Materials Research Express

PAPER

Corrosion behavior of Cr-bearing steels in CO$_2$-O$_2$-H$_2$O multi-thermal-fluid environment

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Keywords: stainless steel, Cr content, corrosion, exfoliation, multi-thermal fluid, corrosion scale

Abstract

Multi-thermal fluid is used to enhance heavy oil recovery, but it also causes severe corrosion failure of metallic tubing and its mechanism is still open to debate. In the present work, the corrosion behavior of commercial steels with different Cr content (3 wt%, 9 wt%, and 25 wt%) in simulated CO$_2$-O$_2$-H$_2$O multi-thermal fluid environments were studied by immersion corrosion test and microstructural characterization. The results exhibit that the Cr content of steel determines the type of corrosion products. The less protective corrosion scale is mainly composed of FeCO$_3$, (Fe, Cr)$_2$O$_4$, and Fe$_2$O$_3$, which is formed on the surface of 3Cr and 9Cr steels. The protective corrosion scale is mainly Cr$_2$O$_3$, which is formed on the surface of 25Cr steel. Only when the Cr content in steel is sufficient (i.e. 25 wt%) to repair the serious depletion of Cr in the metal near the scale/metal interface in time, can protection of the corrosion scale be sustained, to improve its ability to resist the corrosion of multi-thermal fluids.

1. Introduction

Direct injection of thermal fluids into the oil reservoir has been used as an effective way to extract heavy oil worldwide [1, 2]. Usually, thermal fluids are generated by the burning of fuel and oxidizer. For such wells, metallic tubing would be corroded by corrosive species such as CO$_2$, O$_2$, and H$_2$O. In China, several corrosion failure accidents (as shown in figure 1) have been taken place, which caused serious consequences. In these cases, the corrosion mechanism is much more complex owing to the competition and/or synergy between CO$_2$ and oxidizing gas like O$_2$ and water vapor.

In the oil and gas industries, CO$_2$ corrosion has been widely studied [3–7]. The corrosion mechanism has been clear, and some prediction models have been established. Due to the complex composition of the multi-thermal fluid, the corrosion mechanism of metallic materials in such an environment is still unclear. In addition to the complex multi-component characteristics, the special temperature range is beyond the scope of published literature. For example, the research temperature of CO$_2$ corrosion in the oil and gas industry is mostly below 150 °C, while that in high-temperature combustion environment is more than 500 °C [8, 9]. There is a rare study in the intermediate temperature region (150 ~ 500 °C). Electrochemical corrosion occurs on metallic materials in the CO$_2$-containing oil and gas production environment (usually lower than 150 °C) and forms ferrous carbonate. Once oxygen is introduced into the CO$_2$-containing environment, the corrosion will be accelerated and oxides will be formed. In the high-temperature combustion environment (usually beyond 500 °C), oxidation takes place on the metallic materials, where CO$_2$ reacts directly with metals to form oxides. In
contrast, corrosion caused by the multi thermal fluid at a temperature range of 150 °C ~ 500 °C is still open to debate.

Based on the above considerations, the present work was performed to investigate the corrosion behavior of Cr-bearing steels which is potentially applied as tubing in multi-thermal fluid injected well. On the basis of the characterization of microstructure, chemical compositions, and phases, the effect of Cr content on the corrosion were discussed.

### 2. Materials and methods

Commercial steels with different Cr content (3 wt%, 9 wt%, and 25 wt%) were used in the present work, and their chemical compositions are shown in table 1. The metallographic analysis showed that the microstructures of the three steels are pearlite + ferrite, martensite and austenite, as shown in figure 2.

Rectangular specimens with dimensions of 50 × 10 × 3 mm were cut from above Cr-bearing steel tubes, and a suspension hole with a diameter of 5 mm drilled near an edge. All specimens were ground to a 1200 grit finish, and then cleaned ultrasonically in deionized water and anhydrous ethanol. After that, they were dried out with could air, and weighed with an electronic balance with an accuracy of 0.1 mg.

