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# Crevice corrosion of N80 carbon steel in CO<sub>2</sub>-saturated environment containing acetic acid

### Y.Z. Li, N. Xu, G.R. Liu, X.P. Guo, G.A. Zhang\*

Key Laboratory of Material Chemistry for Energy Conversion and Storage, Ministry of Education, Hubei Key Laboratory of Materials Chemistry and Service Failure, School of Chemistry and Chemical Engineering, Huazhong University of Science and Technology, Wuhan 430074, PR China

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### 1. Introduction

Carbon steels, as economical materials, are widely used in petroleum and gas industry. However, carbon steels could be susceptible to severe corrosion in the aggressive environment in absence of mitigation measures.  $CO_2$  corrosion is one of major issues resulting in the failure of steel pipes [1–4]. Moreover, organic acids are usually present in oil and gas production and transportation systems. Acetic acid (HAc) is the most abundant organic acid with the concentration up to thousands of ppm in the produced aqueous phase, which makes the corrosion of steel pipes much more complicated.

The effect of HAc on the corrosion behaviour of carbon steel in CO<sub>2</sub>-containing environment has been extensively studied for many years. These studies showed that HAc could significantly promote the uniform corrosion of carbon steel by enhancing cathodic reaction [5–8] and inhibiting the formation of protective ferrous carbonate film on the carbon steel surface in CO<sub>2</sub> environment [9–11]. However, the great threat of HAc is that it could cause localized corrosion, resulting in corrosion failure of carbon steel in short period of time. Gulbrandsen et al. [12] and Zhu et al. [13] found that HAc contributed to the initiation of pitting corrosion on carbon steel and the localized corrosion rate increased with HAc concentration. Amri et al. [14–16] studied the effect of HAc on the

\* Corresponding author. *E-mail address:* zhangguoan@gmail.com (G.A. Zhang).

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### ABSTRACT

Crevice corrosion of N80 carbon steel in CO<sub>2</sub>-saturated environment containing acetic acid (HAc) was studied by electrochemical measurements and surface analysis. No obvious corrosion is observed on the half-electrode outside crevice while severe corrosion occurs on the half-electrode inside crevice, and deep corrosion groove appears at the crevice mouth. Crevice corrosion of carbon steel is triggered by the galvanic effect between the two half-electrodes inside and outside crevice. Crevice corrosion is promoted by increasing the HAc concentration because the galvanic effect between the two half-electrodes inside and outside crevice is strengthened with the increase of HAc concentration.

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localized corrosion of carbon steel by an artificial pit and concluded that the localized corrosion was initiated by the concentration difference of acetic acid and carbonic acid between the bulk solution and pitting hole. Hu et al. [17] studied the crevice corrosion of X52 carbon steel and found that the crevice corrosion was induced by the concentration difference of Cl<sup>-</sup> and pH between the crevice solution and the bulk solution. HAc is also known as the critical factor that contributed to the top of line corrosion by field observation and laboratory study [18-21]. When HAc was present in the gas, it could increase the solubility of iron ions in the condensation water, and then enhance the top of line corrosion [12]. Nesic's group [22,23] reported that the thick porous film contributed to the initiation of the localized corrosion at the bottom of pipeline in containing HAc environment and developed a solution supersaturation model and a scaling tendency model to predict localized corrosion of pipeline in the CO<sub>2</sub> environment. However, the role of HAc in initiation of localized corrosion of carbon steel remains controversial. Some researchers [9,11] argued that HAc promoted the occurrence of localized corrosion by generating porous nonprotective ferrous carbonate film on the carbon steel surface while others [15,17] stated that the localized concentration difference of HAc is the major reason to trigger pitting and crevice corrosion of carbon steel.

Crevice corrosion is the corrosion attack on the metals inside crevice due to geometrical figuration. Presently, most of studies on crevice corrosion were about passive metals in neutral system [24–26], which are susceptible to suffer from crevice corrosion. Corresponding theories were also developed, which mainly included

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composition change theory and IR drop theory. The composition change theory, which was developed to be the classical critical crevice solution theory (CCST) by Oldfield and Sutton [27], was based on the composition change of crevice solution. The IR drop theory was proposed by Picking [28] who pointed out that the IR drop inside crevice was the critical factor to initiate crevice corrosion. Crevice corrosion is immediately initiated without induction period when the electrode potential drops to the reactive region of polarisation curve, which has also been confirmed by laboratory studies [29,30]. Naturally, the crevice corrosion of passive metal can be basically comprehended by these two theories. However, it's still unclear how the crevice corrosion occurs in active system, especially under anaerobic environment.

In the present study, the crevice corrosion behaviour of N80 carbon steel in CO<sub>2</sub>-saturated environment containing HAc was investigated by electrochemical measurements and scanning electron microscopy (SEM) analysis. Effect of concentration of HAc on the crevice corrosion of N80 carbon steel was quantitatively discussed. It is expected that this study could promote the understanding of the localized corrosion mechanism of carbon steel in CO<sub>2</sub>-saturated solution containing HAc.

