

## CORROSION STUDIES ON SS-321 IN CHLORIC-ACID SOLUTION MEDIUM

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### ABSTRACT

**CORROSION STUDIES ON SS-321 IN CHLORIC-ACID SOLUTION MEDIUM.** Experimental corrosion studies on commercial AISI-321 have been carried out. The experiments were carried out in a chloric-acid medium with a concentration variation of 0.1 mol. 0.2 mol., 0.3 mol. and 0.4 mol corresponding to pH values of 1.0, 0.7, 0.523, and 0.4 respectively. The experiments were carried out using a type M-273 EG&G potentiostat/galvanometer test instrument. The post-corrosion samples' microstructure were analyzed with the aid of EDS (energy dispersive spectroscopy) equipped SEM instrument to detect the presence of any viable corrosion byproducts. For further verification, X-ray diffraction method was also used to detect any possible emerging corrosion byproducts on the samples' surfaces. Experimental results confirm that AISI-321 commercial alloys immersed in a chloric acid corrosion medium with a variation of concentration experience very little or almost no corrosion, and that according to the so-called Fontana's criteria these test-materials turn out to have an excellent resistance toward chloric acid corrosion. This is also evidenced by the very low corrosion rate value measured in this study. EDS study and X-ray diffraction results indicate that the possible ensuing corrosion byproducts are iron oxides, chrome oxides, and iron chlorides.

**Key words :** Corrosion, SS-321 material, chloride acid medium

### ABSTRAK

**PENELITIAN KOROSI PADA BAHAN SS-321 DALAM MEDIUM LARUTAN ASAM CHLORIDA.** Telah dilakukan eksperimen korosi pada bahan komersial AISI-321 dalam medium asam klorida dengan variasi konsentrasi sebesar 0,1 mol, 0,2 mol., 0,3 mol, dan 0,4 mol. yang masing-masing bersesuaian dengan nilai pH 1,0, 0,7, 0,523, dan 0,4. Eksperimen dilakukan dengan alat uji potentiostat/galvanometer tipe M-273 EG&G. Struktur mikro pada permukaan sampel yang telah dikorosikan diperiksa dengan SEM yang dilengkapi dengan EDS (*Energy Dispersive Spectroscopy*) guna mendeteksi produk korosi yang mungkin. Untuk konfirmasi, hasil produk korosi yang mungkin tumbuh pada permukaan sampel juga dideteksi dengan difraksi sinar-x. Hasil eksperimen menunjukkan bahwa pada bahan komersial AISI-321 yang dikorosikan pada medium asam klorida dengan variasi konsentrasi tersebut hanya terjadi sedikit korosi sehingga menurut kriteria Fontana spesimen uji ini memiliki ketahanan yang sangat baik terhadap korosi dalam asam klorida. Hal ini juga ditunjukkan oleh sangat rendahnya nilai laju korosi dalam eksperimen ini. Dari pemeriksaan dengan EDS dan difraksi sinar-x ditunjukkan bahwa jenis produk korosi yang mungkin adalah oksida besi, oksida krom, dan klorida besi.

**Kata kunci :** Korosi, bahan SS-321, medium asam klorida

### INTRODUCTION

One of the motivation behind the fabrication of stainless steels are their excellent resistance to corrosion facilitated by the presence of chromium. One of the commercially available stainless steel is the SS-321 which is an austenite-type low-carbon steel. SS-321 contains chromium as the main alloying element in iron and steel for inhibiting corrosion combines with other alloying elements such as nickel, silicon, titanium, and carbon with a composition listed in Table 2.

Kyung Seon Min *et al.* have discussed in details all aspects covering the influence of carbide grain boundary density on SS-321 alloy [2], and carbide grain boundary feature correlation with respect to its creep

and fatigue properties [3]. Corrosion measurements on SS-321 samples coated with ion implantation and ceramic film have been carried out by F. Noli *et al.* [4], where sulphate-acid ( $H_2SO_4$ ) solution was used as the corrosion medium. High temperature corrosion measurements on SS-321 in atmospheric environment have been carried out by Nurdin Effendi *et al.* [5,6].

The question arises concerning the extent to which the alloying elements in the steel, especially nickel and chrome could function as a cathodic safeguard in the samples. If nickel and chrome in question do function as a cathodic shield, it would clearly be distinctively present or observable as the cathodic

potential pattern section of the potentiodynamic curves. This question point provides the main objective of this research work. The main objective of this study is to measure the corrosion-resistance of SS-321 steel with respect to a chloride-acid anorganic environment with varying concentration of 0.1M, 0.2M, 0.3M, and 0.4M respectively; especially in this case special attention is paid to the resistance of SS-321 steel with respect to chloride attack due to chlorine existing in chloric acid which turns out to be very destructive in nature. Another aim of this study is to gather information on the various types of byproducts brought about by the corrosion process when the test-samples are immersed in a chloride-acid medium. This experiment would be supplemental to the similar corrosion experiments carried out earlier in an atmospheric medium.