An autoclave with a capacity of 3L were used to carry out corrosion tests. Prior to testing, 1 L simulated formation water was disposed according to table 2, and then high purity nitrogen was introduced to remove oxygen from the solution. After that, the autoclave was heated up to 240 °C, and then CO₂-O₂-N₂ mixture gas was introduced until the total pressure reached 10 MPa. In this case, the $p$CO₂ (partial pressure of carbon

### Table 1. Chemical compositions of Cr-bearing steels in the present work (wt%).

| Steel | C   | Si  | Mn  | P     | S     | Cr  | Mo  | Ni  | Nb | Fe  |
|-------|-----|-----|-----|-------|-------|-----|-----|-----|----|-----|
| 3Cr   | 0.056 | 0.18 | 0.38 | 0.0089< | 0.002 | 2.96 | <0.0009 | 0.0030 | 0.026 | Bal. |
| 9Cr   | 0.11 | 0.35 | 0.38 | 0.014< | 0.0051 | 8.82 | 0.96  | 0.061 | /   | Bal. |
| 25Cr  | 0.06 | 0.40 | 1.20 | /     | /     | 25.0 | /    | 20.0 | 0.45 | Bal. |

### Figure 1. Corrosion failure of metallic tubing used in multi-thermal fluid injected well.

### Figure 2. Metallographic microstructures: (a) 3Cr steel, (b) 9Cr steel, and (c) 25Cr steel.
dioxide) was 2 MPa, the $p_{H_2O}$ (partial pressure of water vapor) was 3.3 MPa, and the $p_{O_2}$ (partial pressure of oxygen) was 0.5 MPa. During the whole test, the temperature was kept at 240 °C, and the total pressure was kept at 10 MPa.

In order to calculate the corrosion rate, three identically tested specimens for each steel were rinsed successively with deionized water, acid solution, sodium hydroxide solution, deionized water and anhydrous ethanol, as described in ASTM G1 standard. Average corrosion rate (CR) was obtained by equation (1),

$$CR(\text{mm/y}) = \frac{8.76 \times 10^4 \times W}{A \times T \times D}$$

Where, $W$ is the mass loss (g), $T$ is the exposure time (h), $A$ is the surface area ($\text{cm}^2$), and $D$ is the density of the metal ($\text{g cm}^{-3}$).

Scanning Electron Microscope (SEM) was applied to examine surface morphology and cross-section morphology of tested specimens, where Secondary Electron (SE) mode was used for the surface morphology investigation while Backscattered Electron (BSE) mode was used to observe the cross-section morphology. The typical acceleration voltage of SEM observation was 20 kV. Energy Dispersive Spectrometer (EDS) coupled with SEM was applied to analyze the chemical composition of corrosion scales. In addition, the phases of corrosion products were identified by X-ray Diffraction (XRD) operating with Cu-Kα radiation with a wavelength of 1.5405 Å at 40 kV and 40 mA in the 2θ range of 10°–90° and a scan rate of 0.1 degree per second.

### 3. Results

#### 3.1. Corrosion of 3Cr steel

The uniform corrosion rate of 3Cr steel is 2.5365 mm y$^{-1}$, which is very large compared with most cases. Figure 3 shows the surface morphology of 3Cr steel after 240 h corrosion. There are several distinguishing features. First, exfoliation of surface corrosion products happened in the local region, as shown in figure 3(a). Second, at high magnification, cracks can be seen near the edge of the exfoliation region, as shown in figure 3(b). Third, coarse granular corrosion products with sharp edges and corners can be seen in the exfoliation region, as shown in figure 3(c). In this region, the EDS analysis result shows that the corrosion scales are composed of oxygen (32.23 wt%), chromium (20.46 wt%), and iron (the balance). Fourth, except for the local cracks, the outermost corrosion products show a more compact appearance, as shown in figure 3(d). The EDS analysis result reveals that this layer consists of oxygen (33.75 wt%), chromium (12.10 wt%), and iron (the balance). The chromium content is significantly lower than that in the exfoliation region.

Figure 4 shows the cross-section morphology (as shown in figure 4(a)) and the EDS element profile (as shown in figure 4(b)) of 3Cr steel after 240 h corrosion. There are several distinguishing features for the corrosion scale. First, the outer corrosion products appear dark gray, the medium layer appears bright gray, and the inner layer appears dark brown. Generally, under the BSE mode, the contrast is related to the average atomic mass of corrosion products, and the brighter contrast indicates the corrosion products with a relatively larger average atomic mass. Second, beneath the outermost layer with dark gray contrast, the corrosion products show coarse grains similar to that in figure 3(c). In some regions, the coarse grains show bright gray contrast while some of them near to the outermost layer show dark gray contrast which is similar to the contrast of the outermost layer. Third, close to the interface of the corrosion scale and substrate, a very thin corrosion layer with dark brown contrast can be seen, as shown in figure 4(c) at higher magnification. And the EDS point analysis as shown in figure 4(d) indicates that the innermost corrosion products would be Cr-rich hydroxide/oxide such as Cr(OH)$_3$.

