



# Observation and analysis of pseudopassive film on 6.5%Cr steel in CO<sub>2</sub> corrosion environment



Bei Wang, Lining Xu\*, Jinyang Zhu, Hui Xiao, Minxu Lu

Corrosion and Protection Center, Key Laboratory for Environmental Fracture (MOE), Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, PR China

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## ABSTRACT

Corrosion rate of 6.5%Cr steel was considerably lower than that of carbon steel and 3%Cr steel. Current density underwent a drop in certain potential range when potentiodynamic polarization curve was tested. Microscope with camera was used for in situ recording of the change in steel surface. The flaking off of the film, which occurred at a certain potential range, was recorded. The results of electrochemical measurements indicated that the film, which mainly consisted of Cr(OH)<sub>3</sub>, was a pseudopassive film. When 6.5%Cr steel was polarized or corroded, pseudopassive film formed. Pseudopassivation of 6.5%Cr steel was closely related to low corrosion rate.

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## 1. Introduction

CO<sub>2</sub> corrosion is a very serious problem in the oil and gas industry and often results in severe damage [1]. Low Cr bearing steels which can improve resistance to CO<sub>2</sub> corrosion by a factor of at least 3, and even up to 10 times and more, whilst maintain a cost penalty less than 1.5 times that of conventional grades of carbon steels become the focus of study in recent years [2–6].

The mechanism of corrosion resistance of 1%–5%Cr steels has been studied by numerous researchers. Pigliacampo et al. [7] indicated that the excellent corrosion resistance of 3%Cr steel was attributed to the formation of a stable protective Cr-rich layer on 3%Cr steel. Wu et al. [8] found that when Cr content increased from 0.5% to 3%, the accumulation of Cr compounds in the corrosion product films increased significantly, leading to a significant change in structure. Thus, the corrosion resistance was improved, and the corrosion scale containing Cr(OH)<sub>3</sub> or Cr oxides was possessed of an anion-selective characteristic. Chen et al. [9] indicated that Cr-containing steels could effectively suppress pitting corrosion and that Cr existed mainly as amorphous compound Cr(OH)<sub>3</sub>. Kermani [10] indicated that a “super protective” layer of low-alloy steels could cause a possible “passivation”. Xu et al. [11]

investigated the corrosion behavior of 3%Cr steels with different microstructures and found that the anode polarization curve of ferrite-pearlite 3%Cr steel and bainite-ferrite 3%Cr steel exhibited pseudopassivation characteristic after 240 and 480 h immersion at 80 °C, 0.8 MPa CO<sub>2</sub>, respectively. Our previous work [12] indicated that, the anode polarization curves underwent a drop in current, which was characteristic of pseudopassivation. The pseudopassivation of low-Cr alloy steel may be closely related to its low corrosion rate. Pseudopassivation characteristic can be obtained through anode potentiodynamic polarization measurement, which may be a fast evaluation method for corrosion resistance. Measuring passivation ability using polarization curve is an effective means of evaluating stainless steels. Similarly, if a low-Cr alloy steel can be evaluated according to its pseudopassivation ability, it can be beneficial for material selection in the oil and gas industry. Although the passive behavior and passivity breakdown of stainless steels have been investigated extensively [13–17], the mechanism of the pseudopassivation of low-Cr steels remains unclear. The direct observation and detailed analysis of a pseudopassive film during potentiodynamic polarization have not been conducted.

In this study, the flaking of the pseudopassive film on 6.5%Cr steel was observed during potentiodynamic polarization. The composition of the film that flaked off 6.5%Cr steel was analyzed using Raman spectra and X-ray photoelectron spectrometer (XPS). Moreover, the relationship between pseudopassivation and corrosion performance was examined through electrochemical methods. This

\* Corresponding author.

E-mail address: [xulining@gmail.com](mailto:xulining@gmail.com) (L. Xu).

**Table 1**  
Chemical composition of 6.5%Cr steel.

Element	C	Si	Mn	S	P	Cr	Mo	V	Nb	Ti	Fe
Content(wt%)	0.07	0.20	0.55	0.003	0.003	6.50	0.15	0.03	0.03	0.03	Bal

**Table 2**  
Composition of the test solution simulating the oilfield formation water.

Composition	NaCl	MgCl <sub>2</sub> ·6H <sub>2</sub> O	CaCl <sub>2</sub>	KCl	NaHCO <sub>3</sub>	Na <sub>2</sub> SO <sub>4</sub>
Content (mgL <sup>-1</sup> )	25318.99	1920.03	2747.25	643.75	519.15	196.73

study contributes to the pseudopassivation mechanism of low-Cr alloy steels in CO<sub>2</sub> environments.

## 2. Materials and methods

### 2.1. Materials and solution

The material used here was 6.5%Cr steel with chemical composition (wt%) listed in Table 1. The specimens, with the size of 50 mm × 10 mm × 3 mm (for weight loss test) and 10 mm × 10 mm × 3 mm (for electrochemical measurements), were ground to 1000 grit, and then cleaned with distilled water and alcohol. The composition of the test solution, simulating an oil field formation water, is shown in Table 2. Prior to the test, the solution was first deaerated by pure CO<sub>2</sub> for at least 8 h. During the test, the CO<sub>2</sub> gas was continuously bubbled into the solution. The pH value of the solution was 5.6.

