Effect of Acidizing Process on the Stress Corrosion Cracking of HP-13Cr Stainless Steel in the Ultra-depth Well Environment

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The effect of acidizing process on the stress corrosion cracking of HP-13Cr stainless steel in the ultra-depth well environment was studied by the slow strain rate test, the electrochemical measurement, the microstructure observation, and the finite element modeling. The results indicated that the acidizing process significantly increased the stress corrosion cracking susceptibility of HP-13Cr stainless steel and induced the fracture mode to the brittle characteristic in the high temperature and CO₂ pressure environment. The stress corrosion cracking susceptibility also increased with the increase of temperature and CO₂ pressure. There were dense defects including pits and cracks in the fracture section from the transverse view. After the acidizing process, under tensile stress condition, the increasing roughness will cause the stress concentration and promote the local anodic dissolution, which induces the initiation of stress corrosion cracking.

Keywords: key word: acidification, HP-13Cr stainless steel, stress corrosion cracking, roughness, anodic dissolution

INTRODUCTION

For the abundant oil and gas reservoir, ultra-depth well exploitation was widely carried out in northwestern China. The well depth was more than 8,000 m with carbonatite rocks and sandstone structures, which brings challenges to effective exploitation (Cui et al., 2004; Lan et al., 2015; Singh et al., 2017; Zhao et al., 2019a). On account of this, many methods such as hydraulic fracturing, steam-assisted gravity drainage, and acidizing are employed to enhance the property value by fast delivery of oil and gas fluid in a cost-effective manner. Among the above effective methods, acidizing has been recognized as the most accessible means to exploit the carbonate and sandstone reservoirs around the northwest of China (Mu and Zhao, 2010; Zhao et al., 2019a). However, the injection of lived acid (LA) during the acidizing process will cause severe corrosion of tubing and further affect the corrosion behavior of tubing in the formation water (FW), which is ejected from the geothermal environment during the exploitation process. The corrosion protection techniques, such as coating, inhibitors, and developing novel materials, are not effective in such an aggressive environment (Cui et al., 2021; Liu et al., 2021; Song et al., 2021). HP-13Cr stainless steel (SS), containing lower C and higher Ni and Mo than traditional 13Cr SS, was widely used as tubing material in the oil and gas industry because of its appropriate mechanical properties, excellent corrosion resistance, and cost-effectiveness. However, severe corrosion failures occurred after LA immersion.

The corrosion behavior of HP-13Cr SS during the acidizing process and in the FW has been investigated in recent years (Moreira et al., 2004; Mu and Zhao, 2010; Marcus, 2011; Qi et al., 2021; Cui et al., 2021; Liu et al., 2021; Song et al., 2021). (Zhang et al., 2020) built the Pourbaix diagram for...
HP-13Cr SS in the aggressive FW and researched the general corrosion rate and pitting (Moreira et al., 2004; Marcus, 2011; Zhao et al., 2018; Li et al., 2019a; Li et al., 2019b; Zhao et al., 2019b; Qi et al., 2021; Cui et al., 2021; Liu et al., 2021). The effect of LA composition and the surface roughness on the corrosion behavior of HP-13Cr SS was also studied (Qi et al., 2021; Zhao et al., 2020). While many ultra-depth well tubes fractured due to stress corrosion cracking (SCC), further research interests should be shifted to SCC in the HTHP environment (Zhu et al., 2011; Lei et al., 2015; Zhang et al., 2019). The SCC susceptibility of HP-13Cr SS was up to 42% exposure at 150°C and 1 MPa CO₂ environment (Yue et al., 2018).

SCC of tubing materials in the high temperature, high CO₂ pressure, and high salinity environments evolved from localized corrosion such as pitting, which may induce the stress concentration (Isaacs, 1988; Cui et al., 2011; Zhao et al., 2012; Ramamurthy and Atrens, 2013; Wang and Han, 2013; Calabrese et al., 2015; Mai and Soghrati, 2017; Wang et al., 2019). Considering the effect of stress on the electrochemical reaction, Gutman built a mechano-electrochemical model to describe the effect of elastic and plastic deformation on the anodic dissolution of metals (Gutman, 1998). The applied stress can shift the pitting potential of 304 SS to a more negative value in 1 M HCl at room temperature (Suter et al., 2001). Meanwhile, the stress and strain concentration at the pitting area changed with the pitting morphology, determined by corrosive environments (Horner et al., 2011; Spencer et al., 2014; Parkins, 1996; Persaud et al., 2019; Lu et al., 2020). The LA
immersion will result in many defects and larger roughness on the metal surface. It is not clear about the LA immersion on the SCC of HP-13Cr SS.