From the XRD pattern of corroded 3Cr steel, as shown in figure 5, it can be seen that the corrosion scales mainly consist of magnetite and iron carbonate. Combined with the above analysis, the outermost layer with dark gray contrast would be iron carbonate, while the layer with bright grey contrast would be magnetite.

It can be speculated that 3Cr steel suffered from serious corrosion in the multi-thermal fluid at 240 °C, and formed thick multi-layer corrosion products containing magnetite, iron carbonate, and Cr-rich hydroxide/oxides. Clear boundaries (or cracks) can be seen through the whole scale, indicating that the corrosion scale is not a good barrier to the corrosive medium. It is too low for the Cr content in 3Cr steel to form a dense and protective Cr-
rich layer in the studied multi-thermal fluid environment, thereby leading to poor corrosion resistance to multi-thermal fluid.

3.2. Corrosion of 9Cr steel

The uniform corrosion rate of 9Cr steel is 11.0140 mm y\(^{-1}\), which is almost 4.5 times that of 3Cr steel. Figure 6(a) shows the blister-like corrosion products appeared on the surface of 9Cr steel after 240 h corrosion, and they are prone to exfoliate. Employing the SE mode to investigate the surface morphology, the image at low magnification.
Figure 5. XRD patterns of 3Cr steel specimens after 240 h exposure in multi-thermal fluid.

Figure 6. Surface morphology of 9Cr steel after 240 h exposure in multi-thermal fluid: (a) Blister-like corrosion products investigated by optical microscope, (b) image at low magnification, (c) image in the exfoliation region, (d) interface of flat and nodule, (e) flat region with cracks, and (f) nodule at high magnification.
magnification is shown as figure 6(b). It can be seen that 9Cr suffered extremely serious corrosion. Extremely significant exfoliation and long enough cracks can be observed on the surface. Besides, nodule-like corrosion products covered the larger part of the surface. Figure 6(c) indicates the morphology of exfoliation region at high magnification. EDS analysis reveals that the corrosion scales contain oxygen (26.71 wt%), chromium (19.93 wt%) and iron (the balance). Figure 6(d) shows the interface of flat and nodule. Figure 6(e) presents the flat region at high magnification, EDS analysis indicates that the corrosion products are composed of oxygen (32.12 wt%), chromium (26.73 wt%), and iron (the balance). Figure 6(f) shows the nodule-like region where coarse grains with sharp edges and corners can be seen. EDS analysis indicates that this kind of corrosion products are composed of oxygen and iron.

Figure 7 shows the full view of the cross-section of 9Cr steel after 240 h corrosion. There are several features. First, the thickness of the corrosion scale is nonuniform, and the largest thickness is about 600 μm. Second, there are two different contrasts in the corrosion scale, and the outer layer is much thicker than the inner layer. Third, a large number of vertical and penetrating cracks (along the thickness direction) can be observed. Fourth, the corrosion scale separates from the substrate (a clear ‘gap’). As the corrosion scale is very thick, three regions (as denoted as b, c, and d in figure 7(a)) were selected for investigation at higher magnification. The morphology is shown in figures 7(b)–(d). Figure 7(e) shows the EDS element profile across the corrosion scale, where the scale is divided into three regions (R-I, R-II, and R-III) according to the relative content of Fe and Cr. Combined with the surface morphology discussed above, it is not difficult to conclude that the outmost corrosion products rich in Fe are not completely displayed on the cross-section morphology due to falling off. Therefore, the corrosion scale observed from the cross-section is mainly the Fe-Cr oxides.

Figure 8 shows the XRD pattern for the corrosion scale formed on 9Cr steel. The main phases are (Fe, Cr)_{2}O_{3}, FeCO_{3}, and Fe_{2}O_{3}.

It needs to point out that the corrosion scale formed on 9Cr steel as shown in figure 7 is somewhat different from the observation in figure 6. The main difference is the absence of the outermost iron oxides in figure 7. This may be ascribed to the exfoliation of the outermost nodule-like iron oxides. As indicated in figure 6(b), the exfoliation region is composed of oxygen, chromium, and iron. From this perspective, the outer layer as shown in figure 7 is the corrosion scale in the exfoliation region as shown in figure 6(b).

