 316L Stainless Steel

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The slow–strain-rate tensile test was used to study the chloride stress corrosion cracking (SSC) behavior of 316L stainless steel under the simulated condition of a CO_2 gas field in the Tarim Basin, and the effects of CO_2, Cl^- concentration, and temperature on the sensitivity of SCC were discussed. The results show that the increase of CO_2 content has no apparent influence on the chloride SCC sensitivity of 316L stainless steel. With the rise in temperature, chloride SCC's sensitivity increases, and the change in the SCC sensitivity index I_{scc} is noticeable. When the concentration of Cl^- increases, the sensitivity of chloride SCC also increases. The degree of influence of the three factors on the experimental results can be ranked as follows: temperature>Cl^- > CO_2. In this experiment, when the partial pressure of CO_2 is 0.1 MPa, the temperature is 65°C, the concentration of Cl^- is 1,20,000 mg/L, and I_{scc} of 316L tensile samples has a maximum of 26.8%.

1. Introduction

Stress corrosion cracking (SCC) refers to the phenomenon of crack nucleation and expansion and subsequent fracture of materials under the joint action of environmental media and stress. Stress and corrosion are synergistic to promote each other, and they are indispensable. Chloride SCC refers to the fracture phenomenon of metal under corrosion media's combined action containing Cl^- and stress. Failure accidents caused by chloride SCC have occurred in 316L composite pipe.

The factors influencing stress corrosion can be divided into three categories: stress, environment, and metallurgy. The environmental factors, which affect the SCC of metals, are mainly discussed. In general, the higher the temperature is, the stronger the stress corrosion sensitivity will be. Some scholars think that the SCC process of austenitic stainless steel in a chloride corrosion medium can occur only when the temperature exceeds 80°C. But, SCC of austenitic stainless steel was also found at room temperature. Cheng [1] believes that the higher the dissolved oxygen content is in the solution, the more easily SCC can occur. In a fully deoxygenated corrosion medium, SCC is rare. Ueda et al. [2] found that the super 13Cr stainless steel is susceptible to SCC

in the completion fluid of $CaCl_2$ containing CO_2 and H_2S gas. Mancia and Liu [3, 4] found that 13Cr steel has a high sensitivity to stress corrosion when H_2S exists, and the cracking is inhibited after the injection of CO_2. Li [5] has studied the early electrochemical corrosion behavior at the crack tip of 316L stainless steel in a sodium chloride solution under different stresses. He found a severe stress concentration at the crack tip, along with the stress load raising and anodic dissolution rate increasing, and the crack tip's electrochemical corrosion has a close relationship with the stress concentration and distribution of the crack tip. Li et al. [6] studied the process of 304L stainless steel from pitting to stress corrosion and proposed roughly dividing the SCC process of austenitic stainless steel in the environment containing chloride ions into two stages: (1) damage and pitting of the metal surface passivation film and (2) the bottom of the pit acting as a crack source and expanding to SCC.

2. Experimental Scheme

2.1. Experimental Apparatuses. The main instruments are a universal testing machine (Figure 1(a)), a slow-strain-rate

(a)   (b)   (c)
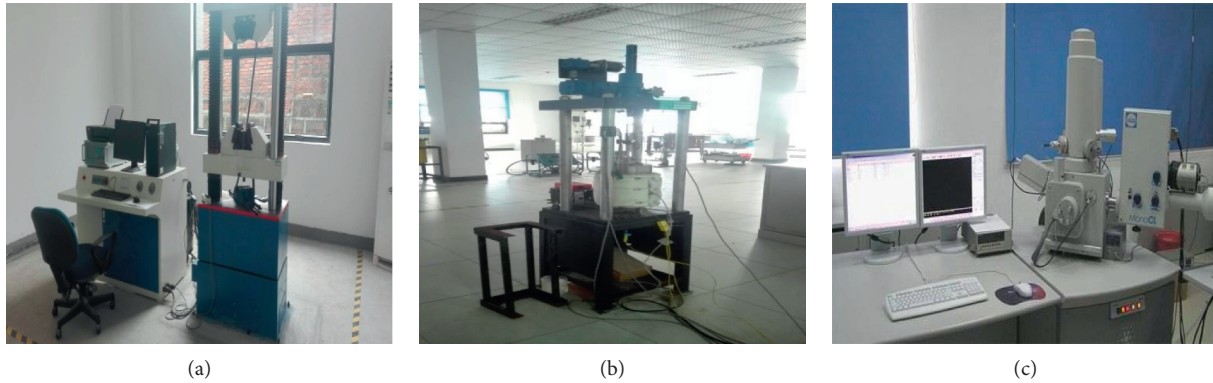
Figure 1: Instruments for stress corrosion experiment: (a) universal testing machine; (b) slow-strain-rate tensile testing machine; (c) scanning electron microscopy.

tensile testing machine (Figure 1(b)), a scanning electron microscopy (Figure 1(c)), a CS electrochemical workstation, and a thermostatic water bath.

