Effects of Iron Oxidation State and Chromium Distribution on the Corrosion Resistance of High Interstitial Stainless Steel for Down-Hole Application

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Abstract: The corrosion properties of Fe-Cr-Mn-C-N high interstitial austenitic stainless cast steels were investigated for down-hole application in sour environments. The two cast alloys contained 0.66% and 0.71% of total nitrogen and carbon. The corrosion properties of the alloys that were solution-treated and fast-cooled were directly responsible for high corrosion resistance in NaCl solution, including resistance to pitting corrosion resulting from a better distribution of chromium chemical compound in the high interstitial stainless cast steel. However, the sour corrosion resistance of the alloys decreased with the fast cooling rate, which can be attributed to the increased amount of ferrite containing Fe²⁺, which causes iron sulfide precipitate formation in H₂S.

Keywords: corrosion resistance; chemical compound; bonding status; high interstitial stainless cast steel; down-hole

1. Introduction

Serious corrosion is encountered in sour gas wells and reservoirs during the extraction of oil and gas. In order to ensure safety and the long-term development of gas wells, materials with superior corrosion resistance are required for well-production systems [1]. Austenitic stainless cast steels are widely used for the development of gas wells in the sour environment due to their superior mechanical performance. Among these, nickel-based austenitic stainless cast steels are the most common.

These austenitic stainless cast steels have a corrosion resistance when they form a passivation layer of chromium (III) oxide (Cr₂O₃) to prevent oxidation. However, degradation in corrosion resistance due to undesirable phases, such as the formation of various chemical compounds (chromium carbides and nitrides) in the steel, is a typical problem for down-hole applications [2,3]. The deleterious effects of the precipitation as either chromium carbide or nitride on pitting corrosion, with the formation of a chromium depletion zone adjacent to those compounds, have been reported and sensitization has occurred as a result [4].

There are many efforts underway to develop substitute materials for these steels, and high nitrogen stainless steels (HNS) have been proposed because of the beneficial effect of nitrogen on corrosion.
properties. The characteristics which can enhance the mechanical and corrosion properties of the steels are attributed to the interstitial solid solution strengthening effects \([5–9]\). Despite the promising properties of nitrogen stainless steels, the development of these types of materials has faced engineering difficulties because of the limited solubility of nitrogen in iron, and high interstitial stainless cast steels (HISSs) have been developed by adding carbon to a Fe-Cr-Mn-N alloy. While carbon generally decreases the solubility of nitrogen in steels \([10]\), recent studies have shown that the addition of carbon resists the loss of nitrogen content through delta ferrite formed during solidification \([11]\). It gives several advantages, such as cost-saving in manufacturing, i.e., avoiding the high-pressure process required for HNS, and excellent performance, including improved wear properties \([5,12–14]\).

In this study, a new grade of Fe-Cr-Mn-C-N high interstitial stainless cast steel with superior mechanical properties was developed for down-hole applications in sour environments. Previous research on Fe-Cr-Mn-C-N high interstitial stainless steels revealed the influence of heat treatment on the mechanical property and wear resistance of the high interstitial stainless steels \([13,15]\). However, few researchers have characterized the corrosion resistance of the high interstitial stainless steel. Thus, this study focused on the corrosion properties of developed alloys, investigated in different conditions of solution treatment, cooling rate, and carbon and nitrogen content. Electrochemical measurements were carried out through potentiodynamic polarization testing. The work was conducted to attempt to find a correlation between the corrosion properties, such as pitting corrosion resistance of the developed alloys and distribution of chromium chemical compounds in HISS as a function of varying conditions. In addition, accelerated corrosion testing was carried out using a high temperature and pressure autoclave at low pH to investigate if the corrosion resistance of the alloys in sour environments was governed by the bonding state of iron.

