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Article

Corrosion Behavior of Ni Steels in Aerated 3.5-wt.% NaCl Solution at 25°C by Potentiodynamic Method

Gan Pavapootanont[1], **Pornwasa Wongpanya**[2,a], **Ekkarut Viyanit**[3,b,*],
and Gobboon Lothongkum[1,c,*]

1 Department of Metallurgical Engineering, Faculty of Engineering, Chulalongkorn University, Pathumwan, Bangkok 10330, Thailand
2 School of Metallurgical Engineering, Suranaree University of Technology, 111 University Avenue, Nakhon Ratchasima 30000, Thailand
3 Failure Analysis and Corrosion Technology Laboratory, National Metal and Materials Technology Center, 114 Paholyothin Rd., Klong 1, Klong Luang, Pathumthani 12120, Thailand
E-mail: [a]pornwasa@sut.ac.th, [b]ekkarutv@mtec.or.th (Corresponding author), [c]Gobboon.L@chula.ac.th (Corresponding author)

Abstract. This work studied the corrosion behaviors of 15Ni, 23Ni, 31Ni and 40Ni steels in aerated 3.5% NaCl solutions with various pH levels (2, 7 and 10) at 25°C. The observed microstructures of 15 and 23 wt.% Ni steels were consisted partially of martensite, while those of 31 and 40 wt.% Ni steels were fully austenitic. Polarization curves of the nickel-containing steels were determined using a potentiodynamic polarization technique. The corrosion potential (Ecorr), corrosion current density (Icorr), corrosion rate (Rmpy), pitting potential (Ep), primary passive potential (Epp) and passive current density (Ip) were evaluated from the polarization curves. The results indicated that an increase in nickel content resulted in an increased corrosion potential, but also a decreased corrosion current density or corrosion rate. Only the solution pH of 10, the passive characteristics of the 23Ni, 31Ni and 40Ni steels were remarkable. As the nickel content of those three nickel-containing steels increased, the primary passive potentials and pitting potentials were increased, but the passive current densities were decreased. Nickel additions improved general corrosion and pitting corrosion resistances of the nickel-containing steels when tested in basic, neutral and acidic 3.5% NaCl solutions. The effects of various nickel contents and solution pHs on general corrosion resistances of nickel-containing steels were also discussed.

Keywords: Nickel-containing steel, corrosion behavior, potentiodynamic, 3.5 % NaCl solutions.

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1. Introduction

Although nickel based alloys and nickel-iron based alloys have been considered as an important class of material for engineering applications under severe corrosion in a wide range of service temperatures [1-5], their behaviors concerning general and localized corrosion resistances are still under discussion [1, 2, 6]. Resistance to stress corrosion cracking of stainless steel wire containing 18-20%Cr in boiling magnesium chloride solution was at a minimum when the nickel content was in a range between 8-10% [6]. Pilling and Ackerman [7] reported that corrosion rates of 15%Cr alloy with nickel contents up to 30% in 5% nitric acid increased with the nickel content, but decreased as the nickel content was greater than 30%. Streicher [8] found that corrosion rates of Fe-10%Ni-Cr alloys having chromium content greater than 16% were decreased to be less than those of Fe-Cr alloys when exposed in boiling 5% sulfuric acid. Considering the effects of nickel in AISI 430, 18Cr-15.5Mn-0.5Ni, 15Cr-17Mn, 1.0Ni, 17.6Cr-5.6Mn-4.4Ni and AISI 304 on corrosion rates, it was reported that nickel additions could decrease corrosion rates of those alloys in boiling solutions of 10% sulfuric acid (H_2SO_4), 10% sulfamic acid (H_3NSO_3) and 10% sodium bisulfate ($NaHSO_4$) [5]. Our previous work [9] revealed that pitting potentials of Fe-Cr-Ni austenitic alloys in artificial seawater reached to a maximum value at chromium and nickel contents of 26 and 16, respectively. This was explained by XPS results that showed that the passive film thickness of the chromium oxide and iron oxide layers containing iron and nickel directly influenced an increase in pitting potentials [10]. Abreu et al. [11] observed that upon increasing the nickel content up to 13% in 22Cr duplex stainless steel, the pitting potentials in 3.5% NaCl solution abruptly increased. The effects of nickel on pitting potentials of Fe-15Cr-(10-60)Ni steels in 0.75% NaCl solution [12] and of Fe-25Cr-3Mo-(0, 4, 30)Ni steels in 10% NaCl solution [13] were investigated. The results indicated that a synergistic effect of chromium and nickel on increased general corrosion and pitting corrosion resistances was possible in Fe-Cr-Ni alloys.