For 9Cr steel, it suffered from much more serious corrosion in the multi-thermal fluid than 3Cr steel, although the chromium content in 9Cr steel is greater than that in 3Cr steel. It is generally considered that the increase of chromium content would improve the corrosion resistance of steels. In the present work, when the chromium content increased from 3 wt% to 9 wt%, corrosion became much more severe. There may be two possible reasons. First, 9 wt% chromium in 9Cr steel cannot form a protective Cr-rich scale. Second, vertical cracks through the corrosion scale would provide effective ways for the inward penetration of the corrosive medium to contact with the substrate, so that the corrosion continues at a fast rate.
3.3. Corrosion of 25Cr steel

The uniform corrosion rate is 0.0953 mm yr⁻¹, which indicates that the corrosion is very slight. Figure 9(a) shows the surface morphology of 25Cr steel after 240 h corrosion. The surface is uniform, where no evident cracks, exfoliation, and nodules can be seen. Besides, the grinding scratches are clear, indicating the corrosion scale is extremely thin. Figure 9(b) shows the cross-section morphology of 25Cr steel after 240 h corrosion. A thin film with a thickness of 1 ~ 2 μm can be seen. EDS spot analysis indicated that the film mainly consists of chromium and oxygen.

4. Discussion

Based on the above results, it can be summarized that the corrosion of Cr-bearing steels in multi-thermal fluids is different from that in CO₂-containing solutions below 150 °C. Also, it is different from that in wet CO₂ at high temperatures.

4.1. Formation of corrosion products

In CO₂-containing aqueous solutions, corrosion products on carbon steels are commonly composed of iron carbonate [3–6] which is usually via the combination of Fe²⁺ and CO₃²⁻. As temperature increases, the water vapor would become important corrosive species, which would result in the formation of iron oxides and/or iron hydroxides [10]. For the low Cr steels, the formation of amorphous Cr(OH)₃ (also containing FeCO₃) enhances the protective and self-repairing properties of the corrosion scale, which significantly reduces the
corrosion rate and local corrosion tendency of low Cr steels in CO₂-containing solutions [11, 12]. When the Cr content in steels is sufficient, the formation of FeO, Fe(OH)₃, and Cr₂O₃ is dominated [13, 14].

While in high-temperature carbon dioxide environment at temperatures above 500 °C, iron oxides, iron-chromium spinel oxides, chromia, and undergoing carburization would form on Fe-Cr steels [15]. And the addition of water vapor would enhance the corrosion of Fe-Cr steels and cause breakaway corrosion. It means that the water vapor would break down the protective Cr-rich oxides, and then prompt the formation of less-protective iron oxides.

In the present work, the temperature is greater than that in conventional CO₂-containing solution (<150 °C) while lower than that in conventional high temperature dry/wet CO₂ environments (>500 °C). From the characterization of corrosion scales, it can be speculated that the corrosion scales formed in the multi-thermal fluid are closer to those formed in high temperature wet CO₂ environments. However, differences can be found that the vertical cracks and coarse iron oxides as observed on 9Cr steel.

Different from the conventional CO₂-containing solutions, iron carbonate was absent on surface corrosion products of 3Cr and 9Cr steels. In our previous work, the stability of iron carbonate, hematite, and magnetite was discussed from the thermodynamical perspective, and it was concluded that the iron carbonate is unstable above 200 °C due to the decomposition into iron oxides [16]. Thermodynamical calculation of the standard free energy changes ΔG°fi for the corrosion of iron and chromium with each single corrosive species (including CO₂, H₂O, and O₂) at the studied temperature also indicates that the reaction with oxygen and heated steam is easier to take place. Thus, the corrosion caused by oxygen and heated steam may dominate the whole process. And for the corrosion products of Cr, Fe₃₋ₓCrₓO₄ seems more possible than Cr₂O₃ and Cr(OH)₂ from thermodynamical and kinetic aspects [17]. As a result, Fe₂O₃/Fe₃O₄ and Fe₁₋ₓCrₓO₄ are the main corrosion products for steels with insufficient chromium (e.g. 3Cr and 9Cr steels in the present work), while Cr₂O₃ is dominant for 25Cr steel.