In SS-321 alloys nickel acts both as an austenite-phase forming agent as well as an anti-corrosion agent [10,11]. Chrome is also known as an excellent alloying element because of its anti-corrosion property. Silicon is used as a nucleation agent because of its high melting and freezing points, as well as its anti-corrosion property [10]. Manganese has an excellent anti-corrosion property and acts as a phase-stabilizer for the austenite-phase[10,11], carbon is an excellent hardness-increasing element, and finally titanium functions as a sensitizing element in the alloy [10,11].

The purpose of this experiment is to investigate corrosion behavior of SS-321 alloys in chloride acid solution medium, and the damage of the sample due to the penetration of chloride element.

## THEORY

Corrosion reaction could be viewed upon a thermodynamic system surrounded by an environment forming an imaginary wall [7]. Corrosive reaction in a certain type of environment occurs rather spontaneously by releasing free-energy from the system into the environment. Normally, a system is endowed with a certain amount of internal-energy, and most of this energy could be transformed into another form of energy, called the free energy [7, 8]. Corrosive reaction is a transitional condition, and is often labeled as a transition-reaction.

According to a theory first formulated by Arrhenius, corrosion rate is determined by two main parameters, free activation energy and temperature. Both parameters form a formula expressing the reaction constant,  $k_{corr}$ , otherwise known as Arrhenius constant [7]. Activation free-energy is a barrier-energy between the alloy and its corrosion product. For this reason it becomes clear that the condition where the alloy is at a higher energy level compared to its surrounding element is undesirable. In this system, corrosion occurs whenever free-energy is available as the potential to generate the flow of electric current (electrons) between

anode and cathode in the sample. In this case the test sample should have more anodicity than the reference potential, to enable the cathode to collect the electron flow generated by the test sample[7, 8]. This electronic current or flow is the main cause of corrosion in the test-sample. The resistance to corrosion is measured using the resistance polarization to produce the polarization resistance parameter ( $R_p$ ). Polarization resistance is a measure of the specimen resistance toward corrosion of the specimen while connected to an external potential source. The main function of  $R_p$  is to calculate the corrosion current,  $i_{corr}$ , which is to be determined prior to calculating the corrosion rate. The following expression elucidate the relation between  $R_p$ , Tafel constants, and corrosion current  $i_{corr}$ .

$$\frac{\Delta E}{\Delta i_{spt}} = R_p = \frac{\beta_a \beta_c}{2.3(i_{corr})(\beta_a + \beta_c)} \dots\dots\dots (1)$$

with :  $\Delta E / \Delta i_{spt} = R_p$  = polarization curve slope  
 $\beta_a$  = polarization tafel constant  
 $\beta_c$  = cathodic tafel constant  
 $i_{corr}$  = corrosion current  
 2.3 = log 10

After  $i_{corr}$  is calculated, the corrosion rate could be determined as follows

$$\text{Corrosion rate (mpy)} = 0.13 \cdot i_{corr} \cdot \frac{E_w}{A \cdot d} \dots\dots (2)$$

with : mpy = mili-inch per year  
 $E_w$  = weight equivalent (g/equivalent)  
 A = area (cm<sup>2</sup>)  
 d = density (g/cm<sup>3</sup>)  
 0.13 = conversion factor

Therefore according to equation 2, corrosion current determines the corrosion rate. In this work the *polarization resistance / polarization linear* technique is carried out first to obtain the polarization resistance ( $R_p$ ). The polarization technique is superior with respect to other techniques because it is faster and it uses a relatively smaller potential scanning which causes insignificant changes or damages to the sample's surface.

The potentiodynamic technique is utilized to determine the active and passive region of the measured alloy system. The overall form of the curve (Figure 2) is an indication of the corrosion behavior of the samples immersed in the test-solution. This technique is capable of producing reliable data if carried out at a relatively low scanning rate. Passivation is defined as the loss of chemical reactivity in materials while subjected to certain environment or conditions. Passivation generally occurs because of the formation of a protective layer in the alloy-electrolyte interface therefore preventing a direct contact between the alloy's surface and the electrolyte. The measurement of an alloy tendency toward passivation when it is immersed inside a certain type of

environment is very important in order to obtain information on the corrosion behavior or characteristics of that particular alloy. The measurement is accomplished by drawing the characteristic anodic polarization curve of the sample. The halogenic ions, especially the chloride ion could destroy the passivation in the spots or local points on the sample which is passive with respect to the air environment. (e.g. Cr, stainless steels). The local spot of the active alloy acts as an anode whereas the cathode forms the wider region of the passive alloy.