2.2. Experimental Sample.
The weld zone is the weak area of corrosion of 316L stainless steel, and 316L welded joints are often sensitive to local corrosion [7, 8]. Therefore, 316L stainless steel samples with weld seams were used as experimental materials. The composition of 316L stainless steel is listed in Table 1. The material components of weld seams are listed in Table 2.

According to the recommended sample size of NACE-TM-0198, the total length of the sample is 80 mm, the gauge length is 25.4 mm, and the diameter of the tensile zone is 3.81 mm. The sample size is shown in Figure 2(a). The samples were polished to a bright color by Grit 400, 600, 800, and 1,200 SiC papers, and then they were cleaned with distilled water and 99.5% ethanol solution. Diameters and lengths of the samples were measured and recorded. The prepared slow tensile test sample is shown in Figure 2(b).

2.3. Experimental Reagent.
The experimental solution should be consistent with the water produced in the field in terms of ion type, pH value, concentration, and total mineralization. Given the experiment's times and feasibility, a water sample produced in the Dabei gas field was used as the experimental reagent in this paper, and four levels of Cl^- concentrations were set according to the testing scheme. The experimental solution was prepared with three-stage distilled water, and pure chemical reagents were used. After preparation, boric acid was used to adjust the pH value to 6.28, and then the experimental solution was placed in a dry and cool place for standby. The composition of the experimental solution is listed in Table 3.

2.4. Experimental Principles and Procedures.
The slow-strain-rate tensile test (SSRT) was used to evaluate the stress corrosion sensitivity of materials. SCC will significantly decrease mechanical performance, such as elongation rate, section shrinkage rate, maximum tensile strength, and fracture absorption energy. The more the factor decreases,

the higher the SCC sensitivity is. At the same strain rate, the shorter the fracture time is, the higher the sensitivity of SCC.

An SSRT determined the stress-strain curve of 316L stainless steel weld samples under simulated conditions of different CO_2 partial pressures, different temperatures, and different Cl^- concentrations. The sample was clamped onto a fixture located in the high-pressure reaction kettle. After the experimental medium was added to the reaction kettle, the top cover was tightened and sealed, the exhaust valve was opened, the air in the reaction kettle was exhausted by adding nitrogen, and then the exhaust valve was closed. Then, quantitative CO_2 was added to increase the kettle's pressure; when the CO_2 partial pressure reached the predetermined value, ventilation was stopped, and the intake valve was closed. The total pressure was controlled at 16 MPa. The experiment temperature was controlled by a heating sleeve outside the reaction kettle. The tensile rate was set as $4e-6\,s^{-1}$. Before the tensile test of slow-strain-rate, a tensile failure experiment of the 316L stainless steel sample was conducted in the air to record its mechanical properties. The experimental data would be used as a blank control for the calculation of stress corrosion sensitivity. The experimental conditions were set as follows:

(1) For the first set of experiments to study the effect of CO_2 partial pressure, the CO_2 partial pressure was set to 0, 0.1, 0.2, and 0.3 MPa, respectively, the temperature was set to 65°C, and the Cl^- concentration was 60000 mg/L.

(2) For the second set of experiments to study the effect of temperature, the temperature was set to 25°C, 45°C, 65°C, and 85°C, respectively, the CO_2 partial pressure was 0.1 MPa, and the Cl^- concentration was 60000 mg/L.

(3) For the third set of experiments to study the effect of Cl^- concentration, the Cl^- concentration was set to 60,000, 80,000, 1,00,000, and 1,20,000 mg/L, respectively, the CO_2 partial pressure was 0.1 MPa, and the temperature was set to 65°C.

2.5. Selection and Calculation of Sensitivity Index.
Based on related research results, the metal SCC sensitivity index (I_{scc})

TABLE 1: Composition of 316L stainless steel.

Composition	C	Cr	Ni	Mo	S	Mn	Si	P	Fe
Percentage	0.025	16.80	13.20	2.38	0.014	1.17	0.029	0.031	66.35

TABLE 2: Material components of weld seams.

