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Effects of solution composition on corrosion behavior of 13 mass% Cr martensitic stainless steel in simulated oil and gas environments

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Abstract: The effects of CH3COONa and CO2 on the corrosion behavior of 13 mass% Cr martensitic stainless steel in simulated oil and gas environments were investigated with electrochemical and surface analysis techniques. The electrochemical results showed that a plateau region and sudden increase in the current density of the anodic polarization curves. The current density of the plateau region decreased with increasing CH3COONa concentrations. The pitting corrosion potential shifted to the positive direction with increasing CH3COONa concentrations, and shifted to a negative value by adding CO2. From the surface analysis, a Cr enriched layer had formed on the sample surface after immersion tests, and the thickness of this layer became thinner with increasing CH3COONa concentration. The surface analysis results after the immersion tests suggested the presence of CH3COO− or HCO3− on the surface.

Keywords: 13 mass% Cr martensitic stainless steel; CH3COONa; CO2; XPS; GDS; oxide film structure
1. Introduction

Oil and gas fields are very severe environments for metals, and high corrosion resistance and strength steels are generally used. Stress corrosion cracking (SCC), sulfide stress cracking (SSC) and hydrogen embrittlement (HE) are principal corrosion problems of steels used in oil and gas environments. The corrosion behavior of steels has been examined in simulated oil and gas environments [1–16]. Kimura et al. reported that the modified 13 mass% Cr stainless steel showed excellent resistance to CO₂ corrosion and resistance to SSC [2]. Sunaba et al. investigated the effects of chloride ion concentration on the passive and corrosion product films of modified martensitic stainless steels at high temperatures in CO₂ environments [7]. They reported that molybdenum improved stability of the corrosion product films in high temperature CO₂ and the presence of chloride ions. Hashizume et al. introduced a solution flow type micro-droplet cell and a glow discharge optical emission spectrometer (GDS) to investigate the electrochemical behavior of low carbon 13 mass% Cr welded joints and the effect of a post welded heat treatment (PWHT) in oil and gas environments [17]. They reported that the PWHT eliminated the Cr depleted layer formed during welding, and reduced the SCC susceptibility.

Hashizume et al. also investigated the effects of chemical composition and tempering of martensitic stainless steel on corrosion rates and pitting potentials in simulated CO₂ gas wells [13]. They reported that the corrosion rate and the pitting potential in environments simulating CO₂ gas wells were governed mainly by the amount of Cr in the steel matrix. The effect of acetate ions on the structure of surface oxide films and corrosion behavior of 13 mass% Cr stainless steel in 25% NaCl solutions were investigated by X-ray photoelectron spectroscopy (XPS) and electrochemical techniques [18]. It was found that the intensity ratios of both Fe⁰/(Fe³⁺ + Fe²⁺ + Fe⁰) and Cr⁰/(Cr³⁺ + Cr⁰) decrease with increasing concentration of CH₃COONa, and after a minimum increased with further increasing in CH₃COONa concentration. Fierro et al. applied XPS for the analysis of the corrosion film formed on 13 mass% Cr martensitic stainless steel in CO₂-H₂S-Cl⁻ [19,20]. The presence of H₂S resulted in more the formation of the less protective Fe sulfide-rich and Fe oxide-rich layers, while the presence of CO₂ resulted in Cr oxide enrichment in the outermost layers of the film. Islam et al. investigated the corrosion layer on pipeline steel in CO₂ containing environments (sweet environments) [21].

However, the effects of environmental parameters such as pH, the concentration of CH₃COONa, and CO₂ on corrosion behavior of stainless steels have not been fully elucidated. The purpose of this study is to investigate the effects of the concentrations of CH₃COONa and CO₂ on the corrosion behavior of 13 mass% Cr martensitic stainless steels in simulated oil and gas environments.

2. Materials and method

2.1. Samples and preparation

Plates of 13 mass% Cr martensitic stainless steel (12.4 mass% Cr, 0.2 mass% C) with 3 mm thickness were used as samples. The sample size for electrochemical and XPS analysis was 7 × 15 mm, and for Fourier transform infrared spectroscopy (FT-IR) analysis was 15 × 15 mm.
For electrochemical measurement, Cu wires were soldered to the samples and then embedded in epoxy resin. The exposed surface of the embedded samples was ground with SiC abrasive paper from #240 to #1200 grit size. For immersion corrosion tests, samples were embedded in epoxy resin. The exposed surfaces of the samples were ground with SiC abrasive paper from #240 to #2400 grit size. The samples were ultrasonically cleaned in ethanol and in highly purified water, before the tests.

