Pitting Corrosion and Microstructure of J55 Carbon Steel Exposed to CO2/Crude Oil/Brine Solution under 2–15 MPa at 30–80 °C

Haitao Bai 1, Yongqing Wang 1,*, Yun Ma 2, Peng Ren 3 and Ningsheng Zhang 2

1 Institute of Petroleum and Gas Engineering, Southwest Petroleum University, Chengdu 610500, China; baihaitao_xsyu@126.com
2 College of Petroleum Engineering, Key Laboratory of Environment Pollution Control Technology of Oil Gas and Reservoir Protection in Shaanxi Protection, Xi’an Shiyou University, Xi’an 710065, China; mayun_xian123@126.com (Y.M.); zhangnsh_xian@126.com (N.Z.)
3 Xi’an Taijin Industrial Electrochemical Technology Co., Ltd., Xi’an 710016, China; repeng_xian@126.com

* Correspondence: wangyongqing_swpi@126.com

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Abstract: This study aimed to evaluate the corrosion properties of J55 carbon steel immersed in CO2/crude oil/brine mixtures present in the wellbores of CO2-flooded production wells. The main corroded position of wellbore was determined and wellbore corrosion law was provided. Corrosion tests were performed in 30% crude oil/brine solution under the simulated temperature (30–80 °C) and pressure (2–15 MPa) conditions of different well depths (0–1500 m). The corrosion behavior of J55 carbon steel was evaluated through weight-loss measurements and surface analytical techniques, including scanning electron microscopy, energy dispersive spectrometer, X-ray diffraction analysis, and optical digital microscopy. Corrosion rate initially increased and then decreased with increasing well depth, which reached the maximum value of 1050 m. At this well depth, pressure and temperature reached 11 MPa and 65 °C, respectively. Under these conditions, FeCO3 and CaCO3 localized on sample surfaces. Microscopy was performed to investigate corrosion depth distribution on the surfaces of the samples.

Keywords: CO2 corrosion; J55 carbon steel; CO2/crude oil/brine mixtures; corrosion depth distribution

1. Introduction

CO2 is internationally recognized as a major greenhouse gas that accounts for approximately 65% of the total greenhouse gas emissions [1–3]. All countries currently attach considerable importance to environmental issues, particularly global warming caused by CO2. CO2 is used as an oil-flooding agent worldwide because it can effectively reduce crude oil viscosity and residual oil saturation, dissolve gum in reservoirs, and increase permeability and crude oil recovery rate [4,5]. CO2 flooding can reduce CO2-associated air pollution and greenhouse effects. Nevertheless, oil pipe failure caused by CO2 corrosion has become a commonly encountered problem in oilfields and results in great economic losses and safety hazards while severely restricting the development of CO2 flooding technology [4–6].

CO2 corrosion and its control in oil casing and surface transmission pipelines have been important topics in the field of oil and gas exploration. Related studies have focused on the influence of environmental and material factors on corrosion behavior [7–16]. For example, the corrosive medium and environment have been identified as the deciding factors of corrosion rate and morphology. The influence of temperature and pressure on corrosion rate is mainly reflected by the changes that they induce in the protectiveness of the corrosion product layer [7–18]. These changes, in turn, lead to changes in the corrosion rate. With increasing temperature, the CO2 corrosion rate of carbon steel
initially increases and then decreases \([8,9]\), and the maximum value of \(\text{CO}_2\) corrosion rate changes. Li \([10]\) stated that the maximum corrosion rate of P110 tubular steel is attained at 100 °C. The \(\text{CO}_2\) corrosion rate of carbon steel increases under increasing \(\text{CO}_2\) pressure \([11–13]\). Choi \([14]\), however, proposed that the corrosion rates of carbon steel (API 5CT L80) in 25 wt.% NaCl solution at 65 °C negligibly changes with pressure (4, 8, and 12 MPa). The \(\text{CO}_2\) corrosion rate of carbon steel increases with the water cut of water/crude oil mixtures \([15–18]\). Nevertheless, efficient anticorrosion measures cannot be established on the basis of the results of preliminary studies that have largely focused on the influence of a single factor on \(\text{CO}_2\) corrosion and of the limited works that have investigated the laws governing \(\text{CO}_2\) corrosion in the wellbores of \(\text{CO}_2\)-flooded wells. Although studies on aqueous \(\text{CO}_2\) corrosion in crude oil/brine mixtures have been performed, comprehensive studies on \(\text{CO}_2\) corrosion in \(\text{CO}_2\)/crude oil/brine mixtures at different well depths remain unavailable.

