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Pitting Corrosion of Nickel Alloys and Stainless Steel in Chloride Solutions and its Inhibition Using Some Inorganic Compounds

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Abstract

The effect of increasing the concentration of chloride ions as pitting corrosion agent on the potentiodynamic anodic polarization curves of nickel(Ni), Inconel 600, Incoloy 800 and 316 Stainless steel (SS) in 0.1M HNO_3 solution at a scan rate 1.0 mV sec^{-1} was studied. The pitting corrosion potential varied with the logarithmic of the molar concentrations of chloride ions in case of Ni and Inconel 600 according to S-shaped curve. This behavior was explained according to re-passivation, propagation and continued propagation of the pits formed. In case of Incoloy 800 and 316SS a broken lines were observed according to re-passivation and propagation of the pits formed. It is found that,316SS is more resistance to pitting attack due to the presence of Mo in their chemical structure. The effect of the increasing concentration of some inorganic anions such as the sodium salts of chromate, tungstate, molybdate, phosphate dibasic and carbonate to inhibit the pitting corrosion was investigated. The values of the pitting corrosion potential were shifted to more positive values indicating the inhibiting action of these anions toward the pitting corrosion.

1. Introduction

The inhibition of pitting corrosion of nickel alloys and stainless steel is the subject of many researchers due to its wide applications in industry [1,2]. The high resistance of these alloys in a large variety of aqueous solutions due to the formation of the stable passive film on their surface [3]. When these alloys are exposed to aggressive solutions such as chloride ions. The passive layer is breakdown and causing pitting corrosion which is the one of the most dangerous form of localized corrosion. [4-7]

Pitting corrosion of nickel alloys and stainless steel, as other metals and alloys occurs when passivity breakdown takes place at local points on the surface exposed to the corrosive environments at which anodic dissolution proceeds whilst the major part of the surface remains passive. It is necessary to protect the metals or alloys from the pitting attack by adding the corrosion inhibitors. Most of the inhibitors are inorganic or organic compounds containing hetro atoms in their chemical structures [8-20]. The inhibiting effect of these compounds due to its adsorption on the metal surface and isolate the metal from the aggressive ions. The power of adsorption depending on several factors such as the nature and the charge of the metal or alloy, the type of the aggressive electrolyte and the chemical structure of the inhibitors [21].

In the previous work the electrochemical behavior of nickel, Inconel 600, Incoloy 800 and 316 stainless steel in HNO_3 solution was investigated using a cyclic voltammetry technique [22]. An extension of this work, the effect of chloride ion as pitting corrosion agent on the stability of the passive film on nickel alloy and stainless steel in 0.1M HNO_3 solution was studied using potentiodynamic anodic polarization technique at scan rate 1 mV/sec. Moreover, the inhibition of the pitting corrosion using some inorganic anions such as the sodium salts of chromate, tungstate, molybdate, phosphate dibasic and carbonate was studied

2 . Experimental Technique

The chemical composition of nickel (Ni) and three alloys, namely,Inconel 600, Incoloy 800 and 316 316SS are given in Table 1. For the potentiodynamic anodic polarization, a cylindrical rod embedded in Araldite with an exposed surface area to the corrosive medium of 1.70 cm^2 for Ni, 1.57 cm^2 for the Inconel 600,1.48cm^2 for the Incoloy 800 and 1.44 cm^2 for the 316SS was used. These electrodes were fixed to Pyrex glass tubing using neutral wax. To obtain a smooth surface before any experiment, the electrodes were mechanically polished using different grades of emery paper until 1200 grade, then degreased with acetone and washed with twice distilled water. The experiments were carried out at 25 ±1°C using air thermostat. All the chemicals were used by AR grade and used as received. All solutions were freshly prepared by distilled water. Measurements were carried out in three compartment electrochemical cell containing work electrode, Pt counter electrode and saturated calomel reference electrode. The potential was measured against a reference saturated calomel electrode (SCE). The potentiodynamic anodic polarization was carried out using a Wenking potentioscan, Type POS-73. The current density-potential curves were recorded on X-Y recorder, Type PL-3.

Table 1. The chemical composition of the alloys used.

