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Influence of sensitizing treatment on the corrosion resistance of Incoloy 028 alloy

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The influence of the precipitated phases on Incoloy 028 alloy was investigated *via* a sensitizing treatment at various sensitizing times and temperatures, weight loss and electrochemical testing methods were used, and the influence of the precipitated phases on the corrosion resistance on Incoloy 028 alloy was discussed on the basis of the significant characteristics of the polarization curves and Nyquist plots. The results showed that the corrosion resistance of Incoloy 028 was influenced by the precipitated phases; the corrosion rate of Incoloy 028 alloy initially increased and then decreased with the increasing sensitizing temperature; the most severe corrosion occurred at 900 ºC; and the elemental contents of Cr and Mo in the corrosion area were lower than those in the non-corroded area on the alloy surface, indicating that the corrosion resistance on the precipitated phase area was weakened.

Key words: Incoloy 028 alloy, Sensitizing treatment, Precipitated phases, Corrosion resistance, Potentiodynamic polarisation curve, Electrochemical impedance spectrometry.

INTRODUCTION

Ni-Fe-Cr alloys possess good resistance to a wide variety of corrosive environments in industrial processes such as chemical and petrochemical processing, marine engineering, oil/gas production and transport, and in nuclear reactors [1-3]. The demand for Ni-Fe-Cr alloys is increasing, and materials with excellent mechanical properties are required [4,5].

Incoloy 028 is a Ni-Fe-Cr alloy with addition of molybdenum, copper, and titanium, which has replaced many other alloys due to its superior local corrosion resistance. It is high-alloyed austenitic steel having excellent resistance to stress corrosion cracking and localized attacks such as pitting and crevice corrosion [5-9]. Many efforts have been made to understand the corrosion behavior [8-12].

Nickel-based alloys also are widely utilized in locations where severe corrosion attacks. However, the influence of precipitated phases on Incoloy 028 alloy by a sensitizing heat treatment has rarely been investigated in relation to CO_2 corrosion in the oil/gas industry. It is important to understand the corrosion resistance of Incoloy 028 alloy at various sensitizing temperatures by using weight loss and electrochemical tests.

EXPERIMENTAL

Specimen preparation

All specimens were made of the nickel-based alloy Incoloy 028 with original chemical composition of (wt.%): C 0.01, Si 0.41, Mn 1.06, P 0.022, S 0.002, N 0.056, Ni 30.3, Cr 27.6, Mo 3.5, Cu 0.95, and Fe balance. Specimen slices with a size of 30 × 15 × 1.5 mm were processed for corrosion immersion tests, while the electrochemical test specimens were φ 8 × 6 mm rods embedded in epoxy resin with an exposed working area of 0.685 cm^2. A new specimen was used for each experiment, and prior to the electrochemical tests, the working surfaces were wet-ground with SiC sand paper of up to 1200 grits, followed by ultrasonic cleaning in acetone and final rinsing in deionized water.

All specimens were of nickel-based alloy Incoloy 028 treated through a solid-solution process at 1180 ºC for 2 h with subsequent water-cooling. Then, some specimens were treated through sensitizing heat treatment at 700, 800, 900, and 1000 ºC for 2 h, with subsequent air-cooling. Other specimens were treated through sensitizing heat treatment at 900 ºC for 0.5, 2, and 5 h.

Weight loss corrosion test

The prepared specimens were weighed using an electronic balance with a precision of 0.1 mg, and then placed in a constant-temperature water bath. The solution was deoxygenated by bubbling pure N_2 for 4 h before the introduction of CO_2, and the CO_2 gas was bubbled to the water bath during the corrosion test in the range of 30 - 70 ºC. The corrosion tests were carried out for 15 days under static conditions. The test solution came from oilfield produced water, with main ionic concentrations of 12.16 g L^{-1} of Cl^-, 0.43 g L^{-1} of

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SO_4^{2-}, 0.27 g L^{-1} of HCO_3^-, 0.14 g L^{-1} of Ca^{2+}, and 8.16 g L^{-1} of K^+ and Na^+. Five samples were tested for each experimental condition. After each test, specimens were rinsed with distilled water and ethanol, and were then divided into two groups. The specimens in group one were descaled with Clark solution (20 g of Sb_2O_3 + 50 g of $SnCl_2$ + 1 L of 36.5 wt.% HCl), after which the weight loss was measured. The specimens in group two were not descaled, but dried and stored in a desiccator until the analysis with scanning electron microscopy (SEM) and X-ray diffraction (XRD) was performed.