Due to the passive film break-down resulting in pitting corrosion, the characteristics of the passive film in nickel-containing alloys should be emphasized here. The passive film characteristics of nickel and nickel based alloys have been mostly investigated in basic solutions. The passive film of nickel and nickel alloys characterized by XPS, SIM [14-19], SER [20], STM [21], and Ellipsometry [22], were seen to be composed of NiO, NiO_2, Ni_3O_4 or $Ni(OH)_2$, depending on the potentials and the pH levels of tested solutions. It was stated that NiO and Ni_3O_4 films were formed at low potentials, and NiO_2 film was transformed to both NiO and Ni_3O_4 films at higher potentials [14]. In H_2SO_4 and H_3PO_4 acidic solutions, the hydrated nickel oxyhydroxide film was formed in nickel and alloy 600 [23]. According to a thermodynamic calculation based on the stable compounds, the passive film of nickel alloys in basic aqueous solution was identified as NiO_2 and Ni_2O_3 at higher potentials and Ni_3O_4 and $Ni(OH)_2$ at lower potentials [24]. The most acceptable passive film of nickel alloys is probably a double layer composed of NiO as an inner layer and $Ni(OH)_2$ as an outer layer [19]. The above reviews indicated that nickel is involved in the passive film formation and may enhance corrosion resistance of nickel-containing alloys in basic solution. The increased corrosion resistance of nickel-containing steels in basic solution is understandable by considering the standard electrode potential of nickel, which is higher than that of iron [6, 25]. Regarding the passive film formation in stainless steel, the effect of nickel is less than that of chromium in facilitating the film formation, because the chromium oxide is more stable than nickel oxide under ambient atmosphere conditions based on a thermodynamic point of view.

In order to gain a better insight into the effects of nickel additions on corrosion behaviors of nickel-containing steel, general and pitting corrosion resistances of Fe-(15, 23, 31, 40)Ni steels were investigated by a potentiodynamic polarization technique.

2. Experimental Procedure

2.1. Materials and Microstructure Examination

Chemical compositions of the nickel-containing steels used in the current investigation are listed in Table 1. All the nickel-containing steels were austenitized at 1,498 K for 3,800 seconds and then cooled down to room temperature in an argon atmosphere vacuum furnace. Before corrosion testing, microstructure features of the tested steels were observed using an optical microscope (OM). Additionally, structure phase identification was also conducted by X-ray diffraction (XRD) in order to obtain microstructural perspectives for further discussion.

2.2. Potentiodynamic Polarization

The samples of heat-treated nickel-containing steels were prepared and then mechanically wet ground with abrasive SiC paper up to 1200-grit finishing followed by ultrasonic cleaning in acetone. After that, the samples were kept in a desiccator for at least 24 hours prior to potentiodynamic polarization measurements. The schematic corrosion test set-up is illustrated in Fig. 1. The potentiodynamic polarization measurements were conducted in the aerated 3.5 % NaCl solutions with various pHs (2, 7 and 10) at a temperature of 25°C using a μ-Autolab potentiostat controlled by GPES software. The solution was purged by air for the aerated corrosion test condition for at least 1,800 seconds before the experiment. The potential was scanned from -650mV to +150mV, i.e. from cathodic to anodic direction, at a constant rate of 0.1 mV/s. A conventional three-electrode corrosion cell consisting of the sample, a silver/silver chloride (Ag/AgCl) reference electrode and a platinum counter electrode was used in the current investigation. The total area of samples exposed to the testing solution was confined to 4.0 cm^2. In order to confirm the experimental results, the potentiodynamic polarization measurements were repeated 3 times for each test condition. From the polarization curves, the corrosion potential, corrosion current density, primary passive potential, pitting potential and average passive current density were determined as described in our previous work [26]. The corrosion potential and corrosion current density were evaluated by the Tafel-slope extrapolation method. The corrosion rate was calculated from the corrosion current density based on Faraday's law. The pitting potential was determined at the potential where the current density introduced by corrosion attack was rapidly increased. The average passive current density was determined from the mathematic mean value of those in the whole passive range. The morphology of pits present on the surface of tested steel after corrosion testing was also observed using an optical microscope.

Table 1. The chemical compositions (wt.%) of the tested nickel-containing steels.

Steel	Chemical composition								
Name	C	Si	Mn	P	S	Ni	Cr	Al	Fe
Fe-15Ni	0.017	0.006	0.003	<0.003	0.0019	14.99	<0.2	0.015	bal.
Fe-23Ni	<0.01	<0.005	<0.01	0.002	0.003	23.00	<0.2	0.015	bal.
Fe-31Ni	0.005	0.005	0.01	0.002	0.008	30.80	<0.2	0.015	bal.
Fe-40Ni	0.01	0.008	<0.01	0.004	0.015	39.99	<0.2	0.023	bal.ance