The aim of this work is to reveal the effect of the acidizing process, especially the LA process, on the SCC susceptibility of HP-13Cr SS in FW and clarify the interaction of surface roughness, water chemistry, and stress concentration on SCC by slow strain rate test (SSRT) (Henthorne, 2016), electrochemical measurements, micromorphology observation, and numerical simulation.

**EXPERIMENTAL**

**Material and Solution**

The material applied in this work is a commercial HP-13Cr SS, with a chemical composition (wt%) of Si 0.15, Mn 0.51, Cr 12.77, Mo 2.19, S 0.002, P 0.02, Cu 0.047, Ni 5.36, V 0.014, Al 0.037, and Fe balance. The yield strength is 700 MPa and the elongation is 21.6%. A typical tempered martensitic structure was observed in Figure 1. The dimension of the SSRT specimen was given in Figure 2. Prior to the experiment, the gauge section of the specimens was ground to 2000 grit abrasive paper along the tensile direction, then degreased with ethanol, washed with distilled water, and dried by the flow of air. The chemical composition of LA and FW have been listed in our previous work (Cui et al., 2021). Before the SSRT in FW, the specimen was immersed in LA for 6 h.

**Slow Stain Rate Test**

The slow stain rate test (SSRT) was carried out in the HTHP-SCC system at 95°C/2.8 MPa CO₂ and 120°C/3.2 MPa CO₂ FW conditions, with a strain rate of 10⁻⁶ s⁻¹. Before SSRT, the specimen was immersed in LA for 6 h at 95°C/2.8 MPa CO₂ and 120°C/3.2 MPa CO₂. There was also a SSRT as a normal group conducted at room temperature (25°C) and 0.1 MPa N₂ atmosphere.

The SCC susceptibility of the HP-13Cr SS was evaluated by the elongation loss ratio ($I_δ$) and reduction-in-area loss ($I_ψ$) according to the following Eqs 1, 2 (Tian et al., 2018):

$$I_δ = \left(1 - \frac{δ_s}{δ_0}\right) \times 100\% \quad (1)$$

$$I_ψ = \left(1 - \frac{ψ_s}{ψ_0}\right) \times 100\% \quad (2)$$

where $δ_s$, $ψ_s$ and $δ_0$, $ψ_0$ were the elongation and reduction-in-area of the HP-13Cr SS in the FW and at room temperature (25°C) and N₂ atmosphere, respectively.

**Micromorphology Examination**

The fracture surface of the HP-13Cr SS was cut off after removing the corrosion products based on ASTM standard G1-03 (ASTM, 2003). The fracture morphology viewed from the top and the transverse of the specimens was observed by SEM (Quanta 200 F, U.S.). The cross-section morphology of HP-13Cr SS after LA immersion has also been observed by SEM (Quanta 200 F, U.S.).
Electrochemical Measurement

The potentiodynamic polarization measurements were carried out in FW using the high temperature and high pressure static electrochemical measurement autoclave at 95°C/2.8 MPa CO₂ and 120°C/3.2 MPa CO₂, respectively (Li et al., 2019a). The specimen was also immersed in LA for 6 h before potentiodynamic polarization measurements.

The relationship between the electrode potential vs. standard hydrogen electrode (SHE) and the observed potential vs. the Ag/AgCl reference electrode is shown as Eq. 3,

\[ E_{SHE} = E_{obs} + 0.2866 - 0.001(T - T_0) + 1.754 \times 10^{-7} (T - T_0)^2 - 3.03 \times 10^{-9} (T - T_0)^3 \]

where the \( E_{SHE} \) is the electrode potential vs. SHE, V; the \( E_{obs} \) is the observed potential vs. the Ag/AgCl reference electrode, V; \( T \) is the system temperature, K; and \( T_0 \) is the room temperature (25°C).