This work explored the laws governing \(\text{CO}_2\) corrosion at different depths of \(\text{CO}_2\)-flooded wells. Specifically, this study investigated the common effects of temperature and pressure on \(\text{CO}_2\) corrosion in a \(\text{CO}_2\)/crude oil/brine environment. The average corrosion rate was quantified through the weight-loss method. The surface morphologies of corrosion samples were observed through scanning electron microscopy (SEM). The compositions of corroded surfaces were analyzed through energy dispersive Spectroscopy (EDS) and X-ray diffraction (XRD) analyses. Corrosion depth distribution was analyzed through microscopy, 42 percent of the exposed surface area was studied, which can accurately reflect the surface conditions of the samples after corrosion.

2. Materials and Methods

2.1. Experimental Materials

J55 carbon steel was processed into rectangular samples (50 mm \(\times\) 10 mm \(\times\) 3 mm, \(\varphi = 6\) mm) for the weight-loss test and surface analysis. The chemical composition of J55 carbon steel is shown in Table 1. The samples were placed in acetone to remove surface oil and then immersed in ethanol for 5 min for degreasing and dehydration. The samples were collected, dried with cold air, packed in filter paper, and placed in a dryer for 4–7 h. The sizes and weights of the samples were measured within an accuracy of 0.1 mg.

| Elements (wt.%) |
|----------------|
| C   | 0.34–0.39 |
| Si  | 0.2–0.35  |
| Mn  | 1.25–1.5  |
| P   | \(\leq 0.020\) |
| S   | \(\leq 0.015\) |
| Cr  | \(\leq 0.15\) |
| Ni  | \(\leq 0.20\) |
| Cu  | \(\leq 0.20\) |
| Fe  | Bal.      |

The corrosive medium comprised crude oil and brine. Crude oil was collected from the Chang-8 oil reservoir, a certain block in Changqing Oilfield, and the composition of simulated brine was based on the composition of the water produced in the Chang-8 oil reservoir. The compositions of crude oil and brine are shown in Tables 2 and 3, respectively.

| Property                  | Units       | Value |
|---------------------------|-------------|-------|
| Density (20 °C)           | kg·m\(^{-3}\) | 848.3 |
| Kinematic viscosity (65 °C)| mm\(^2\)·s\(^{-1}\) | 7.254 |
| Acid value                | mg KOH·g\(^{-1}\) | 0.107 |
| Sulfur content            | wt.%        | 0.08  |
| Wax content               | wt.%        | 12.86 |
| Colloid                   | wt.%        | 2.31  |
| asphaltene                | wt.%        | 0.60  |
Table 3. Properties of simulated brine.

| Property   | Units | Value  |
|------------|-------|--------|
| NaCl       | g·L⁻¹ | 18.5028|
| CaCl₂      | g·L⁻¹ | 13.7338|
| MgCl₂      | g·L⁻¹ | 0.5897 |
| Na₂SO₄     | g·L⁻¹ | 0.2440 |
| NaHCO₃     | g·L⁻¹ | 0.0631 |
| Salinity   | g·L⁻¹ | 33.0000|

2.2. Weight-Loss Corrosion Test

The corrosion test was performed in accordance with the weight-loss method with a PARR-4578 autoclave. The schematic of the test is shown in Figure 1. 1 L the mixture of crude oil and brine (v:v = 3:7) was added to the autoclave and purged with a small amount of N₂ for 120 min under 0.5 MPa to remove dissolved O₂. Then, the mixture was subjected to 60 min of injection with high-purity CO₂ under 1 MPa to remove N₂ [19]. Finally, autoclave temperature was increased to the test temperature, and autoclave pressure was increased to the test pressure with high-purity CO₂. The test conditions were maintained for 2 days at a running speed of 0.5 m·s⁻¹ (200 r·min⁻¹).