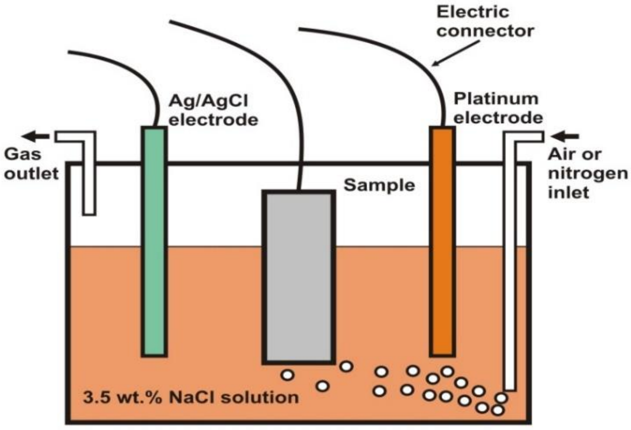
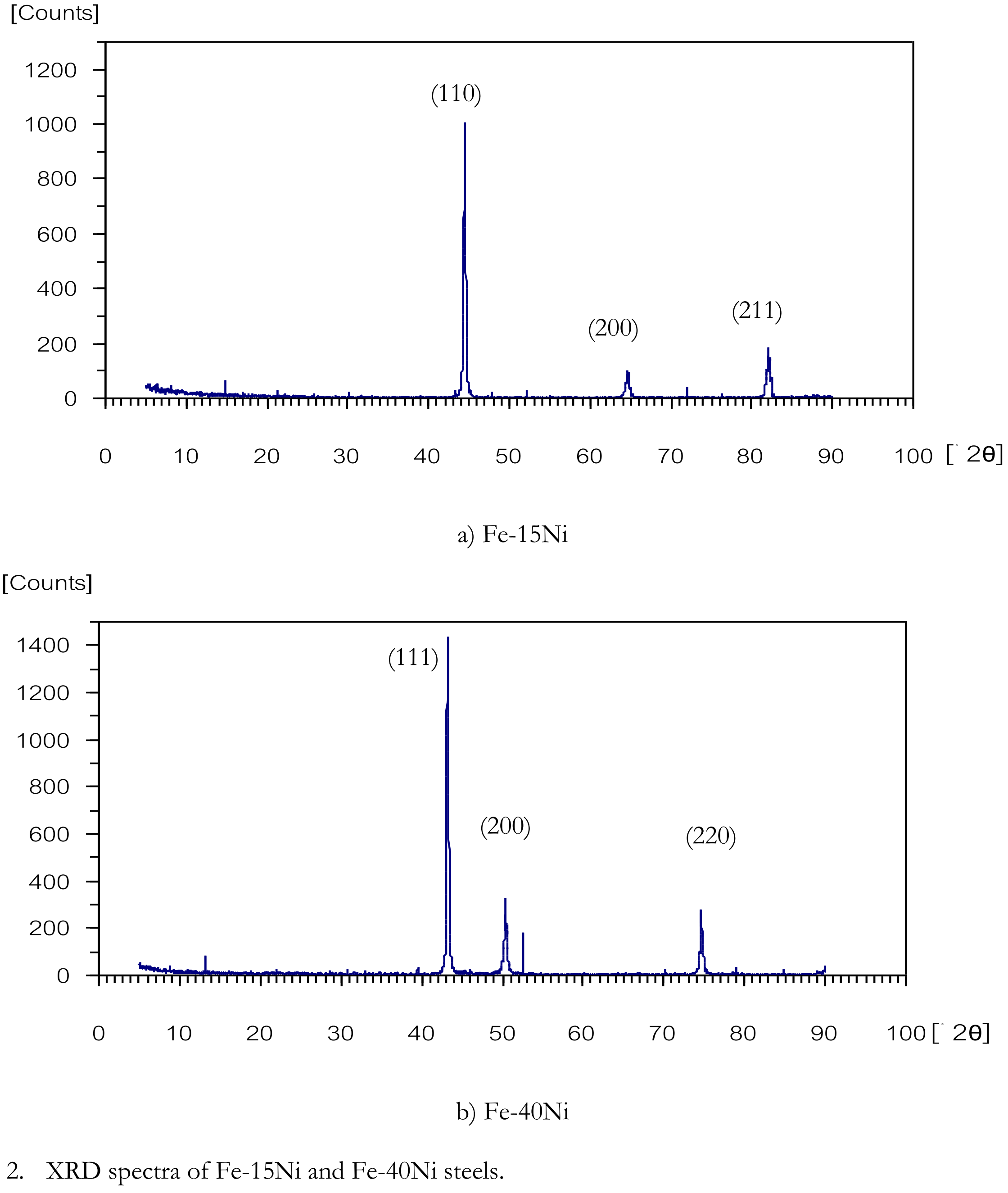


Fig. 1. Schematic illustration of corrosion cell set-up for the experiment.

3. Results and Discussion

3.1. Microstructure

Examples of XRD spectra obtained for the Fe-15Ni and Fe-40Ni steels are shown in Fig. 2. It indicates that Fe-15Ni and Fe-40Ni steels possessed the body-centered-tetragonal (bct) and face-centered-cubic (fcc) crystal structures, respectively. The XRD results of crystal structure determination conformed to the observed microstructures by optical microscopy (see Fig. 7). It is worth noting that the microstructures of Fe-15Ni and Fe-23Ni steels were partially martensitic, while those of Fe-31Ni and Fe-40Ni steels were fully austenitic. These results should be attributed to the martensite start temperature (Ms-temperature) which was decreased as the nickel content of the steel increased. The austenite phase tended to be more stable when a higher content of nickel was added into the steels [27]. However, the austenite-martensite partial transformation could be predominant for the steels containing a relatively low content of nickel during cooling from austenization temperature.