In a potentiodynamic-test measurement, the assigned potential values cover a far broader range compared to the potential values used in a polarization-resistance measurement, with the consequent that samples usually experience some damages after a potentiodynamic experiment. The corroding medium's concentration also influences the corrosion rate because the concentration will modify the pH value. Every solution concentration used in this experiment is converted into its corresponding variation of pH values as tabulated in Table 1.

**Table 1.** Molar to pH values conversion of chloride acid solution.

Molar	pH
0.1	1.0
0.2	0.7
0.3	0.522
0.4	0.4

## EXPERIMENTAL METHOD

### Materials

The test samples used in this work are plates of SS -321 alloys with a thickness of 5 mm; the chemical composition is shown in Table-2. The applied solution used as the corrosion-medium is chloric-acid solution with a concentration-variation of 0.1 moles to 0.4 moles with a steady increment in step of 0.1 moles. The choice of this variation is strongly delimited by the instrumental precision limit, i.e. above 0.4 moles the corrosion rate

**Table 2.** Alloying elemental composition of SS-321 stainless steel (w %) [1].

Element	weight%
Ni	9-12
Cr	17-19
C	0.08 (max)
Si	1.0 - 2.0
Mn	2.0
Ti	min. 5 x %C
S	0.03 (max)
P	0.045 (max)

increases steeply outside the range of the detection limits of the instrument.

### Equipment

This experimental work uses a Buehler cutting machine with a Buehler-type disc blade, a double polishing machine fabricated by Karl Kolb (Denmark); sand papers of various grades for polishing. The corrosion-testing machine used is the potentiostat/galvanostat M273 assembled by EG & G Princeton. Applied Research Corporation, equipped with test-beakers, vials and pipettes test tubes. The supporting analysis instruments for the corrosion-samples are a Shimadzu XD-610 X-ray diffractometer, a Nikon UFX-DX optical microscope (for surface microstructure observations), and an EDS equipped Phillips SEM microscope.

### Methods

Sample preparation is carried out in compliance with the standard procedure [9]. Samples are cut into equal sized and shaped into round-forms by polishing, to a final size of 15 - 16 mm diameters. Afterwards the surfaces of the samples were polished in a polishing-machine until a relatively uniform and surface's smoothness and evenness is achieved. The main objective is to get rid of the oxide-layer in the surface. The first experimental-test performed is the polarization technique experiment. The experimental procedure is outlined as follows: First, the sample is immersed into the three-electrode corrosion-test instrument, which has been filled with a 600 ml of corrosion-fluid, and standard saturated calomel is used as the reference electrode, two carbon-bars are used as supporting electrodes. The sample functions as the working electrode.

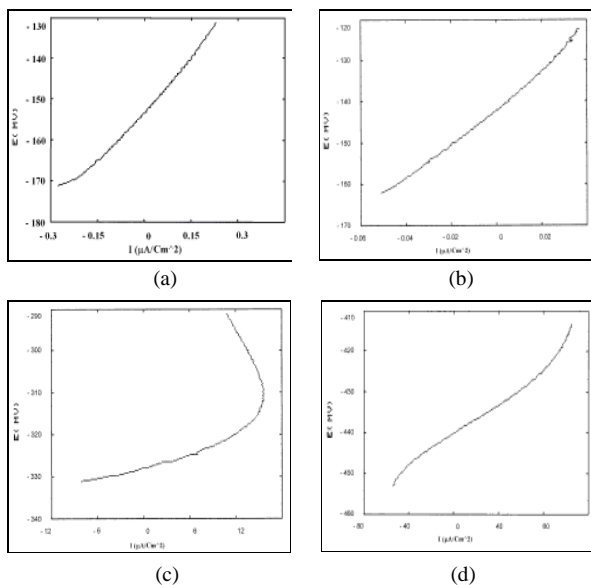
To start the experimental measurement, the corrosion cell is connected to the potentiostat by applying a potential 20 mV below the corrosion potential and 20 mV above the corrosion potential, and the scanning rate is set at 0.1 mVs<sup>-1</sup>; from this measurement both *R<sub>p</sub>* values and anodic-cathodic Tafel constants will automatically be obtained. Potentiodynamic-technique is carried out next. The primary aim of this measurement is to map the active as well as passive characteristic areas of the specimen-solution system by applying a working potential in the range of 500 mV below and 1600 mV above the corrosion potential with a scanning rate of 0.5 mVs<sup>-1</sup>. Finally these experimental curves are analyzed.