![Flow chart of the evaluation system for steel corrosion rate (mass-loss method).](image-url)

The corrosion rate of the corroded steel was determined through the mass-loss method in accordance with the ASTM G1-03 Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens [20]. The samples were immediately rinsed with distilled water. Acetone was used to remove crude oil from the surfaces of the samples after corrosion induction. Then, the samples were immersed in acid cleaning solution (500 mL of HCl and 3.5 g of hexamethylenamine brought to volume with water to 1000 mL) for 10 min. At the same time, the corrosion products were removed from the surfaces of the samples, and the samples were collected from the acid cleaning solution. The acid cleaning solution was thoroughly rinsed off from the surfaces of the samples with distilled water. The samples were then twice immersed in ethanol for cleaning and dehydration, collected,
placed on filter paper, dried with cold air, packed in filter paper, and placed in the dryer for 4–7 h. Finally, the samples were weighed to within an accuracy of 0.1 mg. Corrosion rate was calculated using the following formula:

$$r_{corr} = \frac{8.76 \times 10^4 \times (m - m_t)}{S \times t \times \rho}$$  \hspace{2cm} (1)

where $r_{corr}$ is average corrosion rate (mm·y$^{-1}$); $m$ and $m_t$ are the weights of the test sheet before and after the experiment, respectively (g); $S$ is the area of the whole surface in contact with the solution (cm$^2$); $\rho$ is the density of the tested steel (g·cm$^{-3}$); and $t$ is the duration of immersion (h). Each test was performed with three parallel samples. The mean corrosion rate error was calculated on the basis of the results for the three parallel samples.

2.3. Microstructure Observation

After corrosion induction, samples were extracted from the autoclave and rinsed with distilled water and acetone. The surface microstructures of the corrosion product layers on the surfaces of corroded samples were analyzed through SEM with FEI Quanta 600F microscope (Hillsboro, OR, USA). The elemental compositions of the corrosion product layers were determined with OXFORD INCA energy 350. The compositions of the corrosion product characterized were determined through XRD by using Bruker D8 XRD (Billerica, MA, USA).

2.4. Statistics of Corrosion Depth Distribution

The surface depths of the corroded samples cleaned using acid cleaning solution were visualized using an OLYMPUS DSX500 optical digital microscope (Tokyo, Japan). Sample surfaces were subjected to grand horizon 3D image capture under bright-field mode with the adjacent visual threshold splicing mode. As shown in Figure 2, images for the analysis of surface corrosion morphology were acquired at eight observation points on both sides of the same sample through nine-field splicing under 200× magnification and 10% coincidence rate. The display heights of the 3D images were adjusted to the maximum pitting height to ensure that the different ranges of corrosion depths in the same set of images can be represented by different colors. Identical corrosion depth ranges were represented by the same color. Finally, the 3D images were converted into the contour diagrams of corrosion depth distribution.

![Figure 2. Schematic of image acquisition.](image-url)
3. Results and Discussion

3.1. Corrosion Law of the Deepening Well

The CO$_2$-flooded well in Chang-8 Oil Reservoir of a certain block in Changqing Oilfield was taken as an example. The experimental well had a depth of 1550 m, a well-head temperature of 30 °C, pressure of 2 MPa, and well-bottom temperature of 82 °C. Temperature and pressure distributions as a function of well depth are shown in Figure 3. Tests were performed at 0 m (2 MPa, 30 °C), 240 m (4 MPa, 40 °C), 580 m (7 MPa, 50 °C), 800 m (9 MPa, 55 °C), 1050 m (11 MPa, 65 °C), and 1500 m (15 MPa, 80 °C). The liquid produced by the well had a water cut of 70%. The crude-oil composition of the liquid is shown in Table 2. The chemical composition of water produced by the well is shown in Table 3.

