



Corrosion Performance of Stainless Steel and Nickel Alloys in Aqueous Sodium Hydroxide as Revealed from Cyclic Voltammetry and Potentiodynamic Anodic Polarization

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ABSTRACT

The electrochemical behavior of nickel, Inconel 600, Incoloy 800 and 316 stainless steel electrodes in different concentration of NaOH solution was investigated using the cyclic voltammetry technique (CV). All the curves in the anodic branch of the CV are characterized by anodic dissolution peak (A), passive region (B), anodic dissolution peak (C) before oxygen evolution. In the case of the cathodic branch of CV there is one reduction peak (D) in case of Ni electrode but there are two reduction peaks (D and E) in case of the three alloys used. All these peaks are explicated. The effect of chloride ion as a pitting agent on the cyclic voltammetry and the potentiodynamic anodic polarization of Ni, Inconel 600, Incoloy 800 and 316 SS electrodes in NaOH solution was investigated. As the concentrations of the chloride ions increase the change of the amount of the anodic charge Δq_a increases and the values of the pitting corrosion potential are shifted to the more negative direction indicating the breakdown of passivity and the imitation of the pitting corrosion. At one and the same Cl⁻ ion concentration, the resistance to the pitting attack decreases in the following order: 316SS > Inconel 600 > Incoloy 800 > 316 SS. This order depends on the chemical composition of these alloys.

Keywords: Nickel, Inconel 600, Incoloy 800, 316 SS, Cyclic voltammetry, Pitting corrosion.

INTRODUCTION

Nickel alloys and stainless steels (SS) are used extensively in several industrial applications where heat resistance and/or corrosion resistance is required. Nickel is one of the materials most resistant to a variety of caustic solutions (sodium hydroxide), as well as to molten caustic. Various nickel alloys and stainless steel also have been

widely used in sodium hydroxide production and in many other industries using caustic applications¹.

The cyclic voltammetry technique is useful for identifying the steps involved in the overall reactions resulting in the formation of various films on the metallic surfaces². It is particularly useful for distinguishing between products resulting from chemical, electrochemical reactions. Its main

limitation is that, it is only useful for examining very thin films formed in a relatively short time.

The characteristic and theory of pitting corrosion has been discussed in details by Kolotykin⁹ who stated that, the pitting corrosion of a certain metal or alloy occurs within a definite potential range lying in the passive region. The formation and development of this type of attack on the metal surface can occur only in solutions containing the aggressive Cl⁻, Br⁻ and I⁻ ions⁴⁻¹². The critical potential at which pitting initiates is reported by several authors and found to depend on a number of factors including the type and concentration of attacking anion, type of metal used and relative concentration of other anions¹³⁻¹⁴.

In the previous work¹⁵ the corrosion behavior of nickel, nickel alloys and stainless steel (SS) in various concentrations of HNO₃ solutions using the cyclic voltammetry technique was studied. Also the pitting corrosion of these samples in chloride containing solutions and its inhibition by some inorganic compounds using potentiodynamic anodic polarization was also studied¹⁶. To complement this work, the cyclic voltammetry of Ni, Inconel 600, Incoloy 800 and 316 SS electrodes were traced in NaOH solution as a function of NaOH

concentration. Also, the effect of chloride ion as a pitting corrosion agent on the cyclic voltammetry and the potentiodynamic anodic polarization of Ni, Inconel 600, Incoloy 800 and 316 SS electrodes in NaOH solution was further examined.

EXPERIMENTAL

The electrodes used in this study are nickel Inconel 600, Incoloy 800 and 316 SS. The chemical structures of these electrodes are given in Table 1. A cylindrical rod embedded in Araldite with an exposed surface area to the corrosive medium of 1.70 cm² for Ni, 1.57 cm² for the Inconel 600, 1.48 cm² for the Incoloy 800 and 1.44 cm² for the 316 SS was used. The electrolytic cell, electrode samples and surface treatment of the electrodes are the same as those described in previous work^{15,16}.