Alloy	**Ni%**	**Fe%**	**Cr%**	**Cu%**	**Si%**	**P%**	**S%**	**Mn%**	**C%**	**Others%**
Nickel	100	-	-	-	-	-	-	-	-	-
Inconel 600	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

3. Results and Discussion

3.1 .Pitting corrosion of nickel alloys and stainless steel

The curves of Figs.1 -4 represent the potentiodynamic anodic polarization curves of nickel, Inconel 600, Incoloy 800 and 316 SS, respectively, in 0.1M HNO_3 contain different concentrations of NaCl as a pitting corrosion agent, at a scan rate of 1 mV/sec.

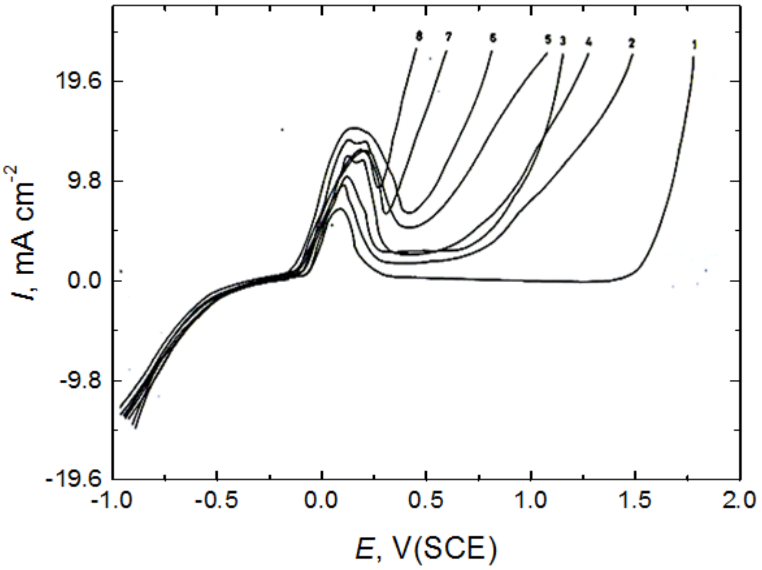

Fig. 1. The potentiodynamic anodic polarization curves of Ni in 0.1M HNO_3 with different concentrations of NaCl at 1.0 mV/Sec. 1- 0.0M NaCl; 2- 1x10^{-2} M; 3- 3x10^{-2} M; 4- 5x10^{-2} M; 5- 7x10^{-2} M; 6- 9x10^{-2} M; 7- 1x10^{-1} M 8- 1.5x10^{-1} M

The slow scan rate allows pitting initiation to occur at a less potential. The general features of these figures indicated that, As the potential increases the current remains constant until at certain potential the current increases rapidly to higher values due to the breaking the passive film and the occurrence of pitting corrosion. This potential is known pitting corrosion potential (E_{pitt}.). The pitting corrosion arises from the **competitive** adsorption between the O_2in the passive film and the Cl^- ions until the adsorption of the Cl^- ions are the predominate hence the pitting corrosion will occur on the surface.