![Figure 3. Distribution of temperature and pressure as a function of well depth.](image)

Figure 4 shows the appearance of samples after the removal of corrosion scales. Figure 5 shows the average corrosion rate and the maximum corrosion depth of the samples corroded at different well depths. The average corrosion rate initially increased and then decreased with well depth, except for 1050 m. The general equations for the anodic and cathodic reactions of CO$_2$ corrosion in deoxygenated solution are shown as Equations (2) and (3), respectively [9].

$$\text{Fe} \rightarrow \text{Fe}^{2+} + 2e^- \quad (2)$$

$$\text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3 \quad (3)$$

Temperature and pressure increased as well depth increased. Increasing temperatures reduced the viscosity and protective effect of crude oil on the sample surfaces while intensifying mass transfer between the samples and corrosive medium and accelerating corrosion. Moreover, CaCO$_3$ and FeCO$_3$ deposits generated from the reaction between CO$_3^{2-}$ and HCO$_3^-$ and between Ca$^{2+}$ and Fe$^{2+}$ in the liquid gradually increased and inhibited corrosion development by forming a protective layer on the sample surfaces [8,9]. The increase in CO$_2$ pressure reduced system pH and is conducive for the formation of the protective corrosion product layer [21]. Hence, corrosion acceleration and inhibitory effects simultaneously occurred in the system. At 580 m, the maximum corrosion depth sharply increased, and corrosion type shifted from uniform to local corrosion because the corrosive environment transformed from a CO$_2$/crude oil/brine environment to a subcritical CO$_2$/crude oil/brine environment [22]. The scaling ability of the solution decreased, the surface of the sample could not be completely covered by precipitates, and the progress of the anodic reaction could only be partially prevented [23]. These effects resulted in local corrosion. Therefore, the average corrosion
rate was low (1.7658 mm·year⁻¹), whereas the maximum corrosion depth was high (164.358 µm). Under increasing temperature and pressure, the corrosive environment transformed to the supercritical CO₂/crude oil/brine environment and was dominated by corrosion. Hence, the maximum average corrosion rate was observed at the depth of 1050 m. The possible reduction in the base level of corrosion depth measurement may have reduced the maximum corrosion depths of the samples corroded at 1050 m. With the further increase in well depth, the pH of the corrosion system became almost constant, and the buildup of CaCO₃ and FeCO₃ deposits on the sample surfaces could suppress anodic dissolution [23]. The average corrosion rate decreased. Under dynamic conditions, however, the uneven coverage of the sample surfaces with CaCO₃ suppress anodic dissolution [23]. The average corrosion rate decreased. Under dynamic conditions, however, the uneven coverage of the sample surfaces with CaCO₃ and FeCO₃ deposits resulted in local corrosion and increased the maximum corrosion depth of the samples corroded at 1500 m.

Figure 4. Appearance of samples corroded at the well depths of (a) 0, (b) 240, (c) 580, (d) 800, (e) 1050, and (f) 1500 m after the removal of corrosion scales.

Figure 5. Average corrosion rate and maximum corrosion depth at different well depths.
3.2. Microstructures and Compositions of Corrosion Scales

The SEM images of the samples after corrosion at different temperatures and pressures are shown in Figure 6. The results for the spectral analysis of the corroded sample surfaces are shown in Table 4. Before CO$_2$ reached a supercritical state, the samples exhibited dense surface coatings that mainly consisted of FeC$_3$ and FeCO$_3$ and almost lacked CaCO$_3$ [14,17,19,24]. The average corrosion rates and maximum corrosion depths of the samples were low. After CO$_2$ reached the supercritical state, loose surface coatings that mainly consisted of FeCO$_3$ and CaCO$_3$ formed and failed to provide effective surface protection to the samples [14,19,24]. Thus, the average corrosion rates and maximum corrosion depths of the samples increased.