For the cyclic voltammetry and the potentiodynamic anodic polarization techniques were carried out using a Wenking potentiostat, Type POS-73. The current density-potential curves were recorded on X-Y recorder, Type PL-3. Before carrying out any experiment, the working electrode was precathodized (for 20 min.) at a potential of -1.5V in order to reduce the pre-immersion oxide film formed at the surface of the electrode.

Table.1: The chemical composition of the alloys used

Alloys	Ni%	Fe%	Cr%	Cu%	Si%	P%	S%	Mn%	C%	Others%
Nickel	100	-	-	-	-	-	-	-	-	-
Incone 100	73.42	9.33	16.10	0.03	0.118	0.007	0.006	0.38	0.04	Al 0.28, Ti 0.24, Co 0.049
Incoloy 800	33.49	44.95	19.32	0.27	0.32	-	0.007	0.81	0.063	Al 0.39, Ti 0.38
316 SS	13.12	65.80	16.50	-	0.50	0.03	0.002	1.63	0.028	Mo 2.35

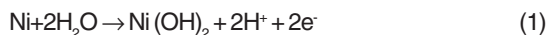
RESULTS AND DISCUSSION

Effect of NaOH concentration

The curves of Figs. 1-4 show the effect of various concentrations of NaOH solutions on the cyclic voltammetry (CV) characteristics of Ni, Inconel 600, Incoloy 800 and 316 SS, respectively, upon the polarization range between -1V to +1V at a scanning rate of 25 mV sec⁻¹ versus the saturated calomel electrode (SCE).

Inspection of the curves of Figs. (1-4) reveals that there is anodic dissolution peak (peak A). As the concentration of NaOH increases, the dissolution current density for peak (A) is increased due to the increase of corrosion rate with increasing the alkali concentration. It is clear from the curves of Figs. (1 and 2) for nickel and Inconel 600 (higher nickel content), that peak (A) appears at nearly the same value of potential (at » - 0.7 V). This peak is thought to correspond to a single electrochemical

step, which may be the oxidation of Ni to Ni(OH)₂ according to the reaction:



The equilibrium potential of this reaction according to Pourbaix¹⁷ was found to be:

$$E_p = 0.11 - 0.0591 \text{ pH} \quad (2)$$

Giving a redox potential of -0.7V (SCE) in 0.1 M NaOH solution, is in a good agreement with the experimentally reported values for peak (A).

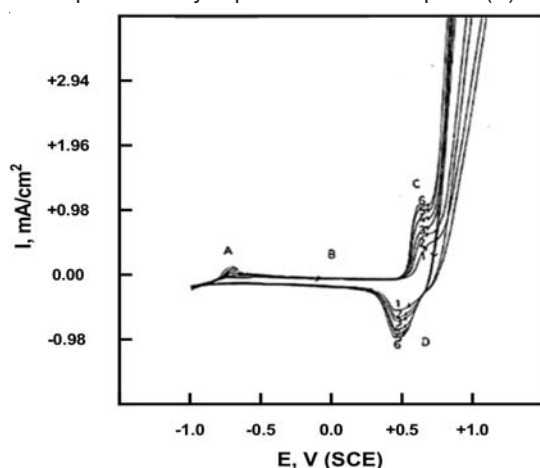
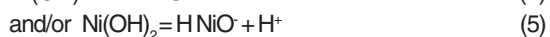


Fig.1. Cyclic voltammety of Ni in different concentrations of NaOH solution at 25 mV/sec.
(1) 0.05 M NaOH (2) 0.10 M NaOH (3) 0.15 M NaOH
(4) 0.20 M NaOH (5) 0.25 M NaOH (6) 0.30 M NaOH

However, there is a sufficient evidence concerning the formation of Ni(OH)₂ in alkaline solutions¹⁸ in the potential range corresponding to the anodic current of peak (A). From consideration of the charge amounts under peak (A), several investigators^{19,20} gave good evidence for the formation of only a mono-layer of Ni(OH)₂ on the electrode surface. It has been reported by Schrebler *et al.*,²¹ that Ni(OH)₂ appears as hydrated species on the metal surface, once its solubility product is exceeded. Furthermore, in aqueous solutions Ni(OH)₂ is bound to a well known equilibrium in solution



The latter species are particularly important in alkaline solutions since it entails the anodic dissolution of nickel as a complex anion.