Weld bead	Type	Brand	Acceptance standard
Root weld	R316LT1-5	ATS-F316L	AWS A5.22
Seal weld	R309LT1-5	ATS-F309L	AWS A5.22
Transition weld	ER309LMo	ATS-309MoL	AWS A5.9
Fill and cap	E5015	CHE507	GB/T5117-1995

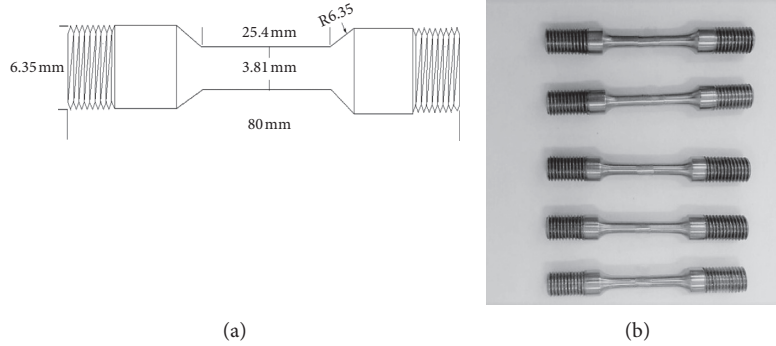(a)   (b)

FIGURE 2: Tensile test samples of slow-strain-rate: (a) sample drawing; (b) sample.

TABLE 3: Ion concentration of reagents and used drugs.

Ion	Concentration (mg/L)	Drugs	Drugs as required per liter (g)
Ca^{2+}	7,030	$CaCl_2$	19.5
Mg^{2+}	760	$MgCl_2 \cdot 6H_2O$	6.4
SO_2^{-4}	670	Na_2SO_4	1.0
HCO^{-3}	900	$NaHCO_3$	0.5
	60,000	NaCl	74.9
	80,000	NaCl	107.8
Cl^-	1,00,000	NaCl	140.7
	1,20,000	NaCl	173.7

can be calculated by comparing the metal's characteristic parameters in the inert medium with those in the corrosive medium. The SCC sensitivity index (I_{scc}) can be calculated according to the following equation:

$$I_{scc} = \frac{I_{air} - I_{corr}}{I_{air}} \times 100\%, \tag{1}$$

where I_{air} represents the experimental parameters in the inert medium and I_{corr} represents the experimental parameters in the corrosive medium.

In this paper, the percentage elongation after fracture, δ_5, of the tensile sample was selected as the SCC sensitivity index, and the chloride stress corrosion sensitivity of the 316L stainless steel tensile sample can be calculated by using the following formula:

$$I_\delta = \frac{\delta_{air} - \delta_{corr}}{\delta_{air}} \times 100\%, \tag{2}$$

where I_δ is the stress corrosion cracking sensitivity index, δ_{air} is the elongation in the inert medium, and δ_{corr} is the elongation in the corrosive medium.

The formula for calculating the elongation after fracture of the sample is

$$\delta_5 = \frac{L_1 - L_0}{L_0} \times 100\%, \tag{3}$$

where L_0 is the starting gauge length of the slow tensile test specimen and L_1 is the gauge length after fracture of the tensile test specimen.

The larger the I_{scc} value, the greater the tendency of metal stress corrosion. According to previous research conclusions [9, 10], the general rules for using I_{scc} to characterize the sensitivity of SCC are as follows: when I_{scc} is <25%, the metal has no apparent stress corrosion tendency; when 25% < I_{scc} < 35%, the metal has a certain degree of stress corrosion tendency; and when I_{scc} is >35%, the metal has an evident stress corrosion tendency.

3. Experimental Results and Analysis

3.1. Blank Tensile Test Results of 316L Stainless Steel. Tensile failure experiments were conducted on the 316L sample in air, and the main mechanical properties of the sample are listed in Table 4.

3.2. Influence of CO_2 Partial Pressure on Stress Corrosion Sensitivity

3.2.1. Experimental Results. The 316L stainless steel weld tensile samples were set at 65°C, and the Cl$^-$ concentration was set to 60,000 mg/L. The stress-strain curve of the sample in the simulated solution under different CO_2 partial pressures is shown in Figures 3. Figure 4 displays the SEM images of fracture surface morphology of tensile test coupons under different CO_2 partial pressure. The test results for the main mechanical properties of 316L stainless steel weld samples under different CO_2 partial pressures are listed in Table 5.

It can be seen from Figure 3 that, with the increase of CO_2 partial pressure, the tensile strength and elongation of the 316L stainless steel weld sample are not apparent. This indicates that CO_2 has little effect on the SCC sensitivity of 316L stainless steel and can be regarded as an "inert gas". From Table 5, it can be seen that, with the increase of CO_2 partial pressure, the maximum tensile strength decreases first and then increases, but the change is small. The extension rate increases slightly but is less than 50%. The section' shrinkage rate and fracture time fluctuate with the increase of CO_2 partial pressure, but the change is not apparent. The section's shrinkage rate is relatively large, which indicates that 316L stainless steel has a strong ability to withstand plastic deformation before fracture in this experiment .