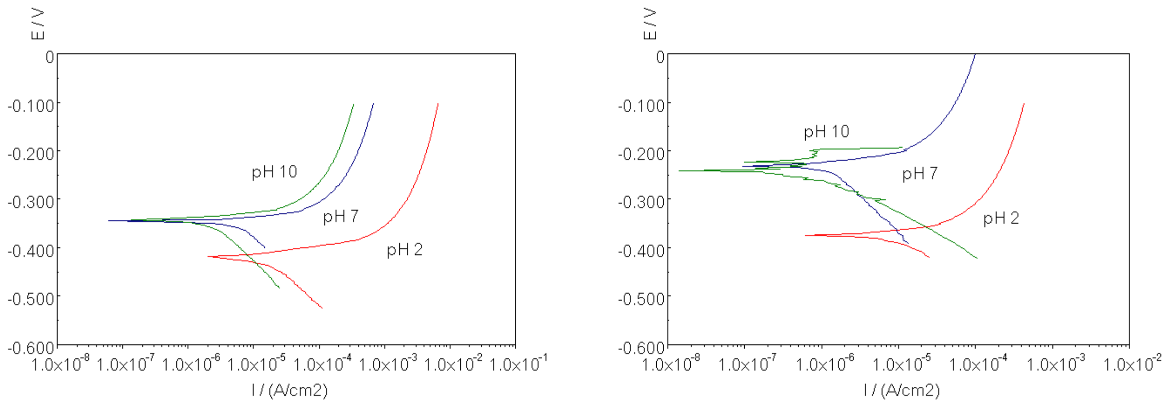
a) Fe-15Ni

b) Fe-40Ni

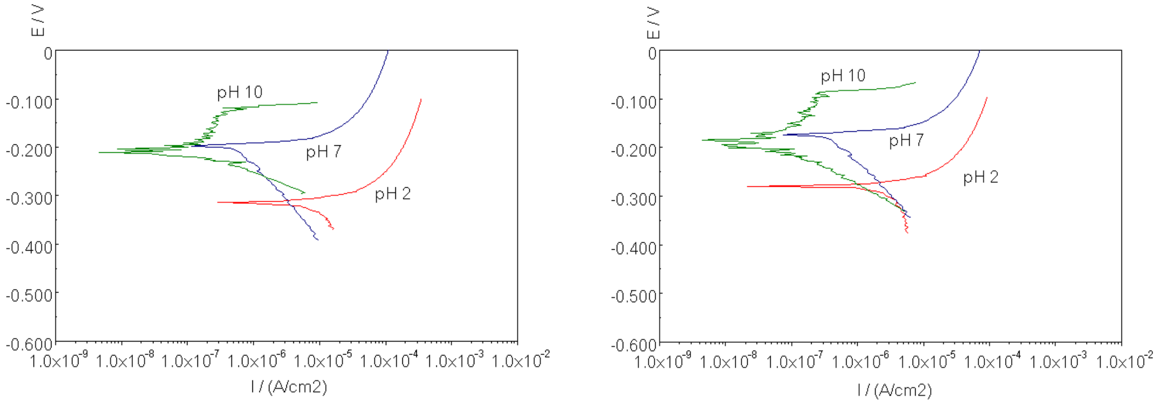
Fig. 2. XRD spectra of Fe-15Ni and Fe-40Ni steels.

3.2. Effect of Nickel on Corrosion Potential and Corrosion Rate

Figure 3 shows examples of polarization curves obtained for the steels tested in aerated 3.5 % NaCl solution with various pHs at 25°C. The Fe-15Ni steel did not reveal passive behaviors when tested in the solution at any pH level. The passivity behavior was noticeable for Fe-23Ni, Fe-31Ni and Fe-40Ni steels only when the solution pH of 10 was used. In order to gain a better insight into the corrosion behavior of the nickel-containing steels, the important corrosion behavior parameters, including Ecorr, Icorr, Epp, Ep and Ip, extracted from the polarization curves were mathematically evaluated and summarized in Tables 2 and 3.



a) Fe-15Ni b) Fe-23Ni



c) Fe-31Ni d) Fe-40Ni

Fig. 3. Examples of polarization curves of the nickel-containing steels tested in aerated 3.5 % NaCl solution at 25°C (E/V vs Ag/AgCl).

Table 2. Mean values of parameters (± standard deviation) evaluated from polarization curves of the nickel-containing steels tested in aerated 3.5% NaCl solution at pH 2, 7 and 25°C.

Steels	pH 2			pH 7		
	Ecorr	Icorr	Rmpy	Ecorr	Icorr	Rmpy
Fe-15Ni	-427±8	12.42±1.25	143.3	-343±2	3.81±0.17	43.9
Fe-23Ni	-385±8	9.28±0.46	107.1	-235±3	0.97±0.04	11.1
Fe-31Ni	-319±4	5.67±0.92	65.4	-204±6	0.58±0.05	6.7
Fe-40Ni	-278±4	1.87±0.08	21.6	-177±2	0.18±0.02	2.0

Table 3. Mean values of parameters (± standard deviation) evaluated from polarization curves of the nickel-containing steels tested in aerated 3.5% NaCl solution at pH 10 and 25°C.