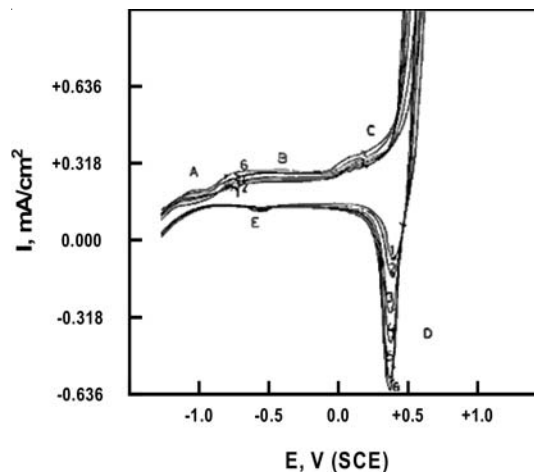
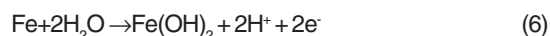


Fig. 2. Cyclic voltammety of Inconel 600 in different concentrations of NaOH solution at 25 mV/sec.
(1) 0.05 M NaOH (2) 0.10 M NaOH (3) 0.15 M NaOH (4) 0.20 M NaOH (5) 0.25 M NaOH (6) 0.30 M NaOH

For Incoloy 800 which contains 33.49% Ni and 44.95% Fe, peak (A) may correspond to the oxidation of Ni and Fe to Ni(OH)₂ and Fe(OH)₂ respectively. In case of 316 SS (of high iron content) this peak appears at $\approx -0.6\text{V}$ (S.C.E.), it may correspond to the formation of Fe(OH)₂ according to the reaction :



After peak (A), there is a passive region (region B). The current flowing along this region is commonly identified as the corrosion current which is used to counteract the chemical dissolution of the passive film. The alkali concentration has no significant effect on the current flowing along region (B) of the CV in the case of nickel as shown in Fig. 1, The current remains constant along a wide potential range amounting to about 1V due to the stability of the passivating oxide film. Along this potential region, the passivation current is totally used in repairing the oxide film attacked chemically by the alkali.

Davis and Barker²² suggested that, the quantity of electricity passed through this region is used in the formation of Ni(OH)₂, which fills in any pores existing in the already formed film, the remainder causes a certain amount of the film growth. In case of Ni and Inconel 600, this region may involve the formation of NiO, either by an irreversible chemical reaction:



or electrochemical reaction :



X-rays photoelectron spectroscopy (XPS)²³ indicates that the film formed on the Ni electrode surface is probably composed of NiO and Ni(OH)₂, the former being the passivating species. The present results are mostly consistent with the spectroscopic conclusions and with the results of Schreiber *et al.*,²¹.

The major constituent of the passive film formed on the alloys containing certain amounts of chromium is hydrated chromium oxyhydroxide CrO_x(OH)_{3-2x}·nH₂O²⁴, where n dependent upon the composition and quality of the underlying alloy and the condition of film formation.