### 2.2. Experimental methods

#### 2.2.1. Weight loss test

A weight loss test was conducted in a 3-L autoclave to investigate the corrosion rate of 6.5%Cr steel in a CO<sub>2</sub> environment. Prior to conducting weight loss test, the original weight ( $W_0$ ) of each specimen was measured using an analytical balance with an accuracy of 10<sup>-4</sup> g. After 240 h of immersion, the corroded specimens extracted from the autoclave were immediately rinsed with deionized water. The corrosion products were removed through a chemical cleaning procedure in a solution of 500 mL HCl + 3.5 g hexamethylene tetramine + balanced distilled water for 5 min, rinsed and dried, and then reweighed to obtain the final weight ( $W_1$ ) [18]. The corrosion rate ( $V_c$ ) was reported in mm/y according to the weight loss calculated using Eq. (1).

$$V_c = \frac{876000(W_0 - W_1)}{t\rho A} \quad (1)$$

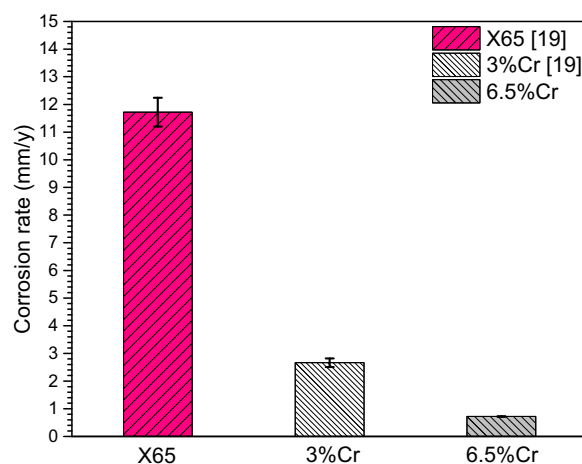
where  $W_0$  and  $W_1$  are the original and final weight of specimen, g, respectively;  $t$  represents the immersion time, h;  $\rho$  is steel density, g/cm<sup>3</sup>; and  $A$  is exposed surface area in cm<sup>2</sup>.

The morphology and energy dispersive X-ray spectroscopy (EDS) analysis of the corrosion scale were investigated using a JSM-6510A SEM and a JED-2300 EDS.

#### 2.2.2. Electrochemical measurements

All electrochemical measurements were performed in a 1-L glass cell with a traditional three-electrode system using a PAR-STAT 4000 electrochemical workstation. The 6.5%Cr steel was used as working electrode, a platinum sheet was used as a counter electrode, and a saturated calomel electrode (SCE) was used as a reference electrode. In the potentiodynamic polarization tests, the potential of the electrodes was swept from -1013 mV to -263 mV (vs. SCE), at a scan rate of 0.166 mV s<sup>-1</sup>.

Five additional fresh specimens were then potentiodynamic polarized from an initial potential of -813 mV (vs. SCE) to the final



**Fig. 1.** Corrosion rate of X65 steel, 3%Cr steel [19] and 6.5%Cr steel exposed under 80 °C and 0.8 MPa CO<sub>2</sub> partial pressure for 240 h.

potentials of -713 mV, -600 mV, -550 mV, -500 mV, -263 mV (vs. SCE), at a scan rate of 0.166 mV s<sup>-1</sup>. The EIS measurements were performed under the open circuit potential (OCP), after the potentiodynamic polarization tests. The frequency range was from 100 kHz to 10 mHz. The perturbing AC amplitude was 5 mV.

Four potentiostatic polarization tests were performed. Four individual fresh specimens were prefilmed through potentiostatic polarization for 1800s at a potential of -550 mV (vs. SCE). The specimens were then potentiostatic polarized for 7200 s at the potentials of -550 mV, -510 mV, -500 mV and -490 mV (vs. SCE), respectively.

#### 2.2.3. Film collection and analysis

A microscope with a camera was used for recording the change of the 6.5%Cr steel surface during the polarization test. After the potentiodynamic polarization test (from -1013 mV to -263 mV (vs. SCE)), the film that flaked off the steel surface was collected, put on the filter paper, cleaned with distilled water and alcohol, and then transferred to a clean glass slide and dried. A LEXTOLS4000 laser-scanning confocal microscope was used for observing and measuring the steel surface and the film. A LabRAM HR Evolution Raman spectrometer and an AXISULTRA-DLD XPS were used for determining its chemical composition.

All tests were performed at 80 °C under stagnant conditions. The weight loss test was conducted at a CO<sub>2</sub> partial pressure of 0.8 MPa. The electrochemical measurements were conducted in CO<sub>2</sub>-saturated solution under atmospheric pressure.

## 3. Results and discussion

### 3.1. Weight loss test

As Fig. 1 shows, the average corrosion rate of the 6.5%Cr steel after 240 h immersion was 0.719 mm/y, which is considerably lower than that of the X65 and 3%Cr steels in identical corrosion environments [19].

Fig. 2 shows the macroscopic morphology of the 6.5%Cr steel after corrosion. As exhibited in Fig. 2(a), the corrosion scale firmly adhered to the substrate. The scale was continuous and compact, providing sufficient protection for the substrate. After the corrosion scales were removed, no evidence of localized corrosion was found. Typical general corrosion morphology was observed (Fig. 2(b)).

Fig. 3 shows the SEM images of the surface and cross-sectional morphologies of the corrosion scale on the 6.5%Cr steel. The scale was intact in the autoclave after 240 h immersion. After the speci-

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