Steels	pH 10					
	Ecorr	Icorr	Rmpy	Ip	Epp	Ep
Fe-15Ni	-346±1	1.33±0.04	15.4	-	-	-
Fe-23Ni	-246±4	0.28±0.03	3.3	0.43±0.08	-242±3	-191±8
Fe-31Ni	-205±3	0.09±0.01	1.1	0.12±0.01	-205±4	-124±5
Fe-40Ni	-179±3	0.05±0.01	0.6	0.11±0.01	-176±3	-86±3

Remark:		
	Ecorr	Corrosion potential (mV vs. Ag/AgCl)
	Icorr	Corrosion current density (μA/sq.cm)
	Rmpy	Corrosion rate (μm/y)
	Ip	Passive current density ((μA/sq.cm)
	Epp	Primary passive potential (mV vs. Ag/AgCl)
	Ep	Pitting potential (mV vs. Ag/AgCl)

Figure 4 shows the corrosion potentials with respect to the nickel contents in the nickel-containing steels tested in aerated 3.5 % NaCl solutions. At all solution pHs, the corrosion potentials of the nickel-containing steels were increased as the nickel content increased. This may be due to the higher standard electrode potential of nickel compared to iron [6, 25]. This implies that the nickel additions could bring up general corrosion resistance of the nickel-containing steels when immersed in acid, neutral and basic solutions. However, a great reduction of the corrosion potentials was observed when solution pH of 2 was used. The corrosion potentials of Fe-23Ni steel at the solution pHs of 10 and 7 were about -240 and -250 mV, respectively, but that at solution pH of 2 it was about -370 mV. No significant difference in the corrosion potentials was observed at solution pHs of 7 and 10 for all the nickel-containing steels. Based on Pourbaix diagrams composed from testing in water at solution pHs of 7 to 10, iron revealed passive behavior due to the formation of $FeOH_2$ film at a specific potential range between -200 and -600 mV, while a potential range between 0 and -580 mV could result in formation of $NiOH_2$ film in nickel (Ni) [24]. Due to the fact that corrosion potentials of all the nickel-containing steels tested in the solution at solution pHs of 7 and 10 were between -170 and -350 mV, the nickel-containing steels used in the current investigation could perform passivity at pH 7 and 10, yielding no significant difference in the corrosion potentials. For the nickel-containing steels, the solution pH of 10 could help the passive film become more stable with respect to the solution pH of 7, resulting in lower corrosion rates as seen in Fig. 5.

Regarding the corrosion rate calculation results (see Fig. 5), it clearly shows that the corrosion rates of the nickel-containing steels were decreased as the nickel content increased. The increased nickel content resulted in an abrupt reduction of corrosion rates when tested in the solution pH of 2, especially. This finding was supported by Potgieter et al. [28] who found that uniform corrosion resistance of 22Cr series duplex stainless steels was improved by adding the higher nickel contents when tested in 1 M HCl solution. As expected, the corrosion rates of nickel-containing steels were depressed as solution pHs increased. But, it should be noted that the increased nickel contents could not strongly affect a decrease in the corrosion rates of nickel-containing steels when tested in the solution pHs of 7 and 10. On the other hand, the increased nickel contents were predominant for a drastic reduction of the corrosion rates in the nickel-containing steels exposed in the solution pH of 2. This was due to more noble behaviors of nickel in neutral to strongly alkaline solutions, not in acidic solutions [29]. From both Figs. 4 and 5, it indicates that the nickel-containing steels performed the higher corrosion potentials, but the lower corrosion rates as the nickel content increased. It can be concluded that the nickel content could play a key role in improving general corrosion resistance of the nickel-containing steels.

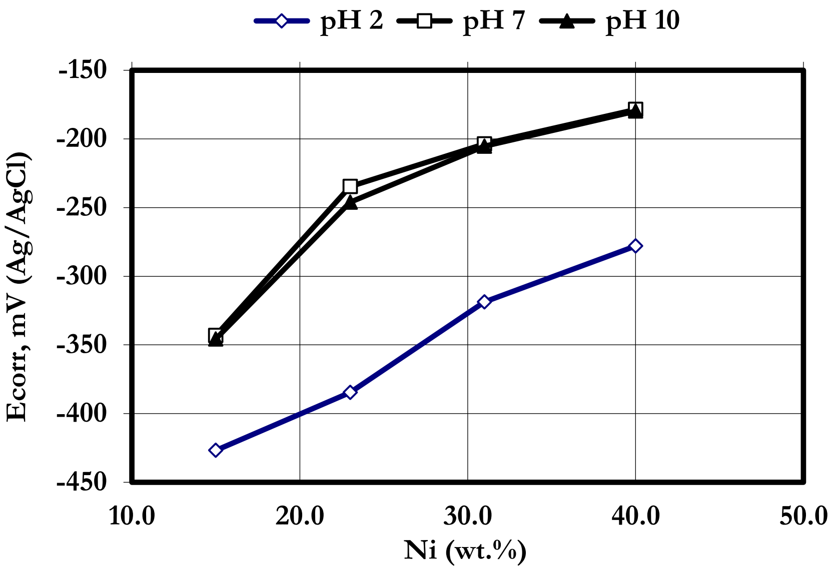


Fig. 4. Corrosion potentials of the nickel-containing steels tested in aerated 3.5 % NaCl solution at 25°C.