Electrochemical corrosion test

The test solution was 3.5% NaCl, de-aerated by purging with nitrogen prior to the test. The electrochemical tests were conducted by polarization and EIS methods in a conventional three-electrode cell.

The polarization tests were applied to the working electrode at a scan rate of 0.2 mV s^{-1} from -0.3 mV to 0.3 mV *vs*. SCE with respect to the open-circuit potential. The EIS measurements were carried out at the open-circuit potential using 5 mV amplitude perturbation at a frequency range of 100 kHz - 10 mHz. Each experiment was performed at least twice at 20 ^{o}C. The ZView software was used to analyze the EIS data.

RESULTS AND DISCUSSION

Corrosion immersion test

Corrosion rate

Figure 1 shows the influence of temperature on the corrosion rate (*CR*) of Incoloy 028. The *CR* increased with the increase in temperature. The *CR* of the Incoloy 028 alloy initially increased, and then decreased with increasing sensitizing temperature. The most severe corrosion occurred in the specimen sensitized at 900 ^{o}C for 2 h.

In addition, under identical temperature conditions, the *CR* of the Incoloy 028 treated at 900 ^{o}C increased with the increase in sensitizing time from 0.5 to 5 h, as shown in Fig. 2. These results indicate that the number of precipitated phases in the specimens for various sensitizing times played a role in corrosion rate.

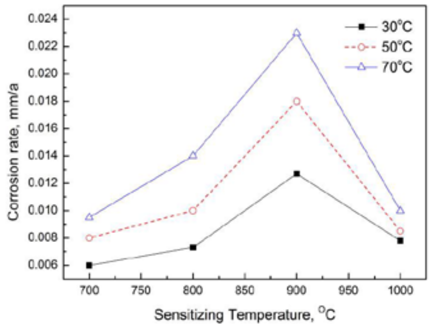

Fig. 1. Influence of temperature on corrosion rates of Incoloy 028 treated at various sensitizing temperatures for 2 h.

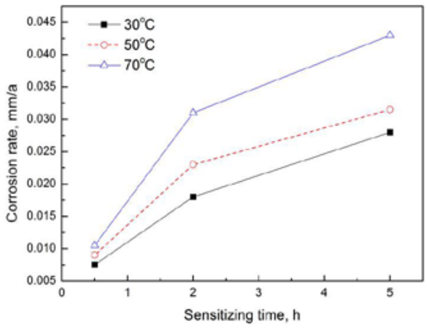

Fig. 2. Influence of temperature on corrosion rates of Incoloy 028 treated at 900 ^{o}C for various sensitizing times.

Corrosion characterisation

The surface morphology is shown in Fig. 3a. It can be seen that pitting corrosion occurred on the surface of Incoloy 028, which could be explained by a second-phase precipitation in the local surface area for the specimen sensitized at 900 ^{o}C leading to a diminishing of corrosion-resistance of Incoloy 028. Figs. 3b and 3c present the energy-dispersive spectroscopy (EDS) plots, the corrosion products included elements such as Fe, Cr, Ni, Mo, C, O, Si, and small amounts of Ca, Ba, and Cl. It was determined that the main phases in all corrosion products were $FeCO_3$ and small sediment compounds such as SiO_2 and $CaCO_3$. In contrast, the contents of both Cr and Mo in the corrosion area were lower than those on the alloy surface. This result indicates that the corrosion attack preferentially took place in the area of precipitated phases, i.e., the area containing low amounts of Cr and Mo.

XRD spectra shown in Fig. 4 further reveals that the peaks were γ-phase in the substrate, which is demonstrated by the presence of the corrosion product $FeCO_3$ due to the superior anti-corrosion performance of Incoloy 028.

