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Effect of CO₂/H₂S and Applied Stress on Corrosion Behavior of 15Cr Tubing in Oil Field Environment

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Received: 10 February 2020; Accepted: 18 March 2020; Published: 23 March 2020

Abstract: The corrosion behavior of a 15Cr-6Ni-2Mo martensitic stainless steel (15Cr stainless steel) in a CO₂/H₂S environment was investigated by conducting high-temperature/high-pressure immersion tests combined with scanning electron microscopy and metallographic microscopy. The presence of H₂S decreased the corrosion resistance of the 15Cr tubing steel. The critical H₂S partial pressure (P_{H_2S}) for stress corrosion cracking in the 15Cr tubing steel in the simulated oil field environment with a CO₂ partial pressure of 4 MPa and an applied stress of 80% σ_s was identified. The 15Cr tubing steel mainly suffered uniform corrosion with no pitting and cracking when the P_{H_2S} was below 0.5 MPa. When the P_{H_2S} increased to 1 MPa and the test temperature was 150 °C, the pitting and cracking sensitivity increased. The stress corrosion cracking at a higher P_{H_2S} is attributed to the sulfide-induced brittle fracture.

Keywords: tubing steel; stress corrosion cracking; high-temperature/high-pressure; pitting corrosion

1. Introduction

Corrosion is a difficult problem and a pervasive issue encountered during the development and exploitation of oil and gas fields. In particular, with the deep exploitation and the aging of oil fields, the environments for oil and gas exploitation are becoming more complex and harsher for the metal pipes, leading to their severe deterioration [1]. Therefore, the corrosion damage of oil casing tubes has increased significantly, causing frequent production halts or oil spill accidents, which leads to substantial economic losses [2–5]. Therefore, effective anti-corrosion technology and measures are urgently needed. To date, various anti-corrosion measures have been developed, which play an important role in the prevention and mitigation of oil field corrosion. At present, the main anti-corrosion measures include the use of corrosion-resistant alloys, coatings and corrosion inhibitors, and the combinations thereof [6–13]. A comparative analysis of the economic situation [14] indicated that, in terms of long-term cost investments, the application of corrosion-resistant materials showed distinct superiority. The use of corrosion-resistant materials during the exploitation of oil and gas fields not only prevents initial investments but also reduces maintenance, repair, and other associated expenses [15–17]. The main objective of developing a new type of oil pipe material is to further reduce the costs. The material 15Cr is a relatively new type of martensitic stainless steel, and compared with 13Cr stainless steel [18–20], it has an increased content of Cr and Ni, which possibly increases the

corrosion resistance of the material. To date, the basic properties of 15Cr stainless steel and its corrosion resistance in the oil field acidification environment have been reported [21–23]. The influences of pH and Cl^- content on the corrosion resistance of 15Cr have also been investigated and the threshold $P_{\text{H}_2\text{S}}$ of 15Cr SS was calculated at 1.5 psi; above this value, 15Cr might suffer sulfide stress cracking [24,25]. Ishiguro et al. [26] studied the stress corrosion cracking of 15Cr-125ksi materials with a 100% specified minimum yield strength (SMYS) applied stress in a H_2S environment, and showed no sulfide stress cracking (SSC) at $P_{\text{H}_2\text{S}} = 0.1$ MPa. However, there are few reports on the corrosion behavior of 15Cr tubing with applied stress under the co-existence of CO_2 and H_2S in simulated oil fields, and the critical value of H_2S affecting 15Cr-110ksi tubing under certain partial pressures of CO_2 still needs to be further determined.

In this study, the corrosion behavior of 15Cr martensitic stainless steel tubing under applied stress was studied under the co-existence of H_2S and CO_2 , and the environmentally assisted cracking susceptibility of the materials under stress and stress-free conditions was compared. Moreover, the threshold values of the H_2S concentration for the 15Cr tubing steel in the H_2S and CO_2 environment were obtained.