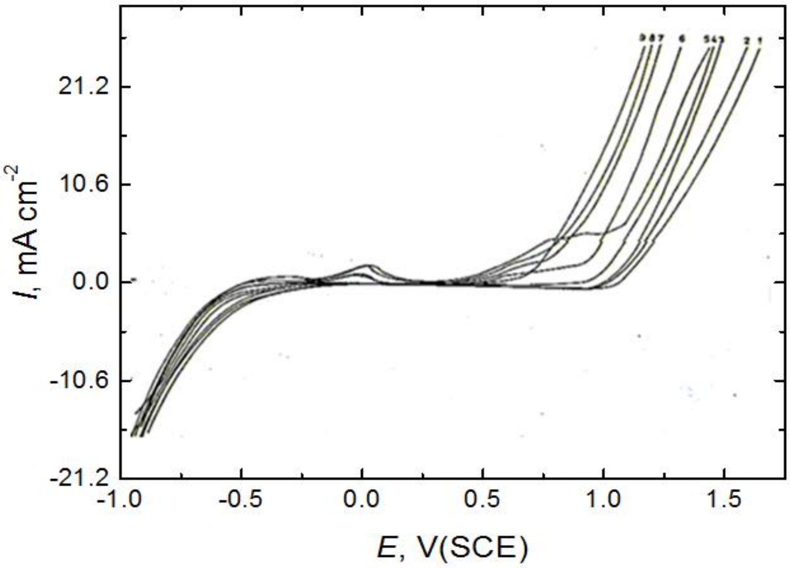

Fig. 2. The potentiodynamic anodic polarization curves of Inconel 600 in 0.1M HNO_3 with different concentrations of NaCl at 1.0 mV/sec. 1- 0.0MNaCl ; 2- 5x10^{-2} M ; 3- 1x10^{-1} M ; 4-.5x10^{-1} M ; 5- 2x10^{-1} M ; 6- 2.5x10^{-1} M ; 7- 2.7x10^{-1} M ; 8-3x10^{-1} M ; 9- 4x10^{-1} M

Inspection of Figs.1 and 2, the behavior of Ni and Inconel 600, have, approximately the same general feature and are characterized by the following aspects.

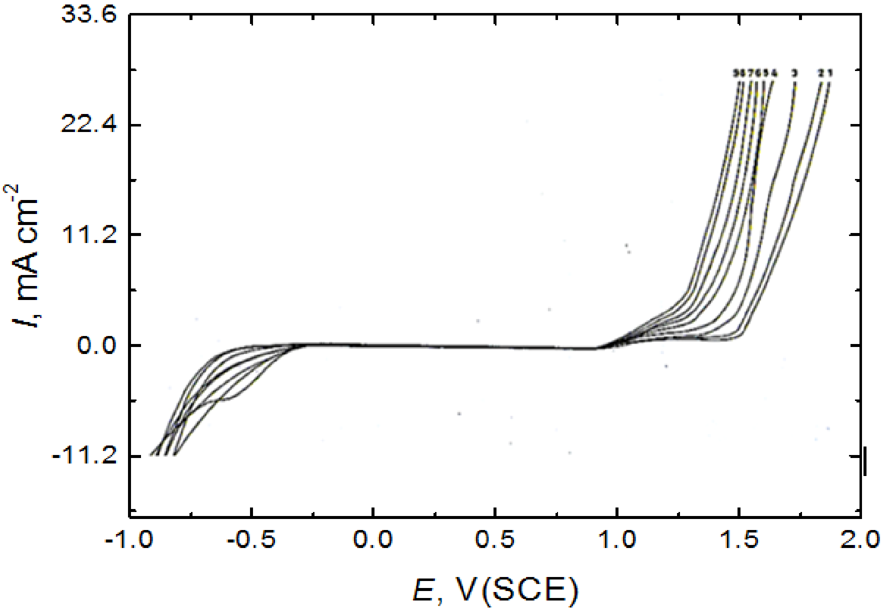

Fig. 3. The potentiodynamic anodic polarization curves of Ni in 0.1 M HNO_3 with different concentrations of NaCl at 1.0 mV/sec. 1- 0.0 M NaCl; 2- 5x10^{-2} M ; 3- 1x10^{-1} M ; 4- 1.5x10^{-1} M ; 5- 2x10^{-1} M ; 6- 2.5x10^{-1} M 7- 2.7x10^{-1} M ; 8- 3x10^{-1} M ; 9- 4x10^{-1} M