2. Experimental Procedure

Fe-Cr-Mn-C-N high interstitial austenitic alloys CN66 (0.38 wt.% N + 0.28 wt.% C) and CN71 (0.44 wt.% N + 0.27 wt.% C) were fabricated using a 500 lb induction melting furnace operating in an air atmosphere with nitrogen inserted before the initial heating process. Nitrogen is one of the key elements in high interstitial stainless cast steels (HISSs) and it is important to ensure that it is retained in the HISS alloys during melting. Generally, melting at a high pressure of nitrogen is required to obtain the required nitrogen content, but to avoid a costly high-pressure process, the carbon and nitrogen contents were targeted at 0.5–1 wt.%, which can be obtained readily by melting at ambient pressure. To obtain superior corrosion-resistant properties, chromium content was aimed at a minimum of 18 wt.%, and manganese was targeted to 8–10 wt.% to stabilize the austenite and to act as a deoxidizer. Nickel, which is conventionally used as an austenite stabilizer in austenitic steels, was reduced to less than 1.0 wt.%. CN66 and CN71, Fe-Cr-Mn-C-N high interstitial stainless cast steels (HISSs), were developed, and Table 1 shows their chemical compositions. It shows that the nitrogen and carbon were successfully incorporated in alloys as much as 0.66% (CN66, C + N = 0.66%) and 0.71% (CN71, C + N = 0.71%) of carbon and nitrogen content. A significant amount of chromium content (over 23%) and 8% of manganese was dissolved in CN66 and CN71.

| Table 1. Chemical compositions of stainless steel 316L, CN66 and CN71, Fe-Cr-Mn-C-N high interstitial stainless cast steels (unit: mass %). |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Alloy           | C               | N               | Cr              | Mn              | Ni              | Si              | Mo              | Fe              |
| 316L            | <0.03           | <0.10           | 16.0–18.0       | <2.0            | 10.0–14.0       | 0.75            | 2.0–3.0         |                  |
| CN66            | 0.28            | 0.38            | 23.9            | 8.33            | 0.79            | 0.75            | 0.56            | Bal.            |
| CN71            | 0.27            | 0.44            | 23.5            | 8.65            | 0.82            | 0.93            | 0.40            |
The chemical composition of the alloys was measured using X-ray fluorescence spectroscopy (XRF) and the interstitial elements (carbon and nitrogen) were analyzed using the Eltra CS 2000 C/S analyzer and Eltra ONH-200 (ELTRA GmbH, Haan, Germany). Solution heat treatment was carried to distribute carbon and nitrogen in alloys at 1473.15 K (1200 °C) for 2 h. The heat-treated specimens were cooled with air (>1 K/s) and water (>130 K/s) media [16].

Potentiodynamic polarization testing was used to examine the overall corrosion behavior of a system to understand the effect of the heat treatment and different cooling rates with air (>1 K/s) and water (>130 K/s) media. For electrochemical measurements, a typical three-electrode electrochemical cell system was accepted, with a saturated calomel electrode (SCE) as a reference electrode, a platinum plate as a counter electrode, and test specimen employed as working electrode which the area of the specimens was in a range of 0.55 to 1 cm² and used to calculate the current density. The polarization scans were examined under atmospheric pressure in a 3.5 wt.% NaCl solution at pH 6 (scan rate: 0.16 mV/s, potential range: −0.4 to 1.3VSCE). Analysis of the data typically yields corrosion potential, corrosion rate, and the potential region for passivity. Compositional analyses of the steels to determine the chromium content and its overall distribution were carried out by electron probe microanalysis (EPMA, EPMA-1600 series, Shimadzu, Kyoto, Japan). The ferrite volume fraction was measured by ferrite content measurement, Fischer FERITSCOPE MP30. The tests were performed with 20 replicates of each condition. The sour-brine environment immersion test is essential to verify if the materials are suitable for use in down-hole applications in sour environments. The investigation of sour corrosion resistance of CN66 and CN71 was conducted with an immersion test in high-pressure and high-temperature (120 °C) autoclave for 40 days. The area of the specimens is given in Table 2. The total pressure was 8 bar (PH2S = 37.5%, PCO2 = 62.5%) and the solution was 3.5 wt.% NaCl. The change in mass of the specimens was measured to assess the alloys’ degree of reaction to this highly aggressive environment.

3. Results and Discussions

The corrosion properties of the developed alloys were investigated according to different conditions, such as solution treatment (1473.15 K for 2 h), different cooling rate (air (>1 K/s), water (>130 K/s)), and carbon and nitrogen content (0.66 wt.%/CN66, 0.71 wt.%/CN71). Figure 1 shows the potentiodynamic polarization curve of each condition, and Ecorr, Icorr, Tafel slope of anodic and cathodic lines, and Epit were measured by polarization curves and are presented in Table 2.