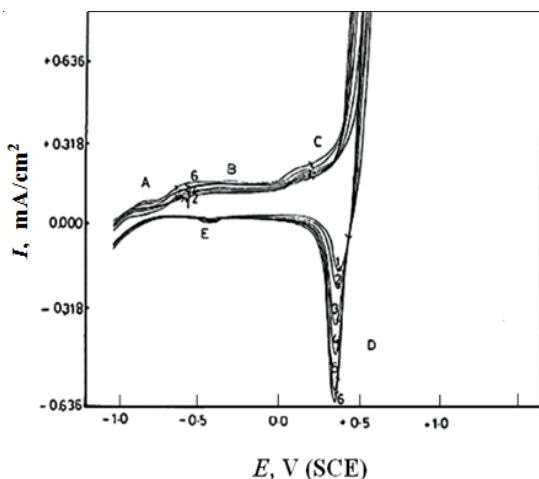
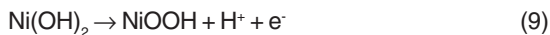


Fig.3. Cyclic voltammetry of Incoloy 800 in different concentrations of NaOH solution at 25 mV/sec. (1) 0.05 M NaOH (2) 0.10 M NaOH (3) 0.15 M NaOH (4) 0.20M NaOH (5) 0.25 M NaOH (6) 0.30 M NaOH

As the potential of the electrode becomes more positive, another anodic dissolution peak (C) has been observed before the transpassive region which followed by oxygen evolution. This peak may be attributed to the oxidation Ni(OH)₂ or NiO formed at the peak (A) and passive region (B) to some higher oxides of nickel. The oxidation of Ni(OH)₂ to a higher nickel oxide²⁵. There-fore, in case of Ni and Inconel 600, this peak can also be related to the oxidation of Ni(OH)₂ to NiOOH according to :



Hashimoto and Asami²⁴ reported that the passive film formed on Inconel 600 consists of

Ni(OH)₂·nH₂O which transforms with further increase in the potential to NiO(OH)_{2-2y}·nH₂O. In case of Incoloy 800 this peak may be due to the oxidation of Fe(OH)₂ and Ni(OH)₂ to FeOOH and NiOOH, whereas for 316 SS, peak (C) is related to the oxidation of Fe(OH)₂ to Fe 00H according to:

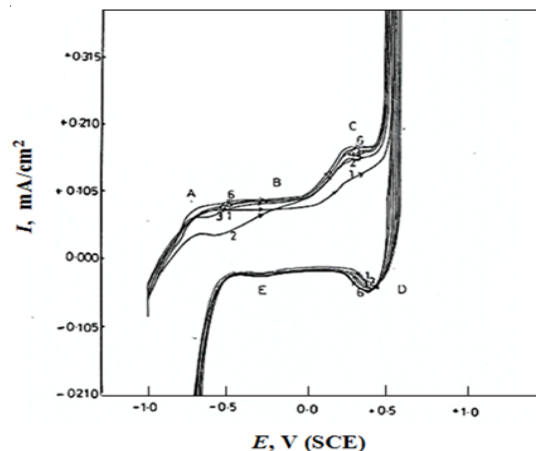
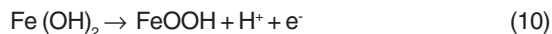


Fig. 4. Cyclic voltammograms of 316 SS in different concentration of NaOH at 25 mV/sec.

(1) 0.05 M NaOH (2) 0.10 M NaOH (3) 0.15M NaOH (4) 0.20M NaOH (5) 0.25 M NaOH (6) 0.30M NaOH



When the potential of working electrode is reversed in the cathodic direction after oxygen evolution, there is one cathodic peak (D) appears in the case of nickel as shown in Fig.1. An additional cathodic peak (E) is observed in case of Inconel600, Incoloy800,316 SS (Figs. 2-4).

In case of nickel the cathodic branch of the CV's exhibits no further reduction peaks before the evolution of hydrogen is attained. Thus, some of the reduction products of peak (D) probably nickel oxide or oxyhydroxide remains on the electrode surface down to the hydrogen evolution potential. For Inconel 600, Incoloy 800 and 316 SS, this peak can be related to the reduction of the oxyhydroxide formed on the anodic branch of the scan. Peak (E) was related to the reduction of the hydroxide remains in the passive region.