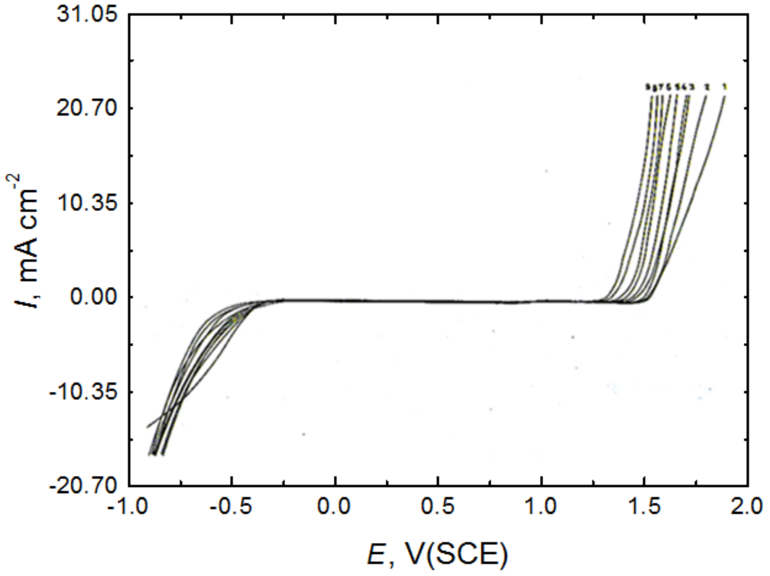

Fig. 4. The potentiodynamic anodic polarization curves of 316 SS in 0.1 M HNO_3 with different concentrations of NaCl at 1.0 mV/sec. 1- 0.0 M NaCl; 2- $5x10^{-2}$ M ; 3- $1x10^{-1}$ M ; 4- $1.5x10^{-1}$ M ; 5- $2x10^{-1}$ M ; 6- $2.5x10^{-1}$ M 7- $2.7x10^{-1}$ M ; 8- $3x10^{-1}$ M ; 9- $4x10^{-1}$ M

Increasing the Cl^- ion concentration lead to:

i. There is one anodic peak is observed. This peak may correspond to the active dissolution of Ni to Ni^{+2} [22]. Increase of the dissolution current density flowing along the active region, while the corrosion potential remains more or less constant. This behavior could be attributed to the adsorption of Cl^- ions on the metal surface at potentials less positive than those accepted for the onset of passivity, with the catalysis of the dissolution reaction.

ii. Shift, of the active / passive transition region towards more positive potentials.

iii. Shortening of the passive region, accompanied by an increase of the current density flowing along it. This could be attributed to the destruction of the passive film with initiation of localized corrosion. Examination of the electrode surface after polarization experiments showed visible pits whose number per unit area increases with increasing of the Cl^- ion concentration.

For Incoloy 800 and 316 SS, the anodic peaks is disappearing and the Cl- ion content has no effect on the passive film formed on the electrode surface until the potential at which pitting corrosion occurred is reached as shown in Figs. 3 and 4. The disappearance of any peaks due to the stability of the passive film. The passive film may be contains mixed oxides of iron, nickel, chromium and molybdenum oxides.

The dependence of the pitting corrosion potential of the four electrodes on the concentration of the Cl^- ion is illustrated in Fig. 5. This Fig. represents the variation of the pitting potential, $E_{pitt.,}$ with the logarithm of the molar concentration of the Cl^- ion in 0.1 M HNO_3.The curves of Ni and Inconel 600 (curves 1 and 2) are sigmoid in nature. (S-Shaped curves)

An inspection of this figure, it is clear that,

i. At low Cl^- ion concentrations, the pitting potential shifts slightly into negative direction because the concentration of the Cl^- ions are not sufficient to destroy completely the passivating film on the metal surface and the specimens exhibit a well-defined active to passive transition .It is called repassivation region.

ii. At relatively higher Cl^- ions concentrations, the pitting potential, E_{pitt}., varies with the log C_{Cl^-} according to a straight-line relationship in the form [23,24]

$$E_{pitt.} = X_1 - Y_1 \log C_{Cl-} \quad (1)$$

where X_1 and Y_1 are constants depending on both the nature and type of the aggressive anion and the electrode sample. This behavior is attributed to the fact that, the passivating film formed on the metal surface is destroyed at higher rates and the pits formed continuously propagate and cannot undergo repassivation [7,9]. The passive film in such cases could be expected to be thin and the passive region of the polarization curve rather small as shown in Figs.1-4.

iii. At higher Cl^- ion concentrations, it can be expected that film breakdown far exceeds film formation. A continuously propagated pit is formed in a limited range of pitting corrosion potential. The film forms but breaks down instantaneously.