3.2.2. Analysis

(1) At present, for Cr–Ni austenitic stainless steel, the most accepted SCC mechanism is anode slippage dissolution. The occurrence of stress corrosion is directly related to the local dissolution of metal material. In the liquid-phase system containing CO_2, the equilibrium conditions are relatively complex. H_2CO_3, HCO^{-3}, CO_2^{-3}, and CO_2 in the solution will be directly or indirectly involved in the corrosion process as depolarizing agents, which will significantly affect the cathode's progress and rate reaction. Therefore, theoretically, increasing the partial pressure of CO_2 will accelerate the cathode reaction, resulting in accelerated corrosion of metal materials.

(2) However, the results show that, for anodic dissolution stress corrosion, the stress corrosion crack's root is the pitting hole and microcracking on the material's surface. Therefore, the passive film greatly influences the sensitivity of chloride stress corrosion. In the CO_2-saturated environment, 316L has good passive film formation characteristics; that is, CO_2 contributes to forming a $FeCO_3$ passive film on the surface, thus playing a role in inhibiting local corrosion of 316L.

(3) However, under these experimental conditions (65°C and Cl$^-$ = 60,000 mg/L), the surface passive film's nature is not stable and plastic deformation of the tensile specimen is serious by continuous external stress. This leads to constant mechanical damage of the passive film, limiting its ability to protect the 316L matrix metal. Consequently, the results do not change obviously with the increase of CO_2 partial pressure.

(4) Figure 5 shows the SCC sensitivity index (I_{scc}) of 316L stainless steel under different partial pressures of CO_2 calculated according to equation (2). The following conclusions can be obtained from Figure 5. When the partial pressure of CO_2 increases from 0 to 0.3 MPa, the overall change in the stress corrosion sensitivity of the 316L stainless steel tensile sample is not apparent, with a slightly decreasing trend, and the minimum value is 20.2% when P_{co_2} = 0.2 MPa. According to the general rules of SCC sensitivity based on I_{scc} value, no significant chloride SCC sensitivity is shown in the tensile samples of 316L stainless steel under this experimental condition.

3.3. Effect of Temperature on Stress Corrosion Sensitivity

3.3.1. Experimental Results. The 316L stainless steel tensile sample was set in a simulated solution in which the CO_2

TABLE 4: Mechanical properties of blank tensile test of 316L stainless steel.

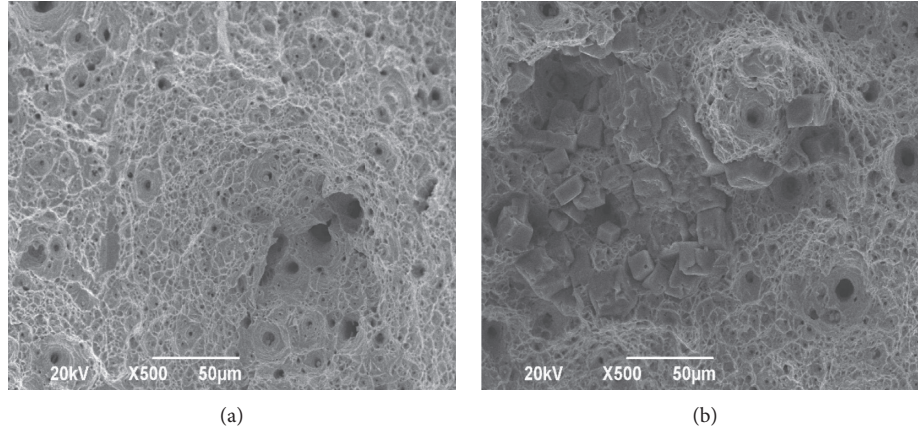| Material mechanics property parameter	Experimental results
Tensile strength (MPa)	607.0
Yield strength ($R_{t0.5}$) (MPa)	339.0
Elongation (A) (%)	62.4
Reduction of area (Z) (%)	86.1
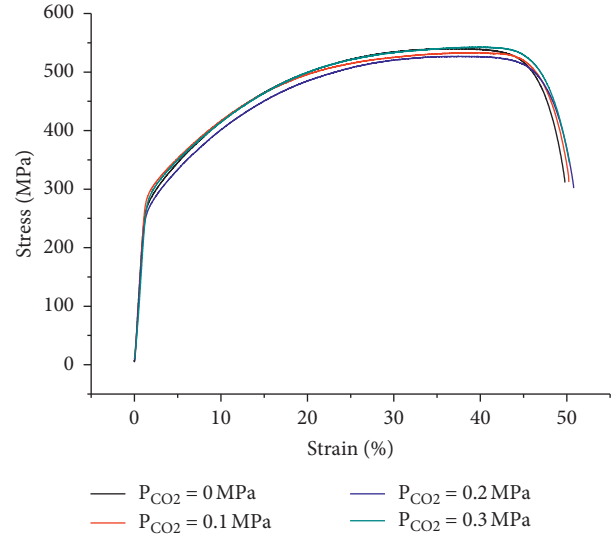
FIGURE 3: Stress-strain curve of 316L stainless steel at different CO_2 partial pressure.