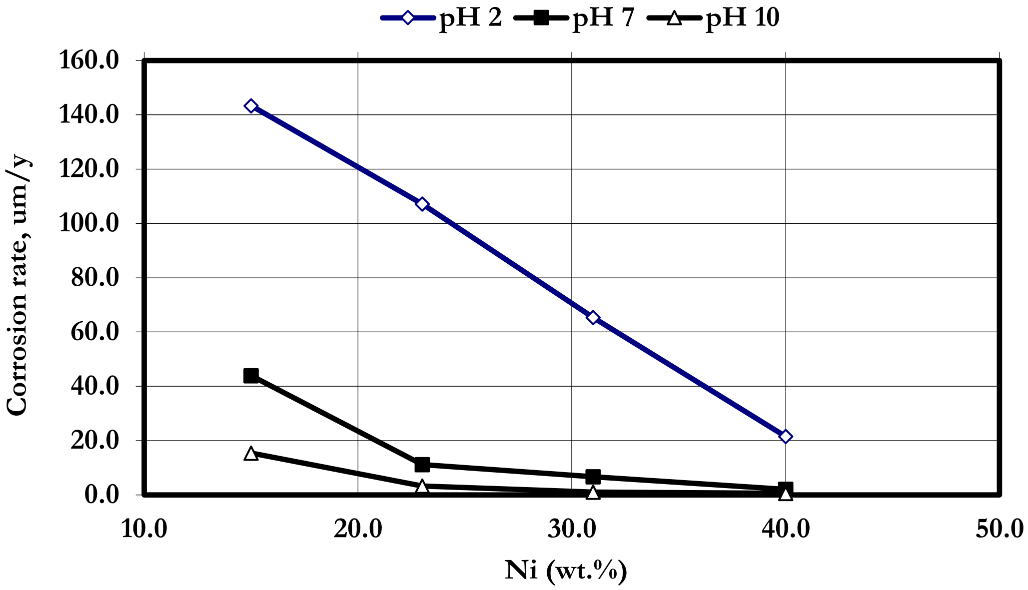


Fig. 5. Corrosion rates of the nickel-containing steels tested in aerated 3.5 % NaCl solution at 25°C.

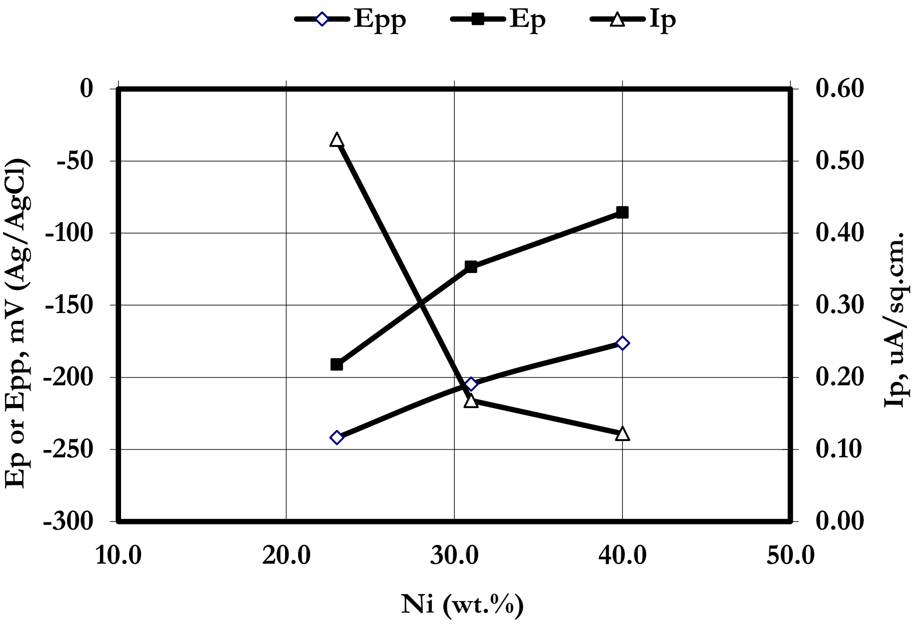


Fig. 6. Pitting potential, primary passive potential and passive current density of the nickel-containing steels tested in aerated 3.5 % NaCl solution at 25°C and pH 10.

3.3. Effect of Nickel on Pitting Potential, Primary Passive Potential and Average Passive Current Density

From the polarization curves as shown in Fig. 3, the nickel-containing steels did not show any passive behaviors when tested in the solution pHs of 2 and 7. However, the solution pH of 10 resulted in passive film formation in the Fe-23Ni, Fe-31Ni, Fe-40Ni steels. This may be due to the presence of nickel in the steel could promote either the formation of NiO_2, Ni_2O_3, Ni_3O_4 and $Ni(OH)_2$ passive films in basic aqueous solution as observed in Pourbaix diagram [24] or the formation of a duplex structured passive film composed of NiO as inner layer and $Ni(OH)_2$ as outer layer [30]. From a viewpoint of mechanochemistry [31], the local stress induced by martensite transformation may shift the 15 % Ni steel into the active state, i.e. no passive film formation. In contrast, this influence might be negligible for the 23 % Ni steel which still exhibited passive behaviors, even though its microstructure partially contained martensite (see Fig. 7a and b). This should refer to the effects of chemical composition on passivity rather than microstructure features.