Susceptibility of nickel alloys and stainless steel to pitting corrosion by chloride ions Cyclic voltammetry measurements

The pitting corrosion of nickel alloys and stainless steel, similar to other metals or alloys,

occurs when the passivity breakdown of local points (anodic sites) on the surface exposed to the corrosive medium, while the major part remains passive. In practice, the pitting may be encountered if the corrosive environments contain chloride, or other aggressive halide ions²⁶⁻²⁸.

The curves of Figs.(5-8) show the cyclic voltammetry of of Ni, Inconel 600, Incoloy 800 and 316 SS respectively, in 0.1 M NaOH solution in the absence and presence of increasing concentrations of NaCl as a pitting corrosion agent at a scanning rate 25 mV/sec.

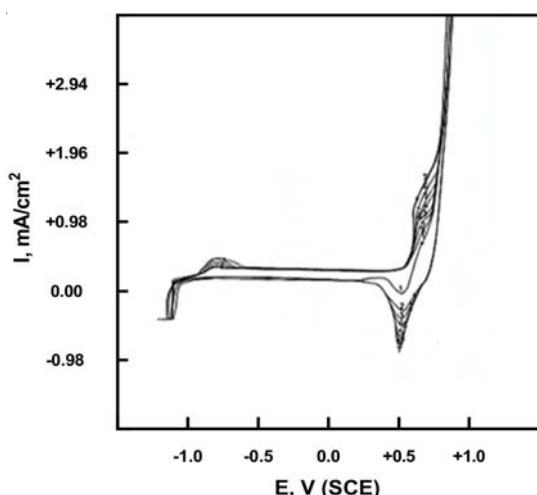


Fig. 5. Cyclic voltammetry of Ni in 0.1 M NaOH containing different concentration of NaCl solution at 25 mV/sec. (1) 0.00 M NaCl (2) 1×10^{-2} M NaCl (3) 3×10^{-2} M NaCl (4) 5×10^{-2} M NaCl (5) 7×10^{-2} M NaCl (6) 1×10^{-1} M NaCl (7) 1.5×10^{-1} M NaCl

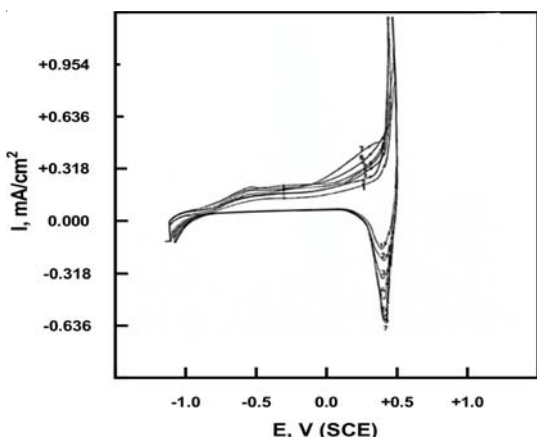


Fig. 6. Cyclic voltammetry of Inconel 600 in 0.1M NaOH containing different concentration of NaCl solution (at 25 mV/sec.). (1) 0.00 M NaCl (2) 1×10^{-2} M NaCl (3) 3×10^{-2} M NaCl (4) 5×10^{-2} M NaCl (5) 7×10^{-2} M NaCl (6) 1×10^{-1} M NaCl (7) 1.5×10^{-1} M NaCl

Inspection of the curves of these Fig shows that, the presence of NaCl up to a certain concentration, which depend on the electrolyte bulk concentration and the type of the electrode, is tolerated by the surface film and has practically no effect on the shape of the CVs reported in chloride-free solution. These halide concentrations are assumed to have no influence on the dissolution kinetics of the passive film on the electrode surface. However, higher concentrations of Cl⁻ ions on the other hand, show a tendency to destroy the passive film formed on the electrode surface. The effect of Cl⁻ ions on the passive film formed on the electrode can be recognized by the marked increase of the flowing currents at potentials, in the passive region, which are more active the higher the concentration of the Cl⁻ ions. This increase in current could be attributed to the destruction of the passive film with the initiation of localized corrosion²⁹⁻³⁰.