2. Experimental

2.1. Materials and Methods

The samples were cut from 15Cr-6Ni-2Mo (UNS S42625) martensitic stainless steel commercial tubing, with the chemical compositions: 0.024 wt.% of C, 0.002 wt.% of S, 0.013 wt.% of P, 0.28 wt.% of Si, 15.22 wt.% of Cr, 0.18 wt.% of Mn, 6.31 wt.% of Ni, 2.11 wt.% of Mo, 0.41 wt.% of Cu, 0.02 wt.% of N, and the remaining (up to 100) wt.% of Fe, and an actual yield strength (σ_s) of 825 MPa was used. The samples were divided into two groups: with and without applied stress. The samples without applied stress were cut to the size of $40 \times 10 \times 3$ mm (length $\times$ width $\times$ thickness), and the size of the samples for the stress corrosion test was $57 \times 10 \times 3$ mm. Prior to each experiment, all the samples were carried out in accordance with the treatment procedures specified in the standard GB/T 15970.2-2000 (ISO 7539-2), so as to avoid and reduce the existence of residual stress. For the samples with applied stress, the longitudinal edge was polished to prevent stress concentration caused by the edges and corners. After polishing, the samples were cleaned with distilled water and ethanol, dried under cool air, and stored in a dry N_2 atmosphere.

The tests were carried out in an autoclave at 90 and 150 °C at the CO_2 partial pressure of 4 MPa. The H_2S partial pressures ($P_{\text{H}_2\text{S}}$) were 0.1, 0.5, and 1 MPa, respectively. The test solution with the Cl^- concentration of $150 \text{ g}\cdot\text{L}^{-1}$, simulating the formation water in oil fields, was prepared from analytical grade sodium chloride and deionized water.

2.2. Immersion Tests

The immersion tests were conducted in an autoclave. Prior to the weight loss tests, the samples were cleaned with distilled water and acetone, dried, and then weighed using a balance with a precision of 0.1 mg. The weight values were recorded as the original weight (W_{0i} , $i = 1, 2 \dots$). In order to ensure the reproducibility of the results, four parallel samples were taken from each group. The solution was deoxygenated by bubbling pure nitrogen to ensure that the low oxygen concentration was below 10 ppb (according to the requirement of oil field water quality). Then, H_2S , CO_2 , and heat were introduced. The total pressure was brought up to 10 MPa by purging nitrogen through the system.

After the completion of the tests, the corroded samples were divided into two groups. One sample was used to observe the corrosion morphology, and the other three were used to calculate the average corrosion rate to ensure the reproducibility of the results. The corrosion products were removed via the chemical method according to GB/T 16545-1996 (idt ISO 8407: 1991), then rinsed and dried, and

then finally reweighed to obtain the final weight (W_{1i}). The corrosion rate (V_i) was calculated in mm/y (average corrosion thinning depth in years) from the weight loss by using Equation (1) [27] as follows

$$V_i = 8.76 \times 10^6 \times (W_{0i} - W_{1i}) / (t \times \rho \times S) \quad (i = 1, 2 \dots) \quad (1)$$

where W_{0i} and W_{1i} are, respectively, the original and final weights of the samples in g, S is the exposed surface area of the samples in mm^2 , t represents the immersion time in h, and ρ is the steel density equaling $7.8 \times 10^{-3} \text{ g} \cdot \text{mm}^{-3}$. An average corrosion rate of the three different samples for each test condition was reported as an overall corrosion rate for each set of conditions.

2.3. SCC Testing

Stress corrosion cracking testing was carried out by the immersion method under high-temperature and high-pressure conditions in an autoclave. To ensure the reproducibility of the results, there were three parallel specimens in each stress test, and the test was only effective when the specimen broke or cracked in the working area. In these tests, all sample surfaces required precision machining and a surface roughness of $R_a \leq 0.2 \mu\text{m}$. These stress corrosion cracking tests were adopted a four-point bending loading method [28]; this method was operated according to the standard GB/T 15970.2-2000 (ISO 7539-2: Corrosion of metals and alloys-Stress corrosion testing—Part 2: Preparation and use of bent-beam specimens). The schematic diagram of the four-point bending loading device is shown in Figure 1.

Figure 1. Schematic presentation of the four-point bending loading method.