![SEM images of corrosion scales](image-url)

**Figure 6.** SEM images (2000×) of the surface morphologies of J55 steel after corrosion at (a) 0, (b) 240, (c) 580, (d) 800, (e) 1050, and (f) 1500 m.
Table 4. EDS results for the surfaces of J55 steel after corrosion (%).

| Well Depth/m | 0    | 240  | 580  | 800  | 1050 | 1500 |
|--------------|------|------|------|------|------|------|
| Element      |      |      |      |      |      |      |
| C K          | 25.04| 35.63| 45.36| 17.94| 19.14| 9.92 |
| O K          | 31.97| 22.40| 17.52| 34.22| 42.21| 39.24|
| Cl K         | 0.26 | /    | /    | /    | 0.57 | 1.38 |
| Ca K         | 1.10 | /    | /    | 4.09 | 10.51| 4.70 |
| Mn K         | 0.69 | /    | 0.23 | /    | 0.54 | 1.39 |
| Fe K         | 38.89| 41.97| 36.89| 36.47| 26.78| 42.08|
| Cu K         | 2.04 | /    | /    | 6.12 | /    | 1.29 |
| S K          | /    | /    | /    | 1.16 | 0.26 | /    |
| total content| 100  | 100  | 100  | 100  | 100  | 100  |

Figure 7 shows the XRD spectra of the surface layers of the corroded samples immersed in CO₂/crude oil/brine mixtures under the given temperatures and pressures at different well depths. FeCO₃ is the main product of the CO₂ corrosion of carbon steel [7–19,25]. Similarly, the corrosion product layer that formed on samples immersed in CO₂/crude oil/brine mixtures mainly comprised CaCO₃ and FeCO₃ complex salts. The specific composition of the product layer may be attributed to the isomorphous substitution of metal cations during CO₂ corrosion [23]. When \([\text{Fe}^{2+}] \times [\text{CO}_3^{2-}]\) in the medium exceeded the FeCO₃ solubility product \(K_{sp}(\text{FeCO}_3)\), that is, when the FeCO₃ supersaturation in the medium was \(S = [[\text{Fe}^{2+}] \times [\text{CO}_3^{2-}]]/K_{sp}(\text{FeCO}_3) > 1\), FeCO₃ was deposited on the surfaces of the samples. FeCO₃ deposition can be represented as follows [26]:

\[
\text{Fe}^{2+} + \text{CO}_3^{2-} \rightarrow \text{FeCO}_3(s) \tag{4}
\]

Figure 7. XRD spectra of the surface layers of the corroded samples.

Ca²⁺ in the solution replaced Fe²⁺ in the FeCO₃ crystal and ultimately formed the Fe(Ca)CO₃ complex:

\[
\text{Ca}^{2+} + \text{FeCO}_3(s) \rightarrow \text{Fe}^{2+} + \text{CaCO}_3(s) \tag{5}
\]

3.3. Law of Corrosion Depth Distribution

Figure 8 shows the results for the corrosion depth analysis of the sample corroded in 30% crude oil/brine at the well depth of 800 m. Figure 8a,b were obtained through optical digital microscopy under 200× magnification with nine-field splicing and 10% coincidence rate. Figure 8c shows the contour diagram of the corrosion depth distribution of Figure 8a, which was transformed from...
Figure 8b. The size of one contour diagram of corrosion depth distribution was 7612 µm × 7612 µm, and the total image area observed was 57.94 mm², which accounted for 42% of the sample surface area. Therefore, this corrosion depth analysis method can accurately reflect the surface conditions of the samples after corrosion. Figure 9 shows the contour diagrams of corrosion depth distribution at different observation positions after the removal of corrosion scales from the sample corroded in 30% crude oil/brine at the well depth of 800 m.

![Figure 8](image1)

![Figure 9](image2)

**Figure 8.** Corrosion depth analysis of the sample corroded in 30% crude oil/brine at the well depth of 800 m: (a) corrosion morphology, (b) 3D diagram of corrosion depth distribution, and (c) contour diagram of corrosion depth distribution.

**Figure 9.** Surface height distribution of the sample corroded in 30% crude oil/brine at the well depth of 800 m after the removal of corrosion scales (100×).