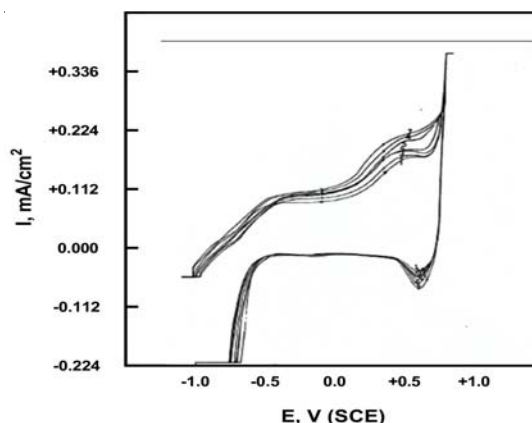


Fig.7. Cyclic voltammetry of Incoloy 800 in 0.1M NaOH containing different concentration of NaCl solution at 25 mV/sec. (1) 0.00 M NaCl (2) 1×10^{-2} M NaCl (3) 3×10^{-2} M NaCl (4) 5×10^{-2} M NaCl (5) 7×10^{-2} M NaCl (6) 1×10^{-1} M NaCl (7) 1.5×10^{-1} M NaCl

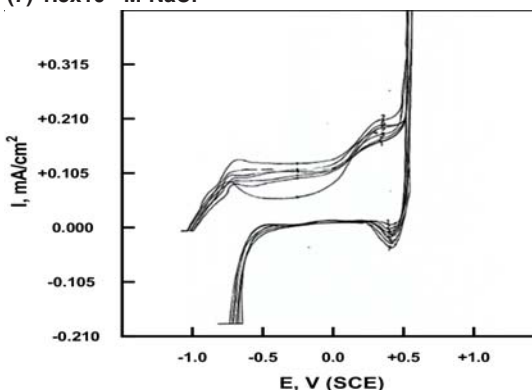


Fig. 8. Cyclic voltammetry of 316 SS in 0.1M NaOH containing different concentration of NaCl solution (at 25 mV/sec.). (1) 0.00 M NaCl (2) 1×10^{-2} M NaCl (3) 3×10^{-2} M NaCl (4) 5×10^{-2} M NaCl (5) 7×10^{-2} M NaCl (6) 1×10^{-1} M NaCl (7) 1.5×10^{-1} M NaCl

An interesting feature is observed when the potential of the working electrode is reversed in the cathodic direction. The current starts to decrease gradually with decreasing the electrode potential. Finally the current reaches a zero value, then a large dissolution current peak (areas) formed on the cathodic branch of the CVS. These peaks are enlarged with in-creasing the Cl⁻ ion content of the solution.

The change of the amount of the anodic charge Dq_a (coulomb. cm⁻²), which is the difference between the anodic charge amounts integrated in the presence and absence of Cl⁻ ion, is taken as a measure of the extent of the pitting corrosion to take place³⁰. Fig. 9 represents the variation of q_a with the logarithm of the molar concentration of Cl⁻ ion for Ni, Inconel 600, Incoloy 800 and 316 SS. Dq_a changes only slightly in the presence of low Cl⁻ ion concentrations. This may be attributed to repassivation of the formed pits, while at higher concentrations of the Cl⁻ ion, Dq_a changes markedly and linearly with $\log C_{Cl^-}$ according to:

$$Dq_a = a_1 + b_1 \log C_{Cl^-} \quad (11)$$

Where, a_1 and b_1 are constants which depend on the solu-tion composition and the scanning rate. Under such conditions of Cl⁻ ion concentrations the formed pits may continuously propagate.