### 2. Experimental

### 2.1. Material and solution

All of the specimens used in this study were cut from API N80 carbon steel pipeline, with chemical composition (wt.%): 0.42% C, 0.24% Si, 1.55% Mn, 0.012% P, 0.004% S, 0.051% Cr, 0.18% Mo, 0.005% Ni, 0.01% Ti, 0.06% Cu, and Fe balance. The specimens for electrochemical measurements were machined into rectangular cuboid with dimension of  $20 \times 10 \times 3$  mm, which half of specimen (exposed area of  $1 \text{ cm}^2$ ) was outside crevice and half of specimen (exposed area of  $1 \text{ cm}^2$ ) was inside crevice. To be compared, specimens with dimension of  $10 \times 10 \times 3$  mm (exposed area of  $1 \text{ cm}^2$ ) were also used for electrochemical tests without crevice or totally inside crevice. All of the specimens were embedded in epoxy resin, and then abraded with 800 grit silicon carbide paper, degreased with acetone and cleaned with distilled water.

The test solution was 1.65 wt% NaCl solution with different concentrations of HAc, which was prepared from analytical grade reagent and deionized water. Prior to test, the solution was purged with pure  $CO_2$  gas (99.95%) for 4 h. Specimens were then immersed into the solution and the  $CO_2$  gas-purging with a flux of 20 mL/min was maintained to ensure a full saturation throughout the test. All of the experiments were performed at atmosphere pressure and 30 °C.

### 2.2. Configuration of crevice and electrochemical measurements

The schematic diagram of the setup and the configuration of crevice for open circuit potential (OCP) and galvanic current measurements was shown in Fig. 1. Two identical working electrodes (WEs) were embedded together in epoxy resin with a distance of 3 mm, as shown in Fig. 1a. One of them (WE1) was placed inside crevice and another one (WE2) was exposed to the bulk solution. The exposed area of each of these two electrodes was 1 cm<sup>2</sup>. Another epoxy mould was used as crevice former. A rectangle polytetrafluoroethylene (PTFE) gasket (one side of the rectangle PTFE gasket was cut) with a thickness of 300  $\mu$ m was located between these two epoxy resin moulds to create a crevice of 300  $\mu$ m. Although PTFE is a compressible material, the change in the thickness of the rectangle PTFE gasket can be neglected after fastening these two epoxy resin moulds together by hand. A PTFE tube, which had a porous ceramics cap at one end, was embedded

in the crevice former. This PTFE tube was filled with test solution. Then an Ag/AgCl reference electrode was inserted the PTFE tube as reference electrode to measure the potential of WE1 (inside crevice). Another similar Ag/AgCl reference electrode located at bulk solution to measure potential of WE2 (outside crevice). The test solution (1.65% NaCl), not the usually used KCl solution, was used in the PTFE tube in order to reduce the possible effect on the solution inside crevice. Moreover, the porous ceramics located at the bottom of the PTFE tube had high density in present work. No obvious solution leaked out after 24 h. However, to be convenient, all the electrode potentials in this work were converted into the potential vs. Ag/AgCl (1 mol/L KCl) according to the potential difference of Ag/AgCl electrode in the electrolyte with different Clconcentrations. The galvanic current between WE1 and WE2 was measured by using a zero resistance ammeter (ZRA). In some measurements, such as galvanic current and weight loss measurements, WE1 and WE2 were connected together. For comparison, some measurements, such as weight loss and OCP measurements, were also performed in which these two electrodes were not connected.

The schematic diagram of the setup for electrochemical impedance spectroscopy (EIS) and pH measurements was showed in Fig. 2. Electrodes with an exposed area of  $2 \text{ cm}^2$  were used in the test which half of electrode located in the crevice and another half of electrode was exposed to bulk solution. Ag/AgCl reference electrode (inserted in a PTFE tube), platinum sheet, and W/WO<sub>3</sub> microelectrode were embedded in the crevice former (epoxy resin), which were used as reference electrode, counter electrode and pH microelectrode, respectively, for the electrochemical and pH measurements inside crevice. Another Ag/AgCl reference electrode, platinum sheet, and W/WO<sub>3</sub> microelectrode in bulk solution were used for electrochemical and pH measurements in bulk solution, as shown in Fig. 2. When the working electrode, the reference electrode (RE1) and counter electrode (CE1) inside crevice were connected to the electrochemical workstation, the electrochemical information of the half-electrode inside crevice was collected. When the working electrode, the reference electrode (RE2) and counter electrode (CE2) outside crevice were connected to the electrochemical workstation, the electrochemical information of the half-electrode outside crevice was obtained. By using this setup, the difference in the corrosion behaviour of the parts of electrode outside and inside crevice can be determined by electrochemical measurements.

An electrochemical workstation was used for electrochemical measurements. EIS was measured at OCP with a sinusoidal alternating amplitude of 5 mV and frequency range from 10,000 Hz to 0.01 Hz.

### 2.3. Weight loss measurement

Weight loss measurement was used to determine the corrosion rate of the specimens without and inside crevice. For the weight loss measurement, the size of each specimen was  $10 \times 10 \times 3$  mm. Two kinds of specimens were used: one kind of specimens without crevice and another kind of specimens totally inside crevice. One copper wire was soldered to each specimen, and then weighed and sealed with epoxy with only one face (area of 1 cm<sup>2</sup>) exposed to solution. After corrosion test, the epoxy and corrosion products on the specimens were removed, and then the specimens were weighed again to determine the weight loss. Weight loss measurement was performed with these two kinds of specimens when they disconnected or connected each other.

#### 2.4. Morphology observation

The corrosion morphologies of the specimens after corrosion in the solution with different concentrations of HAc were char-

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2

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