### Table 2. The polarization test results and the sour corrosion rate of CN66 and CN71 with different heat treatment conditions (as-cast, AQ: solution treated and air-cooled, WQ: solution treated and water quenched), and its ferrite fraction.

| Alloy | Cooling Condition | Ecorr (mV) | Icorr (mA/cm²) | Epit (mV) | Ipit (mA/cm²) | Corrosion Rate (mpy) | Sour Corrosion Rate (mg cm⁻²·year⁻¹)/Area (cm²) | Ferrite Fraction (%) |
|-------|--------------------|------------|----------------|-----------|---------------|-----------------------|-----------------------------------------------|--------------------|
| CN66  | as-cast            | −368.4     | 3.95 × 10⁻²    | −159      | 1.57 × 10⁻²   | 1.80 × 10⁻¹            | 1.96/9.32                                     | 3.36               |
|       | AQ                 | −307.4     | 1.20 × 10⁻²    | −106      | 3.08 × 10⁻³   | 5.47 × 10⁻²            | 0.22/4.13                                    | 5.76               |
|       | WQ                 | −213.2     | 8.00 × 10⁻²    | −75       | 7.98 × 10⁻⁵   | 3.65 × 10⁻³            | 1.13/3.33                                    | 8.86               |
| CN71  | as-cast            | −294.7     | 5.82 × 10⁻⁴    | −114      | 7.41 × 10⁻²   | 2.65 × 10⁻²            | −                          | 1.31               |
|       | AQ                 | −233.3     | 3.82 × 10⁻⁴    | −96       | 6.31 × 10⁻⁵   | 1.74 × 10⁻²            | <0.01/5.04                                | 3.84               |
|       | WQ                 | −223.7     | 4.00 × 10⁻⁹    | −12       | 9.33 × 10⁻⁵   | 1.82 × 10⁻³            | 0.29/3.18                                    | 5.69               |
In particular, all corrosion rates of CN66 and CN71 were calculated by reflecting the factors derived from the potentiodynamic polarization test. By discerning the corrosion rates of Fe-Cr-Mn-C-N high interstitial austenitic alloys and a stainless steel 316L, a benchmark alloy, it concluded that the benchmark alloy showed the highest corrosion rate (1 mpy), compared with high interstitial stainless steels. Even as-cast CN71 that showed the highest corrosion rate, among high interstitial stainless steels, exhibited a lower corrosion rate (0.074 mpy) than the stainless steel 316L [19]. It can be deduced that Fe-Cr-Mn-C-N high interstitial austenitic alloys would have higher corrosion resistance than the benchmark alloy, a stainless steel 316L.

The values of $E_{\text{corr}}$ with solution-treated CN66 increased to $-307.4$ (air quenched) and $-213.2$ (water quenched) mV as compared to that of as-cast ($-368.4$ mV). This trend can also be seen in the case of CN71. The $E_{\text{corr}}$ of as-cast CN71 was $-294.7$, and increased to $-233.3$ mV (air quenched), and $-223.7$ mV (water quenched) in the solution-treated sample. In addition, solution treatment not only affects the $E_{\text{corr}}$, but fast cooling and water quenching also increased the $E_{\text{corr}}$ of CN66 and CN71 to $94.2$ mV (CN66) and $9.6$ mV (CN71), compared to that of the respective air quenched sample. The corrosion current density of the alloys was markedly reduced by both solution treatment and a faster cooling rate. As shown in Table 2, the value of $I_{\text{corr}}$ of CN66 in the solution treated and air quenched condition was decreased to $1.20 \times 10^{-7}$ mA/cm$^2$ from $3.95 \times 10^{-7}$ mA/cm$^2$ and that of the water quenched sample also decreased to $8 \times 10^{-9}$ mA/cm$^2$, respectively. The $I_{\text{corr}}$ values of CN71 showed consistently lower values when compared with the same conditions for CN66, while the $I_{\text{corr}}$ of CN71 with the solution treated and water quenched samples shows the lowest value in all of the conditions. All the results shown in Table 2 indicate that both the solution treatment and the higher cooling rate enhance the corrosion properties of CN66 and CN71, with a high $E_{\text{corr}}$ and low $I_{\text{corr}}$.