The frequency density distribution of corrosion depth on the surface of the sample corroded at the well depth of 800 m is shown in Figure 10. The class intervals used to represent corrosion depth ranges in Figure 10 are the same as colors used to represent corrosion depth ranges in Figure 9. The plot of frequency density was bell-shaped with bilateral symmetry, wherein high values clustered in the center of the plot and low values clustered at both ends of the plot; these characteristics are indicative of typical Gaussian distribution [27,28]. The frequency density distribution maps of samples corroded at different well depths shown in Figure 11 exhibit similar characteristics of Figure 10.
where $w$ is the offset, $y_0 = 0$; $A$ is the area, where $A = 1$; $x$ is corrosion depth (μm); $x_c$ is the expected value (μm); $w$ is twice the standard deviation; $x_c$ determines the location of the distribution curve; and $w$ determines the amplitude of the distribution curve.

**Table 5.** Parameters for the Gaussian model fitting of corrosion depth distribution.

| Well Depth/m | 0      | 240    | 580    | 800    | 1050   | 1500   |
|--------------|--------|--------|--------|--------|--------|--------|
| $x_c$/μm     | 40.250 | 27.680 | 71.189 | 67.189 | 47.268 | 52.028 |
| $w$          | 23.923 | 22.363 | 45.154 | 42.617 | 28.408 | 45.292 |
Table 6 shows the results of variance analysis through the Gaussian model used to fit the curve shown in Figure 11. The Prob > F value of less than 0.01 indicates that the frequency density distribution curve in Figure 11 shows Gaussian distribution. In Gaussian distribution, $x_c$ indicates the average value of the random variables and represents the average corrosion depth. The corrosion of the sample surface may have reduced the datum plane during image acquisition. Moreover, the trend followed by $x_c$ with the change in well depth differed from that followed by the average corrosion rate, especially when the average corrosion rate was high. By adding/subtracting a constant value, the frequency density curve for new random variables generated from random variables with Gaussian distribution was transformed into the translated frequency density curve for former variables in the $x$-direction without changing the shape of the frequency density curve. Therefore, the absence or presence of the datum plane during image acquisition will not affect the $w$ of the fitting results for Gaussian distribution. The trend followed by $w$ with the change in well depth was the same as that followed by the maximum corrosion depth. This similarity indicates the existence of a strong linear correlation between $w$ and corrosion type, as illustrated in Figure 12. Moreover, $w$ was positively related to the span of the frequency density curve. A small $w$ value represents a density distribution curve with a narrow range and limited corrosion depth distribution. These characteristics indicate uniform corrosion. A high $w$ value represents a density distribution curve with a broad range and corrosion depth with broad distribution. These characteristics indicate local corrosion.

![Graph showing correlation between $w$ and maximum corrosion depth](image)

**Figure 12.** Relationship between the maximum corrosion depth and $w$.

### Table 6. Results for variance analysis through Gaussian model fitting.

| Statistics       | DF      | Sum of Squares | Mean Square | F Value | Prob > F |
|------------------|---------|----------------|-------------|---------|----------|
| Regression       | 12      | $8.11 \times 10^{-3}$ | $6.76 \times 10^{-4}$ | 719.55  | 0        |
| Residual         | 54      | $5.07 \times 10^{-5}$ | $9.39 \times 10^{-7}$ |         |          |
| Uncorrected Total| 66      | $8.16 \times 10^{-3}$ |             |         |          |
| Corrected Total  | 60      | $5.11 \times 10^{-3}$ |             |         |          |

**4. Conclusions**

Based on the observed corrosion behavior of J55 carbon steel in CO$_2$/30% crude oil/brine mixtures under the simulated conditions of different well depths (0–1500 m), we conclude the following:

1. The average corrosion rate of J55 carbon steel initially increased and then decreased in the CO$_2$/crude oil/brine environment as partial CO$_2$ pressure increased. Corrosion type shifted from uniform to local corrosion;

2. The main corrosion products on the surfaces of J55 carbon steel were FeCO$_3$ and CaCO$_3$;
(3) The distribution of corrosion depth obeyed Gaussian distribution, and \( w \) was positively correlated with the maximum corrosion depth.

**Author Contributions:** H.B., Y.W. and N.Z. conceived and designed the experiments; P.R. and H.B. were involved in carrying out the corrosion test, SEM and XRD experiments, analyzing the experimental data; H.B. and Y.M. analyzed the data and wrote the paper.

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