2.2. Solutions

Solutions of 5% NaCl with 0, 0.4, 4.0 g/L CH₃COONa were used as test solutions. The pH of the solutions was adjusted to 3.5 by HCl. Before the experiments, N₂ gas was bubbled through the solutions for 3.6 ks to reduce the concentration of dissolved O₂ as low as possible, and then CO₂ gas was bubbled as needed. The solution temperature was 353 K, and detailed procedures of the gas bubbling and pH adjustment are illustrated in Figure 1. During experiments, N₂ or CO₂ gas flowed over the solutions.

![Figure 1](image-url)  
Figure 1. Schematic representation of the experimental procedure of this study.

2.3. Anodic potentiodynamic polarization measurement

Anodic potentiodynamic polarization measurements were carried out in a three electrode cell. A Pt plate and Ag/AgCl sat. KCl was used as counter and reference electrodes respectively. The samples were immersed in the solutions for 21.6 ks and measured the immersion potential, \( E_{\text{im}} \), and then the polarization measurements with a scan rate of 0.5 mV/s were started from \( (E_{\text{im}} - 10) \) mV until current density reached 1 mA/cm². After the measurements, samples were rinsed with highly purified water and dried in a desiccator, and then the surface observations were performed with an optical microscope. Samples with crevice corrosion were discarded.

2.4. Immersion corrosion tests and surface analysis

The samples were immersed in the solutions for 21.6 ks. After the immersion, samples were
rinsed with highly purified water and dried in the desiccator. The sample surfaces were analyzed by XPS (JEOL Ltd., JPS-9200). For the XPS analysis, Mg Ka (1253.6 eV) was used as the X-ray source, and the analysis area was 1 × 1 mm. The XPS depth analysis was carried out by sputtering with Ar ions. The sputtering time was converted to depth by the sputtering rate of SiO2. The wide range of depth profiles of Fe, Cr, and O were obtained by GDS (Horiba-Jobin Yvon, JY 5000 RF). The analysis area was 4 mm in diameter. The FT-IR (JASCO, FT-IR 4700) was used to determine the organic substances on the samples. The IR spectra were measured by reflection absorption spectroscopy (RAS) with the resolution of 1 cm⁻¹. The background spectra were obtained with gold evaporation mirror and the subtracted the obtained samples spectra. The surface observation was carried out with a scanning electron microscope, SEM (JEOL Ltd., JSL6510-LA).

3. Results and discussion

3.1. Anodic polarization curves

Anodic polarization curves were measured after 21.6 ks immersion and are shown in Figure 2. All curves except for 0 g/L CH3COONa in the presence of CO2 have the plateau region in the current, which implies that the corrosion reaction reaches a temporary steady state. The currents of plateau regions are higher than that of general passive current [22]. This result suggests that the passive state may not be achieved in the experimental conditions of this study. The current suddenly increased as the potential moves to a positive direction, which may imply the initiation of pitting corrosion. Hereafter, the plateau region in the current is called the average current density of the flat region, \( i_{\text{flat}} \), and the potential at 1 mA/cm² is called pitting potential, \( E_{\text{pit}} \).

![Figure 2. Anodic polarization curves after 21.6 ks immersion in solutions with (a) absence of CO2 and (b) presence of CO2.](image)

The relationships between these two parameters, \( i_{\text{flat}} \) and \( E_{\text{pit}} \), and the CH3COONa concentration are shown in Figure 3. In Figure 3a, the \( i_{\text{flat}} \) decreases with increasing CH3COONa concentration independent of CO2 bubbling, implying that adding CH3COONa inhibits the corrosion reaction. In Figure 3b, \( E_{\text{pit}} \) obtained in the solutions of the presence of CO2 increases with increasing
CH$_3$COONa concentration, however, the effect of CH$_3$COONa addition is not significant in the solutions of an absence of CO$_2$.

The surface optical images of the samples after the polarization tests are shown in Figure 4. Dark spots which may be related to pitting corrosion are observed, and the number of the spot decreased with increasing concentration of CH$_3$COONa. This result coincides with Figure 3b.