In this study, according to the engineering requirements, the actual yield strength (825 MPa) value of the material was used to calculate the stress value loaded on the sample, rather than the specified minimum yield strength (SMYS) (758 MPa), so an appropriate stress ratio coefficient of 80% was used. Stress loading can be achieved by Equation (2) as follows [29]

$$\sigma = 12E \times t \times y / (3H^2 - 4A^2) \quad (2)$$

where H represents the distance between the two outermost fulcrums, E is the elastic modulus, A represents the distance between the inner and outer fulcrums, t is the sample thickness, and σ represents the stress value of the loading. Using these known parameters to calculate the maximum deflection y between the outer fulcrums, the stress is loaded by detecting the deflection change of the specimen.

2.4. Characterization

After the corrosion tests, the samples used for the surface analysis were removed and rinsed with deionized water, followed by an alcohol rinse, and then dried.

The corrosion products and cracks were studied using scanning electron microscopy (SEM) and optical microscopy (OM). Energy-disperse spectroscopy (EDS) was used to analyze the compositions of the corrosion products.

3. Results and Discussion

3.1. Average Corrosion Rate

The average corrosion rates under different H_2S partial pressures are listed in Table 1. When the P_{CO_2} and P_{H_2S} equals 4 and 1 MPa, respectively, the specimen breaks and the fracture surface becomes corroded, and thus the corrosion rate cannot be calculated accurately.

Table 1. The average corrosion rates of samples under high-temperature and high-pressure.

Condition Number	Test Conditions	V_{corr} ($\mu m/y$), w —Range			
		90 °C		150 °C	
		0%	80% σ_s	0%	80% σ_s
1	$P_{H_2S} = 0.1$ MPa	0.73 ($w = 0.20$)	1.30 ($w = 0.35$)	1.24 ($w = 0.36$)	1.91 ($w = 0.06$)
2	$P_{H_2S} = 0.5$ MPa	1.0 ($w = 0.36$)	1.60 ($w = 0.40$)	1.7 ($w = 0.20$)	3.2 ($w = 0.42$)
3	$P_{H_2S} = 1$ MPa	11.03 ($w = 0.20$)	15.20 ($w = 0.43$)	29.03 ($w = 0.4$)	Fracture

The corrosion rates of the samples with 80% σ_s were higher than those of the samples without applied stress. The average corrosion rate of the samples increased gradually with the increase in the H_2S partial pressure and test temperature. Under the high H_2S partial pressure, the sample under the applied stress cracked, indicating that the sulfide-caused stress corrosion cracking gradually became more dominant with the increase in the H_2S concentration [24]. H_2S dissolves in water and reacts chemically to form metal sulfides and hydrogen atoms. The hydrogen atom diffuses into the material at the crack tip with the highest tensile stress. The diffusion and accumulation of hydrogen on the lattice, the surface of the lattice, and the grain boundary reduce the plastic deformation ability of the material and cause hydrogen embrittlement, making it easier for the crack to expand.

On the other hand, H_2S is a hydrogen recombination poison which significantly affects brittle fractures of materials [30]. The hydrogen ions formed on the cathode cannot effectively form hydrogen molecules due to the poisoning effect of the HS^- and S^{2-} ions [31,32], and thus the hydrogen concentration on the surface of the steel increases, resulting in the diffusion of hydrogen into the steel. Steel enrichment by hydrogen eventually makes the steel brittle. Moreover, under the applied stress, the surface activity of the samples gets enhanced, and fractures are more likely to occur [33]. The H_2S partial pressure also directly affects the pH of the solution [34]. When the partial pressure increases, the corrosion products on the metal surface become loose and they gradually increase the corrosion rate [35,36]. At the same time, when the loose product film falls off, there is a potential difference between the exposed substrate and the product film with good adhesion, and thus many tiny corrosion couples are formed on the surface of the sample which promote the accelerated corrosion in the anode region with a low potential.

Under the co-existence of CO_2 and H_2S , the corrosive medium has a competitive and synergistic effect on the corrosion behavior of the materials. With the increase in the test temperature, the solubility of the CO_2/H_2S corrosion gas decreases, yet the reaction rate in the solution system accelerates, promoting corrosion. Therefore, the overall outcome depends on the synergistic effect among the temperature, stress, and corrosive gases.