The potentiodynamic polarization curves swept from −0.3 V vs. open circuit potential (OCP) to 1.6 V vs. SHE with a scanning rate of 0.167 mV/s. To ensure reproducibility, identical experiments were repeated at least three times.

Numerical Simulation

The profile of HP-13Cr SS (OS) and HP-13Cr SS after immersion tests was observed by Ultra-Depth 3D Microscope (Olympus ILS4100, Japan). The roughness of surface was evaluated as \( S_a \), which is the average absolute deviation of the roughness in a small area. \( S_a \) (μm) was defined as Eq. 4,

\[ S_a = \left( \frac{1}{MN} \right) \sum_{i=1}^{M} \sum_{j=1}^{N} |Z_{ij}| \]

where \( M, N \) is the collected data points from two perpendicular directions in the measurement area; and \( Z_{ij} \) is the distance from the mean area to the number \( i \) \( j \) point.
The tensile stress was set as 350 MPa (half of yield strength) by the finite element method (FEM) in Figure 3. The profile of the surface applied in the FEM was captured from the actual data of the specimen surface after LA immersion at 120°C/3.2 MPa. The mesh near the surface has been refined.

RESULT

SCC Susceptibility of HP-13Cr SS After LA Immersion

The stress-strain curves of HP-13Cr SS during SSRT were shown in Figure 4. Compared with the normal condition (25°C/N₂) and HP-13Cr SS without LA immersion, the curves of HP-13Cr SS drifted down dramatically and yielded at a low stress; meanwhile, the fracture occurred at a quite low strain. With the temperature and CO₂ pressure increasing from 95°C/2.8 MPa to 120°C/3.2 MPa, the stress-strain curve further deteriorated.

The SCC susceptibility of HP-13Cr SS evaluated by \( I_\delta \) and \( I_\psi \) was demonstrated in Figure 5. Compared to the mechanical property at normal conditions, with the temperature and CO₂ pressure increasing from 95°C/2.8 MPa to 120°C/3.2 MPa, the stress-strain curve further deteriorated.

The SCC susceptibility of HP-13Cr SS after LA immersion increased to 55.5 and 60.3% and \( I_\psi \) increased to 62.9 and 70.1% at 95°C/2.8 MPa and 120°C/3.2 MPa, respectively. The dramatic increase of SCC susceptibility indicated that the LA process promoted the SCC of HP-13C SS in the FW environment and with the temperature and CO₂ pressure increasing, the mechanical properties are more severely reduced.

Fracture Morphology

The fracture morphologies of HP-13Cr SS viewed from the top after SSRT at normal conditions were illustrated in Figure 6. The
remarkable necking phenomena and typical cup-and-cone fracture, evidencing a high ductility, were observed (Figure 6A). There was a considerable amount of fine ductile dimples in corresponding high magnification figure (Figure 6B), indicating ductile fracture characteristics.

The fracture morphologies HP-13Cr SS without LA immersion showed a certain brittle fracture feature, including reduced necking at low magnification (Figures 7A,E) and reduced dimples and increased quasi-cleavage plane at high magnification (Figures 7B,F) at 95°C/2.8 MPa and 120°C/3.2 MPa FW. After LA immersion, most of the necking disappeared (Figures 7C,G) and the micromorphology showed a characteristic of brittle fracture with an almost flat plane (Figures 7D,H).

Electrochemical Behavior of HP-13Cr SS After LA Immersion in FW
The potentiodynamic polarization curves of HP-13Cr SS after LA immersion in the FW at 95°C/2.8 MPa CO₂ and 120°C/3.2 MPa CO₂ were shown in Figure 8 and the fitting electrochemical parameters were listed in Table 1 with the temperature and CO₂ pressure increasing from 95°C/2.8 MPa to 120°C/3.2 MPa, the corrosion potential (Ecorr) slightly increased, and corrosion current (icorr) doubled from 2.45 ± 0.25×10⁻⁴ A/cm² to 4.14 ± 0.23×10⁻⁴ A/cm². Since the corrosion production film formed after LA immersion, no significant passivation occurred, especially at 120°C/3.2 MPa FW.