Figure 6 shows the effects of nickel content on pitting potentials, primary passive potentials and passive current densities of the nickel-containing steels tested in aerated 3.5 wt.% NaCl solution at 25°C and pH 10. The pitting potentials were increased as the nickel content increased. This observation revealed a similar trend as observed in the corrosion potentials as shown in Fig. 4. Regarding passive film stability, it clearly showed that the increased nickel contents lowered the average passive current densities (see Fig. 6). These results suggested that the passive film stability of the nickel-containing steels was improved by the addition of higher nickel contents and agreed with explanation addressed in many previous research reports [14-22]. It confirmed that nickel alloys or nickel-containing steels could possess corrosion resistance in basic solution due to the complex passive layers composed of NiO as the inner and $Ni(OH)_2$ as the outer layer [30].

From the literature reviews and the result of this study, it should be summarized that the nickel-containing steels can exhibit passive behaviors when their nickel contents reached the critical level required for passive film formation during exposure in suitable environment. Similar trends were also seen in the previous work [10]. The pitting potentials of Fe-Cr-Ni alloys in artificial sea water (pH = 8) increased drastically when the Cr and Ni contents were sufficient for stabilizing their passive films. Moreover, the improvement of pitting corrosion resistance was achieved by increased thickness of passive films that consisted of chromium oxide and iron-nickel oxide layers [10]. This agreed with the experimental results obtained from 22Cr- 5, 7 ,9 and 13 Ni duplex stainless steels in 3.5 wt.% NaCl solution [11], from Fe-15Cr-(10-60)Ni steels in 0.75 wt.% NaCl solution [12] and from Fe-25Cr-3Mo-(0,4,30)Ni steels in 10 wt.% NaCl solution [13]. However, further experiments are still needed in order to specify appropriate nickel contents which can enhance pitting corrosion resistance of the nickel-containing steels exposed to any specific environment.

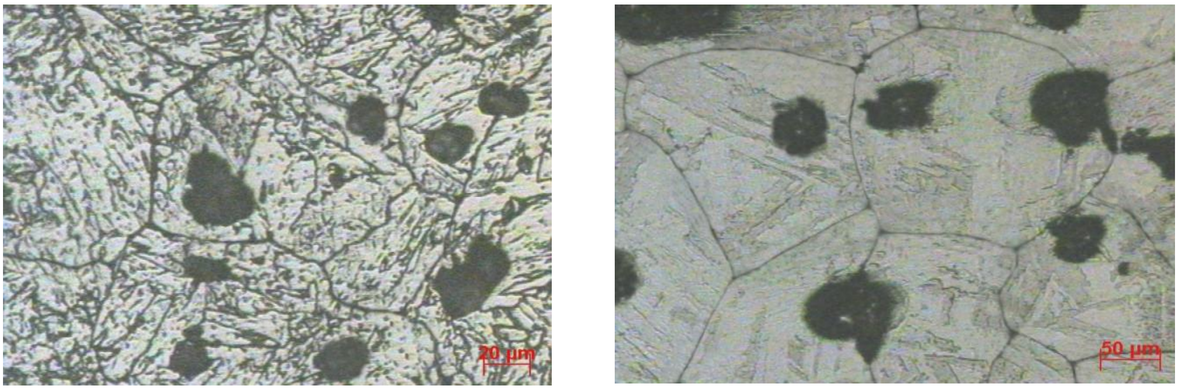
The effects of nickel on primary passive potential should be also addressed. Increasing the nickel content of nickel steel enhanced primary passive film potential in 3.5 wt. %NaCl solution at pH 10 as shown in Fig. 6. This suggested that the higher nickel steel needs higher energy to shift to the passive state from the active state than the lower nickel steel. In the passive state, however, the nickel-containing steel with increased nickel content exhibited the reduction of passive current densities (Fig. 6). In the Pourbaix diagram [24], nickel was shifted from the immune state to the passive state at a higher potential (about -0.5 V (SHE)) than iron (about -0.8 V (SHE)) when tested in water at pH 10. This implied that the passive state of nickel required more energy than iron. The nickel-containing steels could require more energy than steel to be in passive state. Therefore, an increase in nickel content of the steels could bring up the primary passive potential. This reflected in this work.

Regarding the chromium addition to Fe-Cr-(8-10)Ni stainless steels tested in 1M H_2SO_4 at 90°C, it was found that increasing chromium content from 3.5 to 19 wt. % resulted in a reduction of primary passive potentials as well as passive current densities [32]. The experimental study conducted by Jae-Bong Lee [33] also indicated a similar trend when testing Fe-Cr alloys in 1N H_2SO_4solution. This is due to the fact that chromium is one of the metallic elements that can easily react with oxygen to form passive oxide film.