3.2. Observation of Corrosion Morphology

After the high-temperature and high-pressure tests, the surface corrosion morphology was inspected. Figures 2 and 3 show the microscopic morphologies of the corroded samples without

applied stress. The corrosion scales were even. Polishing marks on the surface indicate that the corrosion film was thin (see Figure 2). Figure 3 shows that with the increase in the test temperature, the surface corrosion morphology was relatively rough, and when at $P_{\text{H}_2\text{S}} = 1$ MPa, similar pitting morphology was found on the surface of the samples at 150 °C (see Figure 3c). The results show that the pitting corrosion sensitivity of 15Cr stainless steel increases due to the synergistic action between a high temperature and high H_2S concentration.

Figure 2. Micro-corrosion morphology of the samples without stress at 90 °C, with different experimental conditions (a) $P_{\text{H}_2\text{S}} = 0.1$ and $P_{\text{CO}_2} = 4$ MPa, (b) $P_{\text{H}_2\text{S}} = 0.5$ and $P_{\text{CO}_2} = 4$ MPa and (c) $P_{\text{H}_2\text{S}} = 1$ and $P_{\text{CO}_2} = 4$ MPa.

Figure 3. Micro-corrosion morphology of the samples without stress at 150 °C, with different experimental conditions (a) $P_{\text{H}_2\text{S}} = 0.1$ and $P_{\text{CO}_2} = 4$ MPa, (b) $P_{\text{H}_2\text{S}} = 0.5$ and $P_{\text{CO}_2} = 4$ MPa and (c) $P_{\text{H}_2\text{S}} = 1$ and $P_{\text{CO}_2} = 4$ MPa.

Figure 4 shows the surface corrosion morphology (as shown in the red box area) of the samples under the condition of applied stress. At $P_{\text{H}_2\text{S}} \leq 0.5$ MPa, and with an increase in the temperature, the corrosion film on the sample's surface is similar to that on the surface of the samples without stress, and no cracking or fractures are observed (see Figure 4a), indicating that the applied stress of 80% σ_s under the experimental conditions has no obvious effect on the surface activity of the samples. However, at $P_{\text{H}_2\text{S}}$ of 1 MPa and a test temperature of 150 °C, the samples exhibit cracks and fractures at their center regions. Nonetheless, there are more micro-cracks around the main crack (see Figure 4b).

Figure 4c exhibits that the pitting corrosion occurred around the crack, indicating that when the $P_{\text{H}_2\text{S}}$ equals 1 MPa, the pitting susceptibility of the material under the stress-state reaches its critical value. Pitting corrosion plays an important role in stress corrosion cracking. Surface cracks were also observed on the fractured samples. Clearly, some micro-cracks originated from the center of the sample along its width direction and extended to both ends of the sample, and the two red rings in Figure 4d indicate the origin of the cracks in the middle of the samples. Figure 4e is a high-magnification of the cracks marked with red rings, and the typical characteristics of sulfide stress corrosion cracking at both

ends of the crack can be observed, which indicates that the stress corrosion cracking sensitivity of 15Cr stainless steel is higher, and that cracking failure occurs under these simulated test conditions.

Figure 4. The surface corrosion morphology of the samples under the applied stress at 150 °C: (a) Macro-corrosion morphology of stress concentration surface, (b) Macro-corrosion morphology of fracture area, (c) Micro-morphology of crack and pitting in the stress concentration region, (d) Micro-cracks morphology originating from the center of the sample in the stress concentration region (20× magnification), and (e) Micro-crack morphology under 50× magnification.

The cross-sections of the fracture side morphologies are shown in Figure 5. The main crack propagated almost through the thickness of the sample. Many smaller sub- and micro-cracks were located around the main crack. Distinct dendritic cracks were found at the crack tips, showing typical

sulfide stress corrosion features (marked by the red circles in Figure 5a,b). Thus, 15Cr stainless steel tubing is not suitable for use in the simulated oil field environment, as H₂S-related corrosion occurs easily.

Figure 5. The side morphology of the fractured sample: (a) Main crack tip propagation morphology and (b) Growth morphology of sub-cracks tip.

The color of the corrosion products near the cracks (marked as the “A” region in Figure 6a) was different from that of the other no-cracking areas (marked as the “B” region): the “A” region appears dark gray and the “B” region has a lighter color.