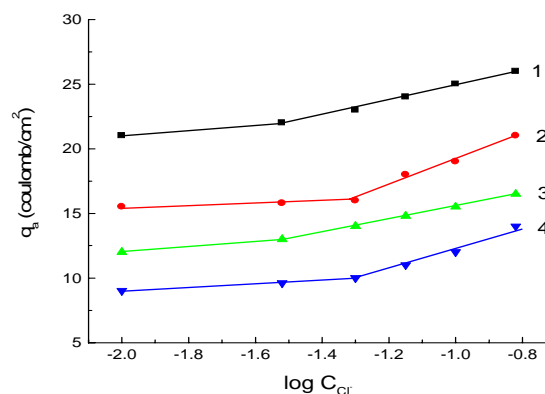


Fig. 9. The relation between the amount of anodic charge (Dq_a) and the logarithm of the molar concentration of Cl⁻ ions.(1) Ni (2) Inconel 600 (3) Incoloy 800 (4) 316 SS

Potentiodynamic anodic polarization

The curves of Fig.10 represent the potentiodynamic anodic polarization behavior of Ni in 0.1M NaOH solution at a sweep rate of 1.0 mV/

sec upon the addition of increasing concentrations of NaCl as a pitting corrosion agent. Similar curves were obtained for the other alloys namely, Inconel 600, Incoloy 800 and 316 SS. Inspection of the curves of Fig. 10 reveals that the following aspects:

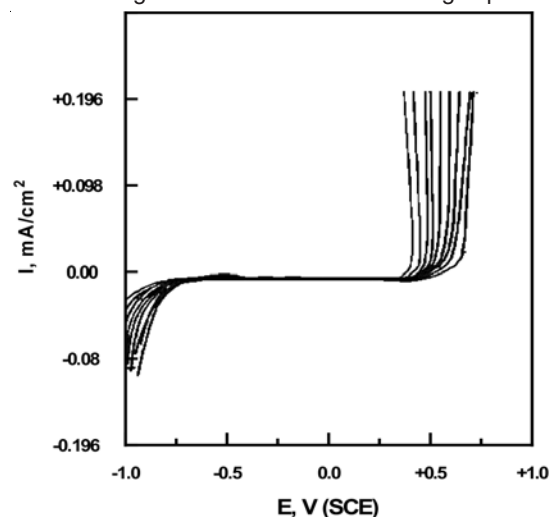


Fig. 10. Potentiodynamic anodic polarization curves of Ni in 0.1 M NaOH + different concentrations of NaCl at 1 mV/sec.

(1) 0.00 M NaCl (2) 5×10^{-3} M NaCl (3) 1×10^{-2} M NaCl (4) 3×10^{-2} M NaCl (5) 5×10^{-2} M NaCl (6) 7×10^{-2} M NaCl (7) 9×10^{-2} M NaCl (8) 1×10^{-1} M NaCl (9) 3×10^{-1} M NaCl

The addition of Cl⁻ ion up to a certain concentration which depends on the OH⁻ ion concentration has practically no effect on the dissolution of the passive film on the metal surface. However, at a certain critical concentration which depends on the alkali concentration, the nature of the metal or alloy, its thermal treatment and the state of its surface. The current flowing along the passive range increases suddenly and markedly at some definite potentials denoting the destruction of the passivating oxide film and the initiation of visible pits. This potential is known the pitting corrosion potential (E_{pitt})³⁰⁻³¹.