Regarding the enhancing ability of an alloying element in stainless cast steel, this study revealed that surface treatment with carburizing or implantation of carbon elevated $E_{\text{corr}}$ and lowered $I_{\text{corr}}$ results in the nobler matrix of stainless cast steels [17,20–22]. Many studies have also reported that the solution treatment and higher cooling rate enhanced the distribution and the solubility of the alloying elements in the matrix, such that the formation of undesired precipitates (carbides or nitrides) in the alloy is inhibited by faster cooling due to insufficient time to form, while carbon and nitrogen remain in the matrix of the alloys [14,18,23–25]. This study showed that the pitting corrosion resistance of CN66 and CN71 improved with the solution treatment and water quenching, which is consistent with previous studies [14,26]. The pitting potential of CN66 and CN71 in the solution-treated and water-quenched samples increased up to $-75$ and $-12$ mV, from $-159$ and $-114$ mV for those of the as-cast samples. The difference between air and water quenching was 31 and 84 mV, respectively.
Thus, the $E_{\text{pit}}$ increased with solution treatment and an increase in the cooling rate. The pitting current density of CN66 and CN71 in the solution-treated and water-quenched samples was decreased to $7.98 \times 10^{-5}$ and $9.33 \times 10^{-5}$ mA/cm$^2$ from $1.57 \times 10^{-2}$ mA/cm$^2$ and $7.41 \times 10^{-2}$ mA/cm$^2$ for those of the as-cast specimen respectively. The $I_{\text{pit}}$ was decreased with solution treatment and an increase in the cooling rate, and it is a consistent result with that of $E_{\text{pit}}$. Pitting corrosion resistance appears to correlate to an aspect of passivation, and it was also reported that the alloyed carbon and nitrogen made the passive film more protective by increasing both the chromium cation fraction ($[\text{Cr}] / ([\text{Cr} + \text{Fe}]$) and fine distribution in the passive film [11, 27].

Figure 2 showed the concentration of chromium in as-cast and heat-treated CN66 and CN71. Among the specimens, the water-quenched CN66 and CN77 exhibited a relatively higher concentration of chromium and fine distribution, rather than other conditioned alloys, on the matrix. In this, the chromium elements were distributed uniformly on the matrix. They might influence the trend of pitting potential, where the passivity of the alloy is increased by the high concentration of chromium.

![Figure 2. Compositional analyses of CN66 and CN71 with different heat treatment conditions (as-cast, solution treated and air-cooled (AQ), and solution treated and water quenched (WQ)), to determine their chromium content by electron probe micro-analysis (EPMA).](image)

The corrosion property of both CN66 and CN71 in sour environments was characterized by a corrosion immersion test in high pressure (total 0.8 MPa) and at a temperature of 393.15 K (120.15 °C), autoclave. The change in mass of the specimens was measured to assess the alloys’ degree of reaction to this highly aggressive environment, and is reported as sour corrosion rate (mg·cm$^{-2}$·year$^{-1}$) in Table 2.

A sour environment creates the harshest conditions for material to resist. The sour corrosion rate of as-cast CN66 was found to be very high, at 1.96, but that of CN71 could not be measured due to significant weight loss during the test. Even as-cast CN66 and CN71 could not completely resist in sour environments; however, those of the solution-treated samples that were cooled with water and in air showed significantly improved sour corrosion resistance. In Table 2, the sour corrosion rate of CN66 with solution treatment and air quenching decreased to 0.22 (mg·cm$^{-2}$·year$^{-1}$), and that of CN71 indicates the value smaller than 0.01 (mg·cm$^{-2}$·year$^{-1}$), indicating superior corrosion resistance.

In contrast with the corrosion properties from polarization tests, faster cooling rates, such as water quenching, decrease the sour corrosion resistance of CN66 and CN71. The sour corrosion rate of
water-quenched CN66 showed an increased value of 1.1 (mg·cm\(^{-2}\)·year\(^{-1}\)), as compared to that of air quenching (0.22 mg·cm\(^{-2}\)·year\(^{-1}\)). Likewise, the corrosion rate of water-quenched CN71 increased to 0.29 (mg·cm\(^{-2}\)·year\(^{-1}\)). The ferrite phase of water-quenched CN66 and CN71 increased to 3.1 and 1.9\% respectively, as compared with that of the air-quenched samples, as the water quenching after solution treatment inhibits the transformation of austenite from ferrite in these alloys. The ferrite fraction in CN66 is measured as 2.05 (as-cast), 1.92 (air quenched), and 3.17\% (water quenched) higher than that of CN71. It can be considered that a higher carbon and nitrogen content (0.08\%) affected the stability of austenite in HISSs.