The EDS analysis (shown in Figure 6b) revealed that the “A” and “B” regions were mainly composed of S, C, O, and Fe elements. However, the sulfur content in the “A” region was higher than in the “B” region. This indicates that the adsorption of H₂S on the active surface, and the anodic dissolution of the matrix, promote the occurrence of stress cracking. However, the higher content of sulfide at the fracture region further demonstrates that the H₂S can elevate the cracking susceptibility of the samples under applied stress [37,38]. Thus, the surface activity of the stress-concentrated region is much higher, and the chemical reaction between the alloy and H₂S is favored over the reaction of CO₂ and the material. Moreover, at a high temperature, H₂S dissolves in the solution and the H atoms get adsorbed and penetrate the sample surface, accelerating the chemical reaction between S^{2−} and the material [39]. Owing to this effect, increased amounts of sulfides were generated in the stress-concentrated regions of the samples. Therefore, the synergistic effect of the applied stress and H₂S corrosion medium contributed to the much more severe localized corrosion in the stress-concentrated region, prompting the formation and extension of the crack [38], and finally inducing the sample fracture. The results are consistent with the stress corrosion cracking characteristics of the preceding broken sample, indicating that the failure of the specimens mainly belongs to the sulfide stress cracking mode. The mechanism for the reaction between H₂S and the materials in the test solution is shown by Equations (3)–(7) [40]:

Figure 6. Surface morphologies (a) corrosion products of two different regions near the crack and (b) Energy-dispersive spectroscopy (EDS) analyses.

The fractured samples are separated along the fracture direction. Subsequently, the corrosion morphologies of the fracture surface are shown in Figure 7. The low-magnification of the fracture surface does not reveal metallic luster (see Figure 7a). The higher magnification microscopy images show a thick corrosion film covering the fracture surface (see Figure 7b), and as a result, the exact fracture morphology of the crack is hard to detect. This corrosion film is mainly composed of S, Fe, C, and O elements (see Figure 7c). This can infer that the sample fractured during the test and the fresh fracture surface were further corroded when exposed to the corrosive medium.

Figure 7. (a) Low and (b) high magnification of the fracture morphology, and its (c) EDS analysis.

Figure 8 shows the micro-morphology of the fracture surface after cleaning the corrosion products, and intergranular corrosion characteristics can be clearly observed (see Figure 8). Secondary cracks along the grain boundaries can also be seen (see Figure 8b). This further illustrates that the samples loaded with 80% σ_s sustained brittle sulfide stress corrosion cracking at a 1 MPa H_2S partial pressure. Thus, this material has a high sensitivity to stress corrosion cracking at the given experimental condition, and therefore is not suitable for oil field environments with similar conditions.

Figure 8. Intergranular corrosion characteristics of fracture surfaces. (a) Micromorphology of intergranular corrosion on the fracture surface and (b) Secondary crack morphology on fracture surface.

4. Conclusions

In this study, the corrosion and stress corrosion cracking behaviors of a 15Cr tubing steel with and without applied stress were studied by the weight loss and four-point bending methods in a high-temperature and high-pressure environment. The main results and conclusions are as follows:

(1) When the P_{CO_2} was 4 MPa and the P_{H_2S} was less than or equal to 0.5 MPa, the average corrosion rate of the 15Cr tubing increased in the simulated oil field environment with the increase of H_2S pressure. The 15Cr tubing steel mainly showed uniform corrosion. No clear signs of pitting were found, and no crack or fracture occurred when the samples were loaded with 80% σ_s ;

(2) At 150 °C, and at $P_{CO_2} = 4$ MPa and $P_{H_2S} = 1$ MPa, the pitting sensitivity of the 15Cr tubing steel was higher, and cracking occurred when the samples were loaded at 80% σ_s .

Author Contributions: X.Z. conducted the research and result analysis under the supervision of Y.F., W.H. and J.Z. contributed in design of the experiments and morphology analysis during the research activities, and G.L. contributed in image processing and data calculation. All authors have read and agreed to the published version of the manuscript.

Funding: This research was founded by the National Key R&D Program of China (2017YFB0304905) and Major projects of China national petroleum corporation (2014E-3603)".

Acknowledgments: The authors greatly acknowledge the financial support from the National Key R&D Program of China and the CNPC Science and Technology Project, and also acknowledge the state key laboratory for its support of high-temperature and high-pressure experiments.

Conflicts of Interest: The authors declare no conflict of interest.

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