DISCUSSION

The Initiation of SCC
The HP-13Cr SS in LA, as a quite low pH solution, will dissolve and fail to passivate (Zhao et al., 2019a). During LA immersion, the instant of the very thin and efficient corrosion-resistant passivating film as in the normal condition, a loose and thickness up to dozens of microns corrosion production film deposited on the surface, as the cross-sectional morphology after LA immersion shown in Figure 9. The corrosion production film
was comprised of an outer layer rich in copper and comes from the inhibitors in LA solution and an FeCO₃ inner layer (Qi et al., 2021; Zhao et al., 2020). There were plenty of defects including cracking and partial holes. Under a tensile stress condition, the cracks and strain will be concentrated in the defects to deteriorate the corrosion production film and the substrate metal under the film. Then, the pitting occurred as the origin of anodic dissolution cracking (Zhao et al., 2020).

The cracking and pitting can be observed from the fracture morphologies viewed from transverse in Figure 10. At 95°C/2.8 MPa CO₂, the cracking in the surface near the fracture area was dense and almost distributed perpendicular to the tensile direction. The cracking had a spindle shape and revealed a characteristic of anodic dissolution cracking. As the temperature and CO₂ pressure went up to 120°C/3.2 MPa, the cracking had a spindle shape and revealed a characteristic of anodic dissolution cracking. The cracking was longer and had a larger opening, indicating the greater anodic dissolution and more stress and strain concentration occurred after higher temperature and higher CO₂ pressure FA immersion.

The Interaction of Roughness, Water Chemistry, and Stress Concentration on the SCC

As a process of mechanical and electrochemical interaction, SCC was closely related to the water chemical environment. In the formation water, the equilibrium reactions during the CO₂ dissolution process and corresponding reaction equilibrium constants were listed as follows (Reaction Eqs 5-9) (Garsany et al., 2002; Nordsveen et al., 2003; Duan and Li, 2008; Zhang and Cheng, 2009),

\[
\begin{align*}
\text{CO}_2(g) & \rightleftharpoons \text{CO}_2(aq) & K_{f1} = \frac{C_{\text{CO}_2(aq)}}{\phi P_{\text{CO}_2(g)}} \\
\text{CO}_2(aq) + \text{H}_2\text{O}(l) & \rightleftharpoons \text{H}_2\text{CO}_3(aq) & K_{hyd} = \frac{C_{\text{H}_2\text{CO}_3(aq)}}{C_{\text{CO}_2(aq)}} \\
\text{H}_2\text{CO}_3(aq) & \rightleftharpoons \text{H}^+ + \text{HCO}_3^- & K_a = \frac{\gamma_{pH} C_{\text{H}^+} C_{\text{HCO}_3^-}}{C_{\text{H}_2\text{CO}_3(aq)}} \\
\text{HCO}_3^- & \rightleftharpoons \text{H}^+ + \text{CO}_3^{2-} & K_b = \frac{\gamma_{pH} C_{\text{H}^+} C_{\text{CO}_3^{2-}}}{C_{\text{HCO}_3^-}} \\
\text{H}_2\text{O}(l) & \rightleftharpoons \text{H}^+ + \text{OH}^- & K_w = \frac{\gamma_{pH} C_{\text{H}^+} C_{\text{OH}^-}}{C_{\text{H}_2\text{O}(l)}}
\end{align*}
\]

where \(K_{f1}, K_{hyd}, K_a, K_b,\) and \(K_w\) denote the mathematical description of reaction equilibriums, respectively. The fugacity coefficient \((\phi)\) can be determined by the following Eq. 10:

\[
\log \phi = P \left( 0.0031 - \frac{1.4}{T_k} \right)
\]

where \(p\) was the pressure bar and \(T_k\) was the temperature in degrees Kelvin. The temperature-pressure-ionic strength \((I)\) dependence relationship for \(K_{f1}, K_{hyd}, K_a, K_b,\) and \(K_w\) were listed in Table 2, and \(I\) was calculated as follows:

\[
I = \frac{1}{2} \sum m_i z_i^2
\]

where \(m_i\) and \(z_i\) were the molar concentration and charge of species \(i\), respectively. The mean ionic activity coefficients \(\gamma_{pH}, \gamma_{pH^{+9}}, \gamma_{pH^{+10}},\) and \(\gamma_{pH^{+11}}\) in the formation water were 0.662.