In sour environments, hydrogen sulfide may speed up carbon dioxide corrosion due to cathodic reactions, and may cause corrosion, due to the formation of iron sulfide (FeS) and iron carbonate (FeCO\(_3\)) as main products of corrosion [28]. The oxidation state of the iron present in CN66 and CN71, under different cooling rate conditions, is thought to control FeS and FeCO\(_3\) formation, due to the presence of H\(_2\)S.

To compare the measurement of the bonding energy of CN66 and CN71 under different conditions, a simulation study of the ideal bonding energy of iron Fe\(^{2+}\) and Fe\(^{3+}\) was performed and is shown in Figure 3. According to the simulation, it could identify multiple Fe\(^{2+}\) splits that start from 708 eV and Fe\(^{3+}\) splits that start from 709 eV.

![Figure 3. The Fe\(_{2p}\) high-energy resolution spectra of (a) Fe\(^{2+}\) and (b) Fe\(^{3+}\) by charge transfer multiples for the X-ray absorption spectroscopy (CTM4XAS) program that was developed by E. Stavitski and F.M.F.de Groot [17,18].](image)

The bonding energy of the oxidation state of the iron present in CN66 with different cooling rates was observed experimentally with an X-ray photoelectron spectroscopy, as shown in Figure 4. As shown in Figure 4a, the bonding energy of iron in CN66 with air cooling consists of peaks at 709.2, 709.7, and 710.3. These peaks mean Fe\(^{3+}\), according to the results of the simulation (Figure 3b). The iron peaks in CN66 that was water quenched, were measured at 708.5, 709.7, 709.2, and 710.2, as shown in Figure 4b. Among the peaks, 708.5 showed the highest peak, and in this case, it indicates the Fe\(^{2+}\) bonding state, according to the findings in Figure 3a. Many researchers have reported that Fe\(^{2+}\) is an oxidation state in which H\(_2\)S reacts with iron and that this reaction is the fastest mechanism to produce FeS and FeCO\(_3\) as corrosion products [28,29]. The amount of Fe\(^{2+}\) has an immense influence on the formation and nature of the iron carbonate and iron sulfide scale, which could affect the corrosion rate of the given steel [28,30]. Thus, Fe\(^{2+}\) is considered as the main oxidation state of iron that tends to readily react with H\(_2\)S, as compared to Fe\(^{3+}\). From these results, fast cooling creates a greater amount of ferrite and the oxidation state of Fe\(^{2+}\) in present alloys. Furthermore, the increased amounts of ferrite and of the Fe\(^{2+}\) state affect the decreased corrosion resistance in sour environments [14].
4. Conclusions

The corrosion properties of Fe-Cr-Mn-C-N HISSs were investigated for down-hole applications in sour environments. The alloys contained 0.66% and 0.71% of total nitrogen and carbon, and the corrosion properties of the alloys with solution treatment and fast cooling showed high corrosion resistance in NaCl solution, including pitting corrosion. The chromium mapping indicates the highest levels of chromium and its fine distribution in fast-cooled CN66 and CN71 alloy. This finding controls the trend in pitting potential. The passivity of the alloy increases by chromium distribution.

Faster cooling rates decrease the sour corrosion resistance of CN66 and CN71. The oxidation state of iron present in CN66 and CN71 under different cooling rate conditions indicates the possibility of FeS formation, due to the presence of H2S as the main corrosion product. The XPS peaks in CN66, which was water quenched, showed the lowest and highest peak at 708.5, indicating Fe2+ bonding. Fe2+, as the main oxidation state of iron, reacts with H2S forming FeS leading to decreased corrosion resistance in sour environments.


diffracted line
Fe3+
 Accumulated line
Intensity (a.u.)
708 709 710 711 712
Energy (eV)

(b)

Figure 4. Background-subtracted Fe 2p3/2 spectra from CN66 with solution-treated and air cooling (a) and water quenching (b), observed by X-ray photoelectron spectroscope.

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