(a)   (b)

FIGURE 4: SEM images of fracture surface morphology of tensile test coupons ($T = 65°C$, $Cl^- = 60,000$ mg/L): (a) $P_{CO_2} = 0.1$ MPa; (b) $P_{CO_2} = 0.3$ MPa.

TABLE 5: Main mechanical properties of 316L stainless steel under different CO_2 partial pressure.

P_{CO_2} (MPa)	Tensile strength σ_b (MPa)	Reduction of area ψ (%)	Elongation δ_5 (%)	Fracture time (h)
0.0	539.0	84.8	48.9	35.0
0.1	532.0	83.7	49.5	35.1
0.2	526.0	85.0	49.8	34.6
0.3	542.0	83.4	49.5	36.2

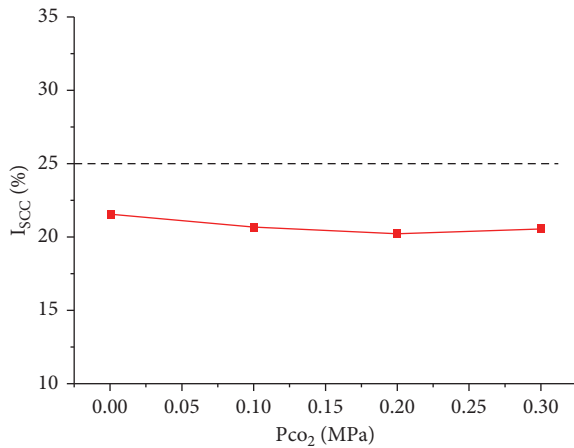FIGURE 5: Stress corrosion cracking sensitivity index of 316L stainless steel in different CO_2 partial pressure.

FIGURE 6: The stress-strain curve of 316L stainless steel at different temperatures.

partial pressure was 0.1 MPa, and the Cl^- concentration was 60,000 mg/L. The stress-strain curve for the sample in the simulated solution at different temperatures is shown in Figure 6. Figure 7 displays the SEM images of fracture surface morphology of tensile test coupons under different temperatures. The main mechanical properties of 316L stainless steel at different temperatures are listed in Table 6.

As shown in Table 6 and Figure 6, the 316L tensile sample's experimental results vary significantly with temperature. When the temperature is 25°C, the material's mechanical properties have a maximum value, which indicates that the sensitivity of SCC is minimal. When the temperature rises to 45°C, none of the mechanical propertie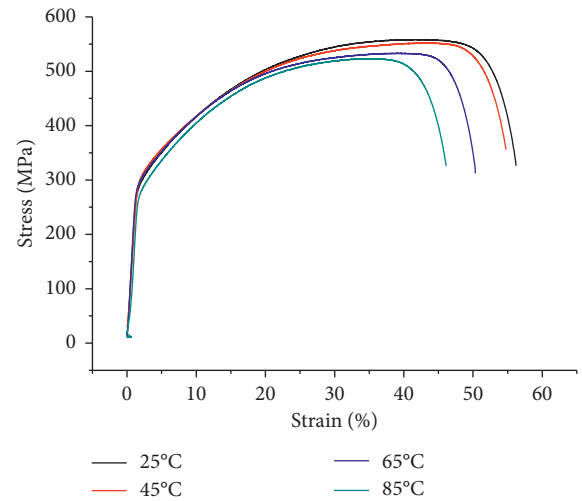s have changed much, indicating that the sample's mechanical properties and plastic deformation capability have not deteriorated significantly with the increase of temperature in this interval. However, when the temperature reaches 65°C, many material properties are reduced considerably. The plastic deformation area in the stress-strain curve narrows significantly, elongation drops by nearly 5%, and tensile strength decreases by 19 MPa. These changes demonstrate that the tensile samples' mechanical properties and their plastic deformation ability have deteriorated. When the temperature rises to 85°C, the elongation further decreases, the fracture strain value is less than 0.5, the plastic deformation of the material dramatically decreases, tensile strength falls from 532 MPa at 65°C to 517 MPa, and section
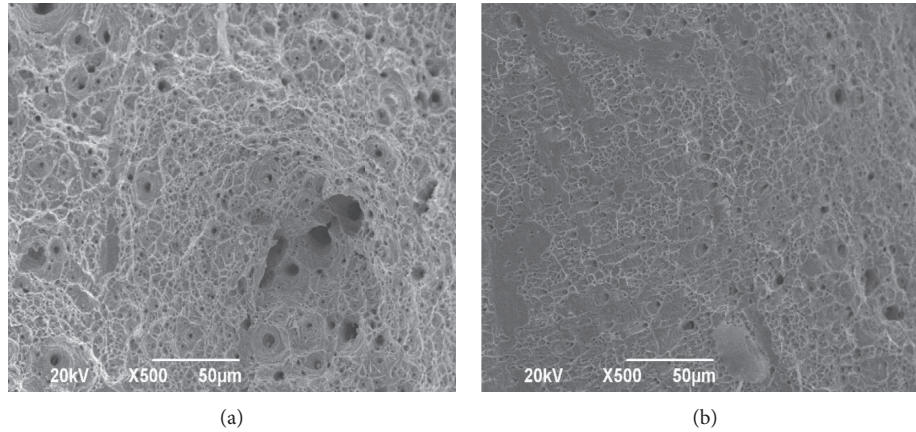
(a)   (b)