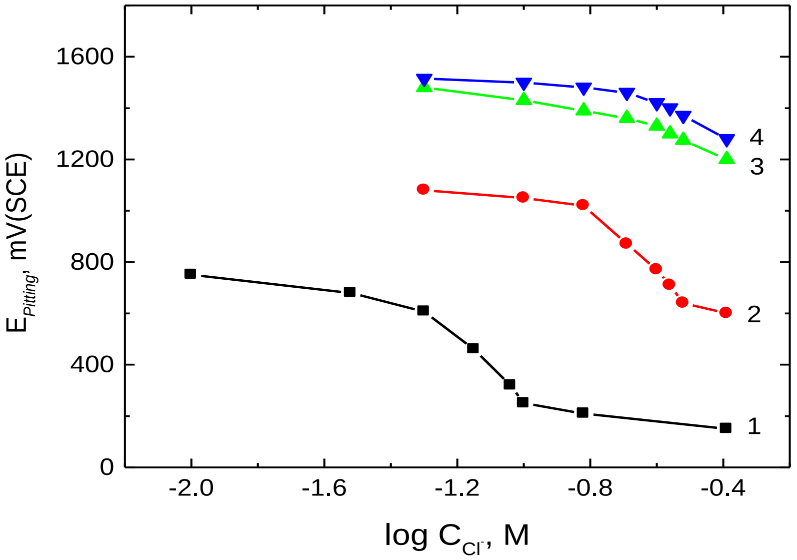

Fig. 5. The relation between the pitting potential (E_{pitt}) vs. the logarithm of molarity of Cl^- ions for:
1) Ni 2) Inconel600 3) Incoloy 800 4) 316 SS

An inspection of Fig. 5 (curves 3 and 4) for Incoloy800 and 316SS, the process occurring on the surface may be viewed as a broken line due to the following :There exists a competition between the formation of the film by anodic polarization and its destruction by Cl^- ions adsorption. When the concentration of Cl^- ions is low, the rate of passive film formation far exceeds than that of film breakdown. This is clear from the small change of the pitting potential in the negative direction. However, the passive film can be expected to be less perfect at the grain boundaries than over the grain interiors. Thus, once the critical potential is reached, the film breaks down at the grain boundaries and localized corrosion occurs. As the concentration of Cl^- ion increases, the rate of film breakdown fast approaches that of film formation. Once a particular concentration of Cl^- ions is reached 0.2M in case of Incoloy800 and 316SS, the rate of change of pitting potential in the negative direction is increased. Under such conditions, any weakness in the passive film resulting from grain boundaries in the specimen and the passive film breakdown becomes controlled by the Cl^- ion availability. The process of competitive breakdown and reformation of the film continues until a critical potential is reached, where the film breaks down completely and pitting corrosion occurs. Since the concentration of Cl^- ions are high, their availability is the same over the entire specimen surface and this aids the complete breakdown of the film.

Further inspection of the curves of Fig. 5 illustrates that, at one and the same Cl^- ions concentration the shift of pitting corrosion potential to more noble values according to the following sequence :

316SS > Incoloy800 > Inconel 600 > Ni

This order reflects the high resistivity of 316SS to pitting attack rather than the other alloys. Such behavior could be attributed to the presence of the Mo (2.35%) in the chemical composition of 316SS. The role of Mo in the improvement the resistance of the alloy to pitting attack due to the following reasons:

i- Thickening of the passive film on stainless steel
ii- Formation of heteropoly acid film, which is less, readily hydrolyzed and more readily polymerized.
iii-Impeding the adsorption of Cl^- ion on the surface of steel by increasing the oxygen affinity of the steel [25].
iv-Formation of an amorphous oxide film with a glassy structure [26].

On the other hand, in case of Fe – Cr alloys without Mo, the amount of Cr needed to attain inhibition of pitting in 1 N HCl is about twice that of the Fe – Cr containing Mo [27]. In addition, XPS examination of the passive film showed undoubtedly the existence of oxidized MO, which was in the hexavalent state in the passive film of stainless steel containing 1-5% Mo.

In addition, Mo (VI) oxide, which is fixed in the state of solid solution in the film, is thought to promote the ability of Cr (III) film for the pitting resistance, since the stability of Mo (VI) oxide is very high in acid solutions containing Cl^- ions. Even if the Cr (III) content of the passive film is relatively low, a high resistance of the film to pitting attack should result from a film containing Mo(VI) in the state of solid solution [28]. Generally,the beneficial effect of the alloyed molybdenum on the localized corrosion resistance may be related to the fact that, the soluble and insoluble molybdate are known to act as corrosion inhibitors for stainless steel.

On the other hand, it is obvious that, the Incoloy800 is more resistant to pitting attack than Inconel 600. This behavior could be attributed to the higher Cr^- content of Incoloy800 than that of Inconel 600 The pitting resistance of Inconel 600 is more than Nickel due to the presence of Cr in the chemical structure of Inconel600 as shown in Table (1).