The effect of increasing Cl⁻ ion content of the solution is to shift the pitting potential of the working electrode into active negative direction. Similar findings were reported by Abdallah *et al.*,³⁰ in the case of pitting of pitting corrosion of nickel alloys and stainless steel in chloride solutions

The dependence of pitting corrosion potential of the Ni electrode, Inconel 600, Incoloy 800 and 316 SS on the concentrations of the Cl⁻ ion is shown in Fig. 11 which represents the plot of E_{pitt}

versus $\log C_{Cl^-}$ in 0.1M NaOH solutions. Usually most of the investigations carried out on the pitting corrosion potentials reveals a straight-line relationship between $E_{pitting}$ and $\log C_{Cl^-}$ satisfying the following equation

$$E_{pitting} = a_2 - b_2 \log C_{Cl^-} \quad (12)$$

Where a_1 and b_1 are constants which depend on both the nature and the type of the aggressive anion and the electrode sample.

In case of Incoloy 800 and 316 SS straight line relationship are obtained which fit the relationship according to equation (12). In case of nickel and Inconel 600, the curves are sigmoid in nature in which equation (12) is obeyed only within a certain range of Cl^- ion concentration. At lower of Cl^- ion concentrations the pitting potential shift slightly into negative direction as the concentration of the chloride ions is increased. Therefore, one can conclude that these concentrations of Cl^- ions are not sufficient to destroy completely the passivating film on the metal surface, or that pits formed are not completely active and may undergo repassivation³².

The pits formed within the concentration range of Cl^- ion, where an equation (12) is applicable are assumed to be of the limiting active type which cannot undergo repassivation. However,

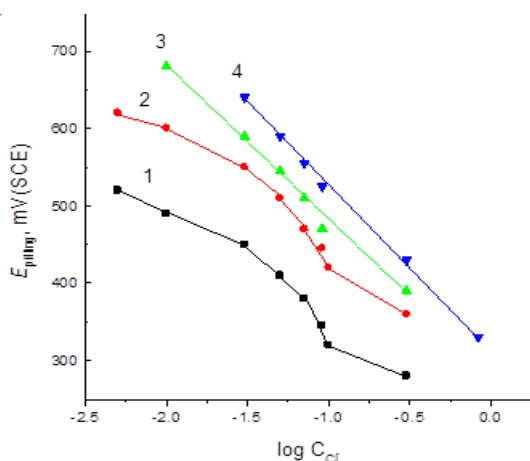


Fig. 11. The relationship between pitting potential and logarithm of molarity of Cl^- ions for nickel and the three alloy samples. (1) Ni (2) Inconel 600 (3) Incoloy 800 (4) 316 SS.

at higher concentrations of Cl^- ions, The pits formed continuously propagated.

Inspection of Fig.11, it is obvious that, at the same concentration of Cl^- ion the noble (positive) shift in the potential decreases in the following order: 316 SS > Incoloy 800 > Inconel 600 > Ni. This sequence is like the previous work in the case of the effect of Cl^- ion on the potentiodynamic anodic polarization curves of nickel alloys and 316 SS in HNO_3 solution¹⁶. It is obvious that the 316 SS is more resistance to pitting attack due to the presence of Mo and Cr in the chemical composition of 316 SS which improve the thickening of the passive film and consequently increase the resistance of this alloy to pitting corrosion. On the other hand, the Incoloy 800 is more resistance than Inconel 600 due to the higher amount of Cr content in its chemical composition.

CONCLUSIONS

1. The cyclic voltammetry technique was constructed of nickel, Inconel 600, Incoloy 800 and 316 SS electrodes in different concentrations of NaOH solutions.
2. All the curves in the anodic scan characterized by anodic peak(A), passive region (B), anodic dissolution peak(C) before oxygen evolution.
3. In the cathodic scan there is one reduction peak (D) in case of Ni electrode but there are two reduction peaks (D and E) in case of the alloys used.
4. As the concentrations of Cl^- ions increase the change of the amount of the anodic charge Dq_a increases and the values of the pitting corrosion potential are shifted to the more negative direction.
5. At one and the same Cl^- ion concentration, the resistance to the pitting corrosion decreases in the following order: 316 SS > Inconel 600 > Incoloy 800 > 316 SS.
6. The resistance of the alloys used to pitting corrosion depends on the chemical composition of its.

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