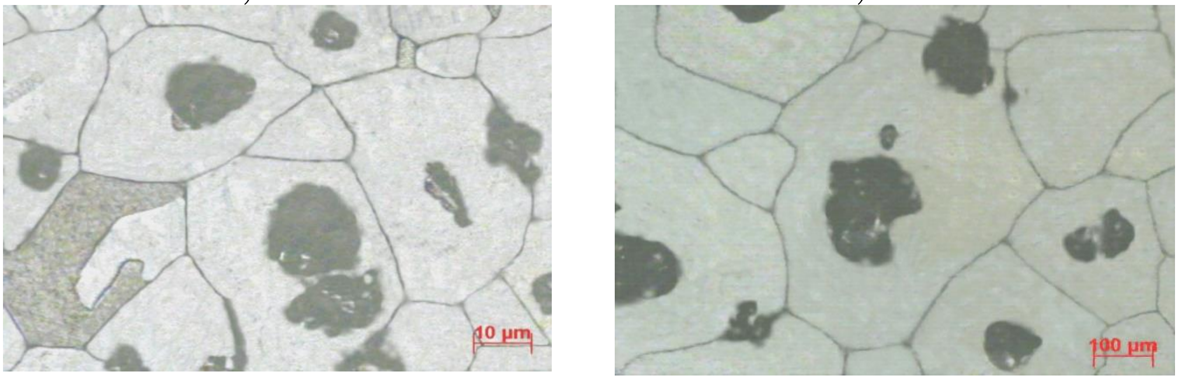
In addition to uniform corrosion behaviors, resistance of the nickel-containing steels to pitting corrosion is very crucial and should be discussed in detail. As aforementioned, the nickel-containing steels could exhibit the passive behavior when exposed in the alkaline solutions. Thus, the important passive film parameters of the nickel-containing steels exposed in the solution pH of 10 were shown in Fig. 6. It revealed that the increased nickel content gradually improved the passive behavior, showing an increase in both primary passive and pitting potentials and a decrease in passive current densities. As reported in the previous literatures [10, 28, 29], the superior pitting corrosion resistance could be related to the formation of more stable passive films enriched with nickel. In case of 22Cr duplex stainless steels containing the nickel content ranging between approximately 5 and 13 wt.%, their passive films were composed of nickel chromium oxide, nickel (II) oxide, nickel (IV) oxide, chromium (II) oxide, and chromium (VI) oxide [28]. For nickels tested in the strong alkaline solutions, the formation of β-$Ni(OH)_2$ in passive film was observed [29]. However, further passive film characterization in the current investigation should be conducted in order to obtain clarification in passivation behaviors of the nickel-containing steels.

3.4. Pit Initiation.

Figure 7 shows the morphology of pits observed on Fe-15Ni, Fe-23Ni, Fe-31Ni and Fe-40Ni steels, which were in transpassive state (no passive film), after testing in aerated 3.5 wt.% NaCl solutions at pH 10. Based on microstructure perspectives, it can be seen that partial austenite-martensite transformation was obtained for Fe-15Ni and Fe-23Ni steels. In contrast, the microstructure of Fe-31Ni and Fe-40Ni steels was fully austenitic. It implied that the degree of austenite phase stabilization in the nickel-containing steels was strongly influenced by nickel additions. From pits observation, it suggested that pits could be initiated not only at the grain boundaries but also within the grains. As the nickel-containing steels were in the transpassive state, pitting corrosion attack was only pronounced. The results from Figs. 4 – 6 indicated that the chemical compositions played an important role in controlling general and pitting corrosion resistance behaviors of the nickel-containing steels more than the microstructure features present in the materials. However, the microstructure features were normally recognized as a crucial factor to influence corrosion resistance behaviors of nickel base superalloy, because of its possible influence on corrosion resistance of passive film by changing film composition [34]. Some examples of stainless steel microstructures that were susceptible to pit initiation were summarized in the previous literature [35]. It revealed that the grain boundary, phase-transformed boundary and any defect points behaved as preferential sites for pits to be initiated.



a) Fe-15Ni b) Fe-23Ni



c) Fe-31Ni d) Fe-40Ni

Fig. 7. Locations of pits formed in the Fe-15Ni, Fe-23Ni, Fe-31Ni and Fe-40Ni steels tested in aerated 3.5 % NaCl solution at 25°C and pH 10.

4. Conclusions

The effects of nickel addition on general and pitting corrosion behaviors of Fe-15Ni, Fe-23Ni, Fe-31Ni and Fe-40Ni steels in aerated 3.5 % NaCl solution at pHs of 2, 7, 10 and temperature of 25°C were investigated. Some significant conclusions drawn from the current study are as followed:

- Fe-15Ni steel did not show passive behaviors at all the tested solution pHs.
- Nickel enhanced general corrosion resistance of the nickel-containing steels by increasing the corrosion potentials and decreasing the corrosion rates.
- Primary passive potentials of the nickel-containing steels were increased as the nickel contents increased.
- Nickel additions could play a key role in the improvement of pitting corrosion resistance of the nickel-containing steels, when the nickel contents of at least 23 % was added. The results were obtained from the tests in the solution pH of 10 only.
- Both the grain interior and boundary were recognized as the susceptible sites for pits to be initiated in the nickel-containing steels.

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