### 4.2. Effect of chromium content
Generally, an increase of chromium content in steels would improve their corrosion resistance due to the formation of the Cr-containing corrosion scale. In CO₂-containing solutions, when the chromium content in low Cr steels increases from 1 wt% to 6.5 wt%, the corrosion rate would be reduced to a great extent due to the protectiveness of Cr(OH)₂ which could block the path of iron’s dissolution, keep apart the corrosion species and decrease the number of active sites of the iron dissolution [18]. However, it seems that 9 wt% Cr in steels cannot form and sustain a protective Cr₂O₃ film on the surface which is the most ideal film for stainless steel with high corrosion resistance. The influence of chromium concentration on high-temperature corrosion of chromium-bearing steels follows a similar law, where the increase of chromium content would prompt the formation of Cr₂O₃ and/or Fe₃₋ₓCrₓO₄ [19–22].

In the present work, a significant difference can be seen on 9Cr steel, which formed the thickest corrosion scale with vertical cracks and a large gap at the scale/substrate interface. These cracks are similar to the findings in Ref. [23] where the mud cracking morphology was ascribed to dehydration. Generally, scale cracks are resulted from intrinsic oxide growth stresses [24] and/or thermal stress, where the growth stress is developed by volume change while thermal stress is developed by temperature change. In addition, the segregation of elements such as Ni and Mo may lead to the formation of depleted area where suffer internal stress and generate micro-crack initiation [25]. Since the Fe₂₋ₓCrₓO₄ appears harder and more brittle than Fe₃O₄, a large number of cracks would form within the Fe₃₋ₓCrₓO₄ scale, and then accelerate the corrosion of 9Cr steel.

### 4.3. Possible corrosion mechanism
Figure 10 shows the schematic illustration of the corrosion scale growth behavior of Cr-bearing steels in CO₂-H₂O-O₂ multi-thermal fluid. For 3Cr steel, the chromium content is too low to form a protective chromium-rich film initially. Therefore, less-protective iron-chromium spinel film would form after immersion. Due to the consumption of chromium, the Cr-depleted zone would form near to the metal surface, leading to the continuous growth of the iron-chromium scale and the formation of the outermost iron-oxide scale. For 9Cr steel, the chromium content is also insufficient to form a protective Cr₂O₃ film initially. Similar to 3Cr steel, iron-chromium spinel film would form after immersion. However, the chromium content in the scale is greater than that for 3Cr steel, due to the relatively higher chromium concentration in 9Cr steel. Under the internal and/or external stress, cracks can nucleate from defects (e.g. pores in the outer scale) and propagate in the corrosion scale by connecting the adjacent cracks [26]. The formation of through-cracks and interface-cracks would damage the protectiveness of the corrosion scale (e.g. Fe-Cr spinel). Corrosion reaction occurs when the corrosive medium reaches the exposed Cr-depleted substrate surface through cracks, which further reduces the protection of the iron-chromium spinel scale. After that, iron in the substrate would reach the outer surface of the iron-chromium spinel scale by solid-phase diffusion, react with corrosive medium, and then form
the outer iron oxide scale. For 25Cr steel, the chromium concentration is sufficient to form and sustain a protective Cr$_2$O$_3$ scale, so that the corrosion rate is extremely low.

5. Conclusions

Corrosion behavior of commercial steels with different Cr content (3 wt%, 9 wt%, and 25 wt%) in simulated CO$_2$-O$_2$-H$_2$O multi-thermal fluid environment was studied by immersion corrosion test and microstructural characterization. Based on the results, the following conclusions can be made.

1. 3Cr and 9Cr steels were corroded to form less-protective compounds (i.e. FeCO$_3$, Fe$_3$O$_4$, Fe$_{3-x}$Cr$_x$O$_4$) instead of protective Cr-rich products (i.e. Cr(OH)$_3$ and Cr$_2$O$_3$), so it showed poor corrosion resistance.

2. Presence of through cracks in the corrosion scale formed on 9Cr steel is the main reason for the deterioration of its corrosion resistance to multi-thermal fluid.

3. For 25Cr steel, the Cr content is sufficient to repair the serious depletion of Cr in the metal near the scale/metal interface in time, so that the protection of the corrosion scale can be sustained. As a result, it exhibits excellent resistance to corrosion of multi-thermal fluids.

Acknowledgments

The authors are grateful for the financial supports from the National Key Research and Development Project (No. 2016YFC0802101), Innovation Capability Support Program of Shaanxi (No. 2019TD-038 and 2020KJXX-063), and Key Research and Development Program of Shaanxi Province (No. 2018ZDXM-GY-171).

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