FIGURE 7: SEM images of fracture surface morphology of tensile test coupons ($Pco_2 = 0.1$ MPa, $Cl^- = 60,000$ mg/L): (a) $T = 65°C$; (b) $T = 85°C$.

TABLE 6: Main mechanical properties of 316L stainless steel at different temperatures.

T (°C)	Tensile strength σ_b (MPa)	Reduction of area ψ (%)	Elongation δ_5 (%)	Fracture time (h)
25	558.0	84.8	56.2	36.3
45	551.0	82.7	54.1	35.4
65	532.0	83.7	49.5	35.1
85	517.0	77.6	45.9	32.9

shrinkage falls to 77.6% (from 83.7% at 65°C). The onset time of breakage is significantly reduced to 32.9 h.

3.3.2. Analysis. Based on the analysis of the experimental results, the influence of temperature on chloride SCC of 316L stainless steel is as follows:

(1) As we can see from Figures 6 and 8, when the experimental temperature is low (<45°C), 316L tensile samples have relatively low SCC sensitivity. This is because the passive film's properties on the material surface are stable, and its self-repair ability is stronger. With the temperature increase, the dynamic equilibrium of the dissolution and formation of the passive film is damaged, leading to a rise in the possibility of local corrosion. Under the action of continuous stress, high temperature speeds up the dynamic cycle of slippage–film rupture–dissolution–repassivation–slippage, and it shortens the time required for stress corrosion nucleation and expansion.

(2) Increasing the temperature will reduce the pitting breakdown potential Eb100 of 316L stainless steel, which will increase the possibility of pitting corrosion. Besides, the influence of temperature on the corrosion potential is more prominent under no oxygen or low oxygen content.

(3) Increasing the temperature will also increase the metal's surface-active point and accelerate the chemical reaction rate. It is known that there is a natural exponential relationship between crack growth rate and the complex reciprocal temperature; that is, an increase in temperature will accelerate the
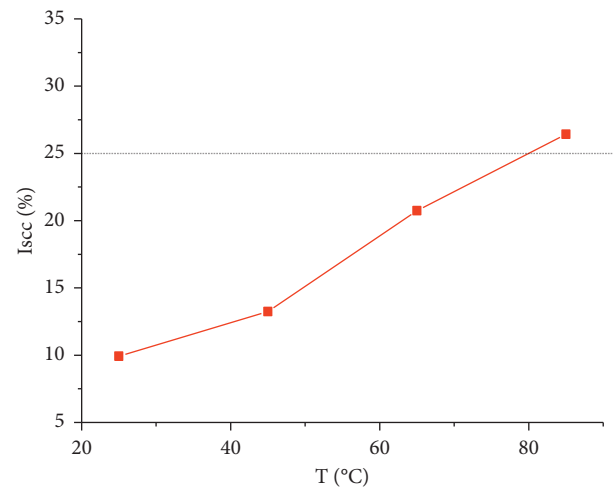

FIGURE 8: Stress corrosion cracking sensitivity index of 316L stainless steel at different temperatures.

crack growth rate. Besides, the chemical adsorption capacity of Cl^- at high temperatures is significantly enhanced, resulting in increased pitting sensitivity of 316L stainless steel and decreased pitting resistance. Under the action of continuous tensile stress, pitting extends to become microcracks and further evolves into SCC.

(4) The figures show the SCC sensitivity index (I_{scc}) of 316L stainless steel at different temperatures calculated according to equation (2). From Figure 6 to Figure 8, we can conclude the following:

(i) When the temperature increases from 25°C to 85°C, the stress corrosion sensitivity of 316L

stainless steel tensile samples significantly increases, demonstrating that, under the environment of a CO_2 gas field in the Tarim Basin, the temperature is an essential factor influencing the occurrence of chloride SCC of 316L. The higher the temperature is, the more likely the SCC will occur.