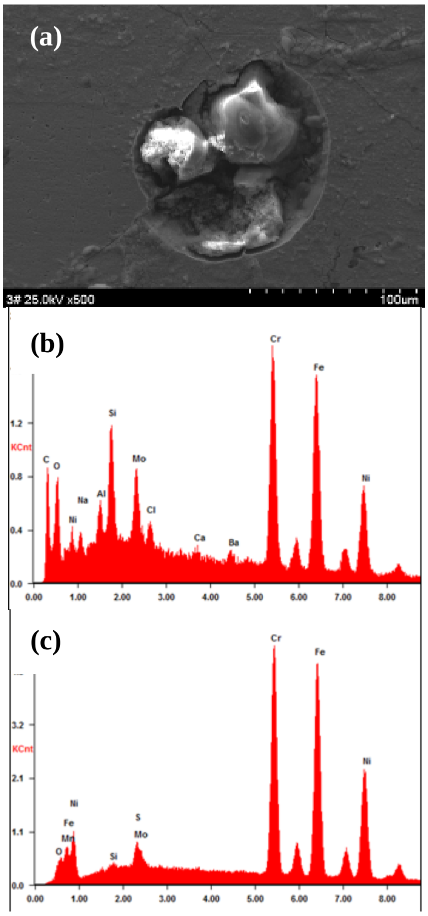

Fig. 3. Surface characterization of Incoloy 028 treated for 2 h at sensitizing temperature of 900 ºC after corrosion test at 70 ºC. (a) SEM; (b) EDS of corrosion area; (c) EDS of alloy surface.

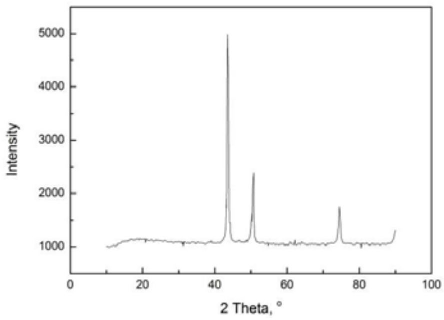

Fig. 4. XRD spectrum of the corrosion product on Incoloy 028 treated at 900 ºC for 2 h after corrosion test at 50 ºC.

Electrochemical test

Polarization

Figure 5 shows the Tafel polarization curves of Incoloy 028. The anodic branches of the polarization curves had similar characterization, which indicates that the anodic reaction maintains the same corrosion mechanism. It is evident that the corrosion potential initially moved in a more negative direction, and then moved in a positive direction, indicating that the self-corrosion tendency initially increased and then decreased with increasing sensitizing temperature. The corrosion current density of Incoloy 028 at 900 ºC was higher than those at 700, 800, and 1000 ºC under the same conditions. This result is in accordance with the result from the weight loss method mentioned above. The cathodic branch slopes (b_c) were close to the corresponding anodic branch slopes (b_a), so it can be proven that the corrosion process is controlled by the combined effects between anodic dissolution and cathodic diffusion.

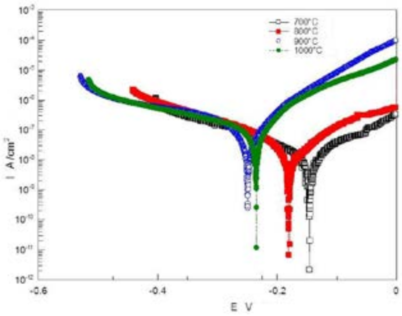

Fig. 5. Polarization curves of Incoloy 028 treated at various sensitizing temperatures in 3.5% NaCl solution at 20 ºC for 2 h.

Figure 6 shows the Tafel polarisation curves of Incoloy 028. The polarization curves moved in the positive direction, indicating that the thermodynamic effect accelerated the transfer rates of corrosive species and the corrosion reactions at temperatures ranging from 20 to 50 ºC. It is worth noting that the anodic branch slopes were similar, while the cathodic branch slopes increased with increasing temperature, which indicates that the entire corrosion process was predominantly controlled by cathodic reactions when the testing temperature increased.

Figure 7 shows the Nyquist plots of Incoloy 028 treated at various sensitizing temperatures in 3.5% NaCl solution at 20 ºC for 2 h. There was one depressed capacitive loop in the intermediate frequency region. The diameter of the capacitive loop decreased when the sensitizing temperature increased, and the minimum diameter was for the specimen sensitized at 900 ºC.