Analysis of the passive film formed on Fe –Cr alloys by XPS, showed that it consists of Cr(III) and Fe (III), and Cr(III) increases with increasing the Cr – content[28]. Since sites where Fe(III)exist in high concentrations in the passive films are thought to be less resistant to attack by Cl- ions, the pitting resistance of Fe – Cr alloys will be considerably improved by overcoming this drawback with increasing of Cr (III) in the passive films. The Cr enrichment in the passive film under pitting corrosion conditions indicates that the formation and growth of the passive film proceeded even if pores including pits were formed.

3.2. Inhibition of pitting corrosion of nickel alloys and stainless steel by some inorganic anions.

The effect of addition of increasing concentrations of the sodium salts of chromate, tungestate, molybdate, phosphate dibasic and carbonate on the potentiodynamic anodic polarization curves of nickel, Inconel 600,Incoloy800 and 316 SS in 0.1M HNO_3 + 0.1M NaCl at a scanning rate of 1 mV/sec.was investigated. Similar curves to Figs1-4 were obtained but the values of E_{pitt}. are changed (not shown).

It is clear that, the presence of increasing additions of CrO_4^{2-}, WO_4^{2-}, MoO_4^{2-}, $HPO_4^{2-}$and CO_3^{2-} anions cause a marked shift of the E_{pitt} into the positive (noble) direction. The higher the concentration of these anions, the greater is the depression of the dissolution current density. Both effects denote increased resistance to both active corrosion and pitting attack by these anions.

The dependence of the pitting potential, E_{pitt}., of nickel, Inconel 600, Incoloy 800 and 316 SS on the concentration of the inhibitor anions can be seen from the curves of Fig. 6. In this figure, $E_{pitt.}$, is plotted as a function of the logarithm of the molar concentration of the added compounds. From the curves of this figure, it is clear that:

i- The presence of increased concentrations of these anions causes a shift of the pitting potential in the noble direction, in accordance with the following equation:

$$E_{pit}. = X_2 + Y_2 \log C_{inh.} \qquad (2)$$

Where, $X_2$and Y_2 are constants that depend on the type of both the inhibitor and aggressive anions as well as on the metal or alloy under test.

ii- At one and the same inhibiting anion concentration ,the efficiency of inhibition of the additive compounds for the different electrodes shows a large intrinsic scatter from electrode to electrode. From the curves of Fig. 3. the inhibiting efficiency of these compounds is in the following sequence:

For Nickel: $CrO_4^{2-} > HPO_4^{2-} > MoO_4^{2-} > WO_4^{2-} > CO_3^{2-}$

For Inconel 600: $CrO_4^{2-} > HPO_4^{2-} > MoO_4^{2-} > WO_4^{2-} > CO_3^{2-}$

For Incoloy800: $CrO_4^{2-} > WO_4^{2-} > MoO_4^{2-} > HPO_4^{2-} > CO_3^{2-}$

For 316 SS: $WO_4^{2-} > CrO_4^{2-} > MoO_4^{2-} > HPO_4^{2-} > CO_3^{2-}$

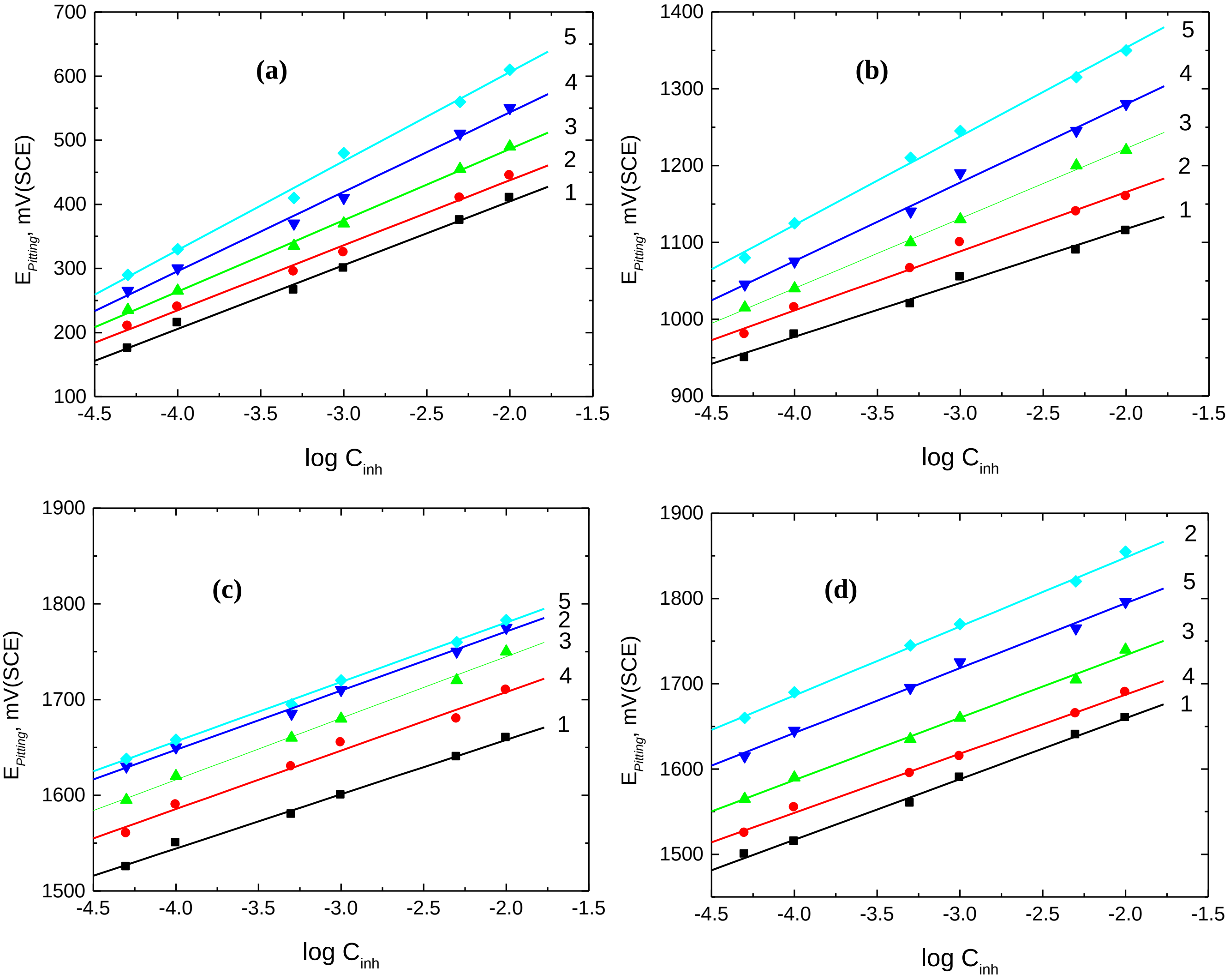

Fig.6. The relation between the pitting potential Vs the logarithm of molarity of inhibitor concentrations for:
a) Ni b) Inconel 600 c) Incoloy 800 d) 316 SS
1) Na_2CO_3 2) Na_2WO_4 3) Na_2MoO_4 4) Na_2HPO_4 5) Na_2CrO_4

From the above sequence, it is clear that CrO_4^{2-} ions have the best inhibitor efficiency. It is followed by HPO_4^{2-} ions for samples of high Ni content. In case of Incoloy 800 and 316SS of higher Fe content HPO_4^{2-} ions have a poor inhibitor efficiency. This phenomenon may be attributed to the solubility product of nickel phosphate, which is lower than that of iron phosphate.

On the other hand, WO_4^{2-} ions have the same or better efficiency than CrO_4^{2-} ions in the case of Incoloy 800 and 316 SS (of high iron content). This behavior could be explained on the basis that, WO_4^{2-} ion is strongly adsorbed on metals having an abundant of Fe_2O_3 on their surfaces. Comparing XPS spectra of samples exposed to inhibitors with a blank indicated that, molybdenum and tungsten are present in the hexavalent state, whereas chromium is present in the trivalent form [29]. The element concentrations observed are in the order:

Chromium > tungsten > molybdenum

Several mechanisms have been forwarded to explain the role of CrO_4^{2-} anions in the corrosion inhibition. The inhibiting effect of chromate can be explained on the basis of a competitive adsorption between the inhibitor (CrO_4^{2-}) and aggressive (Cl^-) ions [30]. If chromate adsorption predominates on the open surface, the passive film formed must involve Cr_2O_3 or $Cr(OH)_3$ [31]. However, if adsorption of Cl^- ions predominates, pitting corrosion will occur on the free surface. XPS studies of the passive anodic films showed that, Cr^{+6} ion is reduced to Cr^{3+} ions with the formation of protective Cr_2O_3 and some CrO_4^{2-} ions remain unreacted and incorporated in the film formed on the metal surface [32]. Although, the various phosphate anions cannot be reduced electrochemically, the inhibiting effect of these anions can be attributed to, i) their competitive adsorption on the metal surface, ii) the formation of an adsorbed layer on the oxide film iii) formation of a highly insoluble salt with dissolved metal ions which prevents the penetration of aggressive Cl^- ions and consequently, decrease the rate of pitting corrosion. The inhibiting action of phosphate dibasic anions was found to increase with increasing the nickel content of the test samples.

The molybdate anions play a direct role in the inhibition process through the formation of a protective passivation layer of molybdenum oxide of lower valence. The passivation occurs also as a result of specific adsorption rather than due to inhibitor concentration. The pitting corrosion of the studied alloys was inhibited by the presence of MoO_4^{2-} ion in solution. The inhibition of pit growth in Mo containing stainless steels is due to the adsorption of MoO_4^{2-} ions which result from the dissolution of the stainless steel at the initial stages of pitting. Analysis of the film formed on 304 stainless steel surface immersed in water containing MoO_4^{2-} ions (using ion micro mass analyzer, IMMA) showed that it is composed of Cr_2O_3 dominant passive film containing a small amount of MoO_3 [33]. Therefore, the mechanism of inhibition may be considered as MoO_4^{2-} ions assist the formation of Cr_2O_3 dominant passive film containing MoO_3 or by adsorption on the metal surface as $HMoO_4^{2-}$ ions. The inhibitive efficiency of molybdate is less than that of CrO_4^{2-} ion. This behavior is almost due to the strong oxidizing ability of chromate, which leads to the rapid formation of the passive film on the metal in the absence of dissolved oxygen. Molybdate, being a much weaker oxidizing agent than chromate, could not be a strong passivator in the absence of oxygen. It was suggested that in aerated environments, the oxygen is considered as the primary passivation, and the inhibiting anion action is only by film repairing.

The positive shift of the pitting potential of the electrode samples with increasing the concentration of molybdate anions indicates an increased protective ability of the film formed on the metal surface. This increased protective nature of the passive film may be a result of film strengthening through increased incorporation of adsorbed molybdate into the nickel, chromium and iron oxide films or due to the increased availability of the adsorbed molybdate ions to repair the defects in the passive film. The dependence of the nickel alloys and stainless steel passivation on molybdate adsorption suggests, as with many other inhibitors that aggressive anions could compete the inhibitor adsorption sites on the surface and determinately influence passivation. The inhibiting action of WO_4^{2-} ion is found to depend on the aggressive Cl^- ion concentration. Tungestate ion has a better inhibiting effect for Incoloy 800 and 316SS (of high iron content). This behavior could be explained on the basis that, WO_4^{2-} ion is strongly adsorbed on the metal surface which possesses a protective film of Fe_2O_3 on its surface. The low inhibiting efficiency of CO_3^{2-} anion may be attributed to partial passivation of the electrode surface because this anion does not manifest oxidizing properties and may exert an indirect effect on the cathodic reaction by increasing its rate and consequently influence the anodic reaction, retarding its rate.

Conclusions

1) Pitting corrosion of Ni, Inconel600, Incoloy800 and 316 SS in 0.1 M HNO_3 solution containing different concentrations of Cl^- ions was studied.
2) Increasing the concentrations of the Cl^- ions shifts the E_{pitt}. into more active direction
3) The E_{pitt} varied with the log C_{Cl}^- ions in case Ni and Inconel 600 according to S-shaped curve.
4) The E_{pitt} varied with the log C_{Cl}^- ions in case Incoloy 800 and 316SS according to broken lines.
5) Addition of the sodium salts of chromate, tungstate, molybdate, phosphate dibasic and carbonate shifts the E_{pitt} into more positive values indicating an increased resistance to pitting attack

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