(ii) The event of SCC of 316L stainless steel requires that a critical temperature value be reached. When the temperature is higher than this critical value, the material will exhibit a more obvious tendency for SCC. Under these experimental conditions, only when the temperature reaches 80°C 316L, stainless steel exhibits SCC tendency.

3.4. Effect of Cl⁻ Concentration on Stress Corrosion Sensitivity

3.4.1. Experimental Results. The stress-strain curves of the 316L stainless steel tensile specimen in a simulated solution at a CO_2 partial pressure of 0.1 MPa and a temperature of 65°C at different Cl⁻ concentrations are shown in Figure 9. The SEM images of fracture surface morphology of tensile test coupons under different Cl⁻ concentrations are displayed in Figure 10. The main mechanical properties of the 316L stainless steel weld sample at different Cl⁻ concentrations are listed in Table 7.

As shown in Table 7 and Figure 9, the tensile strength of the 316L tensile sample decreases slightly with the increase of Cl⁻ concentration, but the change is not significant. The minimum value is 517 MPa when Cl⁻ concentration is 1,00,000 mg/L. The Cl⁻ concentration in the solution has little effect on the 316 L stainless steel's ability to withstand maximum homogeneous plastic deformation between 60,000 and 1,20,000 mg/L. The tensile samples' fracture strain decreases by a certain extent with the Cl⁻ concentration increase, but the variation of the strain within the different Cl⁻ concentration ranges is very different: the strain decreases significantly within the concentration range of 60,000 to 1,00,000 mg/L. The increased Cl⁻ concentration significantly influences the SCC sensitivity of 316L stainless steel in this concentration range. However, when the Cl⁻ concentration increases to 1,20,000 mg/L, the strain change is not apparent. It can be seen from Table 7 that, with the increase of Cl⁻ concentration, the section shrinkage rate of the sample will significantly deteriorate, which indicates that the local plastic deformation ability of the tensile sample is reduced dramatically. Besides, the tensile sample's fracture time tends to shorten overall, indicating that the material's SCC sensitivity increased .

3.4.2. Analysis

(1) The influence of Cl⁻ concentration on the chloride SCC sensitivity of 316L stainless steel is mainly manifested by the promotion of pitting corrosion and microcracks. Under the liquid environment of Cl⁻ enrichment, Cl⁻ selectively adsorbs on the weld and heat-affected zone with a rough surface and
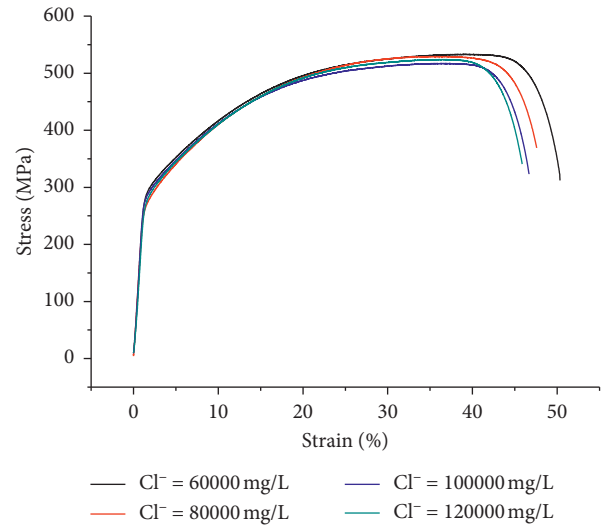

FIGURE 9: Stress-strain curves of 316L stainless steel at different Cl⁻ concentrations.

stress concentration. The Cl⁻ can damage the local passive film of the stainless steel because of its small radius and strong penetration ability, so it is easier to enter the protective film formed by corrosion products. As the concentration of Cl⁻ increases, its tendency to penetrate the metal surface film increases. At this point, the metal surface becomes the battery's anode, while the part that remains passive becomes the cathode area. Because of the continuous tensile stress on the sample, the tensile stress is likely to be locally concentrated at the point with pitting corrosion. The accelerated erosion pit will expand into a crack. Cl⁻ will accumulate at the crack tip, promote the further expansion of the crack tip, reduce the strength and plastic deformation capacity of the material, and eventually cause SCC.

(2) The fracture analysis of numerous slow tensile specimens demonstrates that the metal has corroded before fracture. The corrosion products are mainly composed of Fe and Cr oxides, and the corrosion products do not contain or only contain a small amount of chloride. Cl⁻ does not participate in the corrosion reaction but only plays the role of accelerating the corrosion reaction. However, when Cl⁻ concentration reaches a certain level, the acceleration effect is no longer noticeable.