The corresponding equivalent circuit model is shown in Fig. 8. In this model, R_s is the solution resistance between the working electrode and reference electrode and R_t is the charge transfer resistance of the corrosion reaction. The CPE-n of Incoloy 028 is close to 1, which indicates the presence of an approximately ideal capacitor. CPE-Y_o represents the base admittance of the CPE and the changes in its values indicate that the adsorption layer and/or corrosion film demonstrate no significant difference. As expected, the value of R_t decreased with the increase of the sensitizing temperature, especially at 900 ºC, i.e., the corrosion resistance decreased. The results are in good accordance with that obtained from the Tafel polarization.

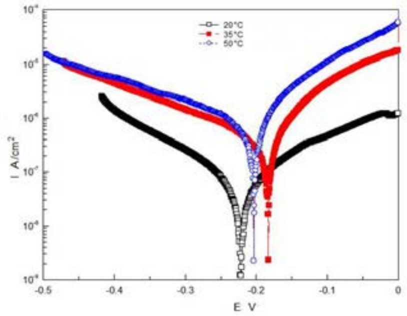

Fig. 6. Influence of temperature on poalrization curves of Incoloy 028 treated at sensitizing temperature of 800 ºC in 3.5% NaCl solution for 2 h.

EIS measurements

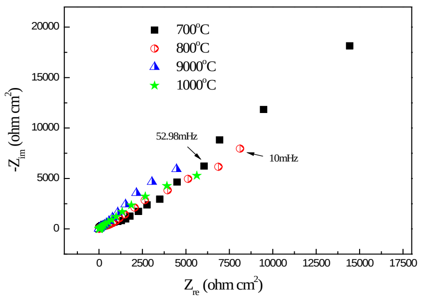

Fig. 7. Nyquist plots of Incoloy 028 treated at various sensitizing temperatures in 3.5% NaCl solution at 20 ºC for 2 h.

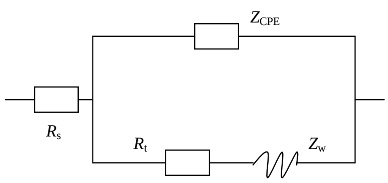

Fig. 8. The equivalent circuit model corresponding to the Nyquist plots of Incoloy 028 under various conditions.

CONCLUSION

The corrosion rate of Incoloy 028 firstly increased, then decreased with the increasing sensitizing temperature; the specimen sensitized at 900 ºC underwent the most severe corrosion. The corrosion processes were controlled by the combined effects between anodic dissolution and cathodic diffusion. The corrosion potential of Incoloy 028 shifted to more negative values before changing direction to positive values with the increasing sensitizing temperature, while the corrosion current density of the specimen sensitized at 900 ºC was higher than that sensitized at 700, 800, and 1000 ºC. And the minimum diameter of the capacitive loop was obtained from the specimen sensitized at 900 ºC, and the charge transfer resistance decreased with the increase in the sensitizing temperature, especially at 900 ºC.

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ВЛИЯНИЕ НА СЕНСИБИЛИЗИРАЩАТА ОБРАБОТКА ВЪРХУ КОРОЗИОННАТА УСТОЙЧИВОСТ НА СПЛАВТА Incoloy 028

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(Резюме)

Изследвано е влиянието на утаените фази върху сплавта Incoloy 028 чрез сенсибилизираща обработка при различна температура и време. Използвани са методи на загуба на тегло и електрохимични методи. Влиянието на утаените фази върху корозионната устойчивост на сплавта Incoloy 028 е дискутирано на основата на значимите характеристики на поляризационните криви и графиките на Nyquist. Резултатите показват, че корозионната устойчивост на сплавта Incoloy 028 се влияе от утаените фази, като скоростта на корозия първоначално нараства, след което намалява с повишаване на температурата на сенсибилизация и най-силна корозия се наблюдава при 900ºC. Съдържанието на Cr и Mo в областта на корозия е по-ниско от това в некородиралата област върху повърхността на сплавта, което сочи, че областта с утаени фази е отслабена.