**Figure 3.** (a) $i_{\text{flat}}$ and (b) $E_{\text{pit}}$ as a function of CH$_3$COONa concentration.

**Figure 4.** Surface images of samples after polarization tests. (a) and (b) 0 g/L CH$_3$COONa, (c) and (d) 0.4 g/L CH$_3$COONa, (e) and (f) 4.0 g/L CH$_3$COONa. (a), (c), (e) absence of CO$_2$; (b), (d), (f) presence of CO$_2$. 
3.2. XPS spectra

The sample surfaces were examined by XPS after immersion in 4.0 g/L CH₃COONa absence of CO₂ for 21.6 ks. Figure 5 shows XPS narrow scan spectra of (a) Fe 2p3/2, (b) Cr 2p3/2, (c) O 1s, and (d) C 1s at different depths. In the narrow scan spectra of Fe 2p3/2 (Figure 5a), no peak is observed from 0 and 10 nm, while peaks around 707 eV are measured depth deeper than 10 nm. The peaks are attributed to Fe⁰⁺ [23–27], and the intensity of the peak increases with depth. In the narrow scan spectra of Cr 2p3/2 (Figure 5b), the peaks around 577 eV are observed depth from 0 nm to 70 nm. The peaks are attributed to Cr³⁺ [23,25–27], and the intensity decreases with depth. The peaks around 574 eV are appeared after sputtering. The peaks are attributed to Cr⁰⁺ [23,25–27], and the intensity of the peak increases with depth.

![XPS spectra](image)

**Figure 5.** XPS narrow spectra of the sample immersed in 4.0 g/L CH₃COONa solution absence of CO₂ at 353 K for 21.6 ks.

In Figure 5c, the peaks around 532 eV attributed to OH⁻ [23,25–27] is observed on the surface.
(at 0 nm depth). As sputtering, the peaks around 530 eV appear. The peaks are attributed to O$^{2-}$ [23,25–27], and the intensity changed with depth. Form Fe, Cr and O narrow scan spectra, corrosion product layer formed on the steel surface has a double layer structure as an outer layer; Cr hydroxide, and an inner layer; Cr oxide. In Figure 5d, peaks are observed on the surface (at 0 nm depth), and no peak is observed after sputtering. The peaks could not be attributed, however, the result suggests that the CH$_3$COO$^-$ in the solution may exist only on the surface.

To investigate the composition of sample surface after immersion, a composition ration of Fe and Cr was calculated from XPS narrow scan spectra. Figure 6 shows composition ratio of Fe/Cr as a function of depth. The ratio increases with increasing depth, however, no significant difference with solution composition is observed. These results indicate that the composition ratio of Fe/Cr ratio at the surface is independent of the chemical composition of the used solution and CO$_2$ bubbling.

![Composition ratio of Fe/Cr obtained from XPS spectra as a function of depth](image)

**Figure 6.** Composition ratio of Fe/Cr obtained from XPS spectra as a function of depth.

3.3. GDS and FT-IR analysis

It is needed to investigate the depth profile of elements in the sample after the immersion tests. GDS was applied to measure the depth profiles of Fe, Cr and O. The depth profile of the sample immersed in 4.0 g/L CH$_3$COONa solution absence of CO$_2$ is shown in Figure 7a. The Fe intensity is low at the initial and it increases sharply as sputter time longer than 5 s. The Cr intensity shows a peak about 2 s and a minimum about 5 s. The O intensity shows peak around 1 s and becomes low after 5 s sputtering. These results suggest that the Cr rich layer exists until Cr intensity reached minimum. The sputter time at a minimum of Cr intensity decided from GDS spectra. Figure 7b shows the sputtering time at a minimum of Cr intensity as a function of CH$_3$COONa concentration. The sputter time at minimum of Cr intensity decreases with increasing CH$_3$COONa concentration. This result indicates that Cr rich layer becomes thinner with increasing CH$_3$COONa concentration.

It was suggested that organic substances in the solution adsorbed on the surface from XPS analysis. FT-IR applied to analyze the organic substances on the surface, and obtained spectra are shown in Figure 8. The background spectrum was subtracted to reduce peaks related to CO$_2$ and H$_2$O in the environments. However, the residue of CO$_2$ related peaks are still observed around 2400–2300 cm$^{-1}$ [28,29]. The peaks around 1700–1300 cm$^{-1}$ are attributed to CH$_3$COO$^-$ or
HCO$_3^-$ [30,31], and the intensity of the peaks increases with increasing CH$_3$COONa concentration. The intensity obtained in presence of CO$_2$ shows larger value independent of CH$_3$COONa concentration. These results indicate that CH$_3$COO$^-$ or CO$_3^{2-}$ adsorbs on the surfaces.

**Figure 7.** (a) GDS depth profile of the sample immersed in 4.0 g/L CH$_3$COONa solution absence of CO$_2$ at 353 K for 21.6 ks. (b) Sputtering time at minimum of Cr intensity as a function of CH$_3$COONa concentration.