In a solution without an externally induced electric field, the concentration of ions must satisfy the electroneutrality constraint. The species Na⁺, Ca⁡²⁺, Mg²⁺, Cl⁻, and SO₄²⁻ dissociated completely in the formation water. Since the addition of NaHCO₃ to the formation water would affect the electroneutrality constraint, the charge relationship between ions can be expressed by Eq. 12:

\[
C_{\text{Na}^+} + C_{\text{H}^+} = C_{\text{OH}^-} + C_{\text{HCO}_3^-} + 2C_{\text{CO}_3^{2-}}
\]

where \(C_{\text{Na}^+}\) was Na⁺ concentration of the artificial addition NaHCO₃ in the simulation formation water, and \(C_{\text{H}^+}, C_{\text{OH}^-}, C_{\text{HCO}_3^-},\) and \(C_{\text{CO}_3^{2-}}\) were the equilibrium concentrations of H⁺, OH⁻, HCO₃⁻, and CO₃²⁻, respectively.

The pH values of FW were calculated as 3.52 and 3.62 at 95°C/2.8 MPa CO₂ and 120°C/3.2 MPa CO₂, respectively.

Surface roughness is of great importance to the chemical and mechanical states at the corrosion interface (Wang et al., 2021). The surface roughness of HP-13Cr SS after FA immersion became larger and increased with the temperature and CO₂ pressure, as is shown in Figure 11. At 95°C/2.8 MPa CO₂, the roughness was 3.5 μm (Figure 11A), which is quite larger than the roughness of the polishing
surface, without LA immersion, less than 0.1 μm. In addition, the roughness increased to 7.8 μm at 120°C/3.2 MPa (Figure 11B). According to the FEM of rough and flat surfaces under 350 MPa tensile stress condition, in Figure 12, the stress (Figure 12A), and the plastic strain (Figure 12B) distribution at 120°C/3.2 MPa were obtained. There was obvious stress and strain concentration in the valley position of the rough surface.
Gutman proposed a kinetic equation for the mecanochemical effect of macroscopic stress on the anodic current \( i_p \) of stressed metal (Gutman, 1998),

\[
i_p = i_e \left( \frac{\Delta \varepsilon}{\varepsilon_0} + 1 \right) \frac{P a}{V_m}
\]

where \( i_e \) was the anodic current of stress-free metal, \( \Delta \varepsilon \) was the plastic strain, \( \varepsilon_0 \) was the strain at onset of strain hardening, \( P \) was the macroscopic stress, \( P a \), \( V_m \) was molar volume, taken as \( 7.1 \times 10^{-6} \text{ m}^3\text{mol}^{-1} \), \( R \) was the gas constant, and \( T \) was the temperature, K.

The anodic current in the area with maximum stress and strain concentration was calculated as 1.7 times than that at a flat surface at 120°C/3.2 MPa.

Therefore, the larger roughness caused by LA immersion promoted the local anodic dissolution and then the greater stress and strain concentration occurred. The further increase of stress and strain concentration will further promote the local anodic dissolution, which forms a mutually reinforcing cycle and results in severe SCC (Cui et al., 2016). Moreover, with the temperature/CO\(_2\) pressure increasing from 95°C/2.8 MPa to 120°C/3.2 MPa, the increase in roughness seemed more crucial to SCC than the pH reduction, and eventually, the more severe SCC occurred after higher temperature and CO\(_2\) pressure LA immersion.

**CONCLUSION**

1. The LA immersion in the acidification process obviously promoted the SCC susceptibility of HP-13Cr SS in FW.
2. With the temperature/CO\(_2\) pressure increasing from 95°C/2.8 MPa to 120°C/3.2 MPa, the surface roughness of HP-13Cr SS after LA immersion increased, resulting in the greater stress concentration and induced the SCC occurring easily.
3. After LA immersion, the interaction of surface roughness, water chemistry, and stress concentration mutually reinforced and deteriorated the SCC of HP-13Cr SS in FW.

**DATA AVAILABILITY STATEMENT**

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

**AUTHOR CONTRIBUTIONS**

WQ: Data curation, Formal analysis, Methodology, Validation, Visualization, Writing—original draft. YZ: Investigation, Validation, Supervision, Writing—reviewing and editing. TZ: Project administration, Conceptualization, Supervision, Funding, acquisition, Writing—reviewing and editing. FW: Conceptualization, Supervision, Funding acquisition.

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