(3) Figure 11 shows the SCC sensitivity index (I_{scc}) of 316L stainless steel at different temperatures calculated according to equation (2). The following conclusions can be obtained from Figure 11: when Cl⁻ concentration increases from 60,000 to 1,20,000 mg/L, the stress corrosion sensitivity of 316L stainless steel tensile specimens increases by a certain extent, and when Cl⁻ concentration is low, the I_{scc} growth trend is more pronounced. When Cl⁻ concentration is greater than 80,000 mg/L, 316L stainless steel exhibits a certain degree of chloride SCC under the experimental conditions.
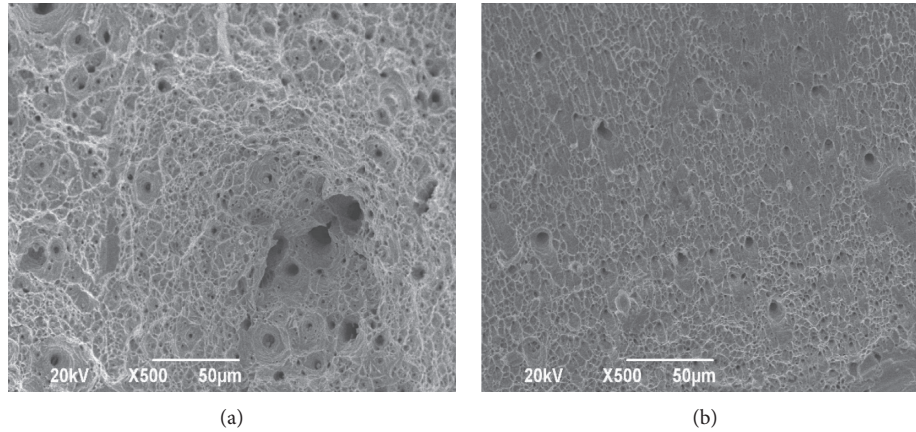
(a)   (b)

FIGURE 10: SEM images of fracture surface morphology of tensile test coupons ($P_{CO_2} = 0.1$ Mpa, $T = 65°C$): (a) $Cl^- = 60,000$ mg/L; (b) $Cl^- = 80,000$ mg/L.

TABLE 7: Main mechanical properties of 316L stainless steel at different Cl^- concentrations.

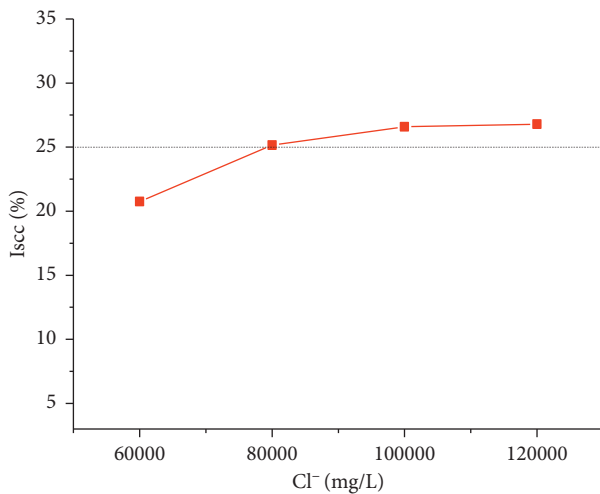| Cl^- (mg/L)	Tensile strength σ_b (MPa)	Reduction of area ψ (%)	Elongation δ_5 (%)	Fracture time (h)
60,000	532.0	83.7	49.5	35.1
80,000	529.0	81.8	46.7	33.6
1,00,000	517.0	79.6	45.8	31.7
1,20,000	524.0	76.2	45.7	32.6

FIGURE 11: Sensitivity index of stress corrosion cracking of 316L stainless steel at different Cl^- concentrations.

4. Conclusions

(1) The slow-strain-rate tensile test results demonstrate that when the partial pressure of CO_2 is between 0 and 0.3 MPa; the effect of increasing CO_2 content on the chloride SCC sensitivity of 316L stainless steel is not apparent.

(2) In the range of 25–85°C, as the temperature rises, the chloride SCC sensitivity of 316L stainless steel increases and I_{scc} changes obviously.

(3) When Cl^- concentration increases from 60,000 to 1,20,000 mg/L, the chloride SCC sensitivity of 316L stainless steel tensile specimens also increases to some extent, but when Cl^- concentration is greater than 1,00,000 mg/L, the I_{scc} value no longer changes significantly.

(4) The degree of influence of the three factors on the experimental results is as follows: temperature>Cl^- > CO_2. In this experiment, when the CO_2 partial pressure is 0.1 MPa, the temperature is 65°C, and the Cl^- concentration is 1,20,000 mg/L, I_{scc} of 316L tensile samples has a maximum of 26.8%.

Data Availability

The data used to support the article are included in the manuscript.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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