**Figure 8.** FT-IR spectra of the samples (a) absence of CO$_2$ and (b) presence of CO$_2$ after the immersion tests.

3.4. **Surface images after immersion corrosion test**

Surface morphologies of the samples after 21.6 ks immersion are shown in Figure 9. In Figure 9a,b, uneven surface with dark lines are observed in 0 g/L CH$_3$COONa. In Figure 9c,d, a small number of dark lines are observed in 0.4 g/L CH$_3$COONa. However, their size and length are almost the same as observed in 0 g/L CH$_3$COONa. Smooth surfaces without dark lines are observed in 4.0 g/L CH$_3$COONa (Figure 9e,f). There are precipitates on the surfaces in all samples, and size and number are changed with CH$_3$COONa concentration. Little difference of the surface morphology is
observed in the presence and absence of CO\textsubscript{2} in the solutions.

![Figure 9](image_url)  
**Figure 9.** SEM images of the samples after 21.6 ks immersion. (a) and (b) 0 g/L CH\textsubscript{3}COONa, (c) and (d) 0.4 g/L CH\textsubscript{3}COONa, (e) and (f) 4.0 g/L CH\textsubscript{3}COONa. (a), (c) and (e) Absence of CO\textsubscript{2}, (b), (d) and (f) Presence of CO\textsubscript{2}.

4. Discussion

4.1. Effect of CO\textsubscript{2} gas bubbling

It is known that dissolved CO\textsubscript{2} produced carbonic acid and H\textsuperscript{+}.

\[
\text{CO}_{2} + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3 \quad (1)
\]

\[
\text{H}_2\text{CO}_3 \rightarrow \text{HCO}_3^- + \text{H}^+ \quad (2)
\]

In this experiment, it is considered that the cathodic reaction is the hydrogen evolution reaction because the pH of the used solutions was 3.5. The reaction may change the pH of the solution during polarization measurement. If CO\textsubscript{2} gas flowing during the experiments, Eqs 1 and 2 may occur
continuously and the carbonic acid acts as the source of H\(^+\). Therefore, the pH may be maintained during the polarization tests. This is a possible reason for \(i_{\text{flat}}\) of 0 g/L CH\(_3\)COONa in presence of CO\(_2\) showed the largest value as shown in Figure 3a. The supply of H\(^+\) by Eq 2 in presence of CO\(_2\), make it possible to increase corrosiveness of the solution and may reduce \(E_{\text{pH}}\) as shown in Figure 3b.

4.2. Effect of CH\(_3\)COONa and CO\(_2\) on corrosion process

In this experiment, \(i_{\text{flat}}\) decreased with increasing concentration of CH\(_3\)COONa, while a small difference in surface composition was observed by surface analysis. The thickness of the Cr rich layer changed with the concentration of CH\(_3\)COONa that was observed by GDS, and the adsorptions of CH\(_3\)COO\(^-\) or HCO\(_3^-\) on the surface were observed by FT-IR. These results may closely relate to corrosion process. Schematic representation of the corrosion process in these experimental conditions is shown in Figure 10. A highly protective layer is formed on the steel surface when the absence of CH\(_3\)COONa and CO\(_2\) in the solution (Figure 10a). The corrosion is progressed by Cl\(^-\). The Cr rich layer is formed on the surface because of preferential Fe dissolution [32,33]. The thickness of the Cr rich layer may become thicker and then reaches a certain value as metal dissolution. In the case of CH\(_3\)COONa and CO\(_2\) are present in the solutions, CH\(_3\)COO\(^-\) and HCO\(_3^-\) adsorb on the steel surface (Figure 10b). The absorbed layer may inhibit the dissolution of metals, therefore, the amount of dissolved metals decreases and relatively thin Cr rich layer is formed.

![Figure 10](image)

**Figure 10.** Schematic representation of the corrosion process in these experimental conditions.

5. Conclusion

The influences of CH\(_3\)COONa and CO\(_2\) on the corrosion behavior of 13 mass% Cr martensitic
steels in simulated oil and gas environments were investigated with electrochemical and surface analysis techniques. Following conclusions may be drawn:

(1) There was the plateau region of the current density, $i_{\text{flat}}$, in the anodic polarization curves, and the $i_{\text{flat}}$ decreased with increasing CH$_3$COONa concentration. In the case of the solutions with CH$_3$COONa, little effect of CO$_2$ on the $i_{\text{flat}}$ was observed. A pitting potential, $E_{\text{pit}}$ shifted to the positive direction as increasing CH$_3$COONa concentration, and showed the negative value by adding CO$_2$.

(2) The Cr enriched layer formed on the surface of the sample after the immersion test, and the thickness of the layer became thinner as increasing CH$_3$COONa concentration. The adsorbed CH$_3$COO$^-$ and HCO$_3^-$ on the sample surfaces prevented the corrosion.

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**Conflict of interest**

All authors have no conflict of interest directly relevant to the contents of this article.

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