To analyze the sample's surface and to determine its corrosion products, the corrosion-test samples are observed under an EDS equipped electronic microscope (SEM). In order to complete the classification of the resulting types of corrosion-product, the samples are each examined using an X-ray diffractometer. X-ray

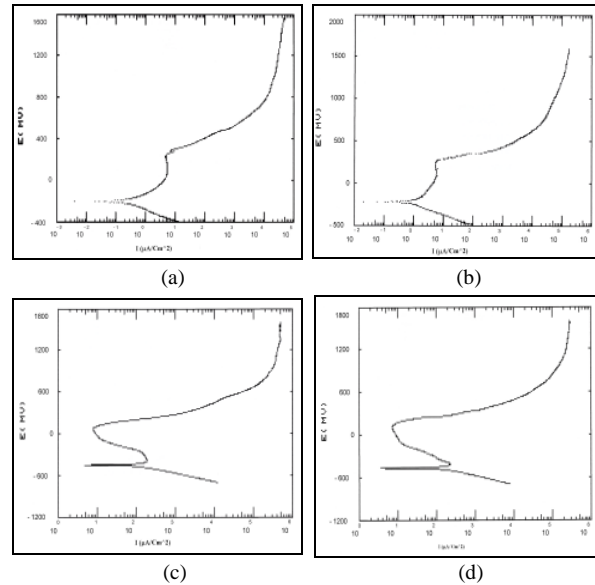
diffraction analysis is accomplished by consulting the software program available from JCPDS (Joint Committee On Powder Diffraction Standard).

## RESULTS AND DISCUSSION

The curves obtained from the corrosion-test measurements on SS-321 samples using the polarization resistance technique in an HCl medium for each pH or concentration value should generate the curves presented in figures 1-a through 1-d and the experimentally measured parameters available in Table 3. The experimental results clearly show that for a SS-321 sample immersed in an HCl solution with 0.1- 0.4 molar concentration (equivalent to a 1.0 – 4.0 pH value ) the lowest corrosion potential of -344.47 mV has been recorded for the pH value of 0.4. The corrosion rate in the sample tends to peak at this pH value, which is around 2.5 mpy (Table-3). According to the rule established by Fontana [7, 8], this particular corrosion



**Figure 1.** Polarization-resistance potential versus current curve pattern obtained from SS-321 sample immersed in a chloride-acid medium : (a). pH = 0.4 (b). pH = 0.522 (c). pH = 0.7 (d). pH = 1.0



**Figure 2.** Potentiodynamic potential versus current curve pattern obtained from SS-321 sample immersed in a chloride-acid medium: (a). pH = 0.4. (b). pH = 0.522. (c). pH = 0.7. (d). pH = 1.0.

rate value should rank SS-321 in a class of materials with excellent corrosion-resistance with respect to chloride acid solution corroding medium.

Figures 2-a to 2-d show several potentiodynamic curves of a SS-321 sample in a chloride acid medium with a variation of pH, the concurrently generated experimental parameters are presented in Table 4. In a measurement procedure of potentiodynamic technique, the assigned potential range is far broader than the range used in a polarization-technique experiment, and this would ultimately lead to the sample(s) being damaged or ruined in the process.

The potentiodynamic curves would display the following general pattern: initially the samples are cathodic, because of cathodic protection process initiated by the anticorrosion elements, and in this potential range and up to its free corrosion potential ( $E_{corr}$ ), the current will concurrently decrease. The free corrosion potential is the scale which eventually determines the virtual equilibrium condition; now for this

**Table 3.** Experimental results of a SS-321 sample immersed in an HCl solution medium Environment with a variation of molar concentration or pH using the potentioreistance method. (The numbers inside the brackets are pH values).

Experimental Parameters	Molar Concentration (pH)			
	0.1 (1.0)	0.2 (0.7)	0.3 (0.523)	0.4 (0.4)
E(I=0) (mV)	-153.06	-141.9	-327.96	-344.47
$R_p$ ( $k\Omega\text{ cm}^{-2}$ )	83.8	44.12	0.4549	0.2158
$I_{corr}$ ( $\mu\text{Acm}^{-2}$ )	0.12	0.35	7.16	9.68
Corrosion Rate (mpy)	0.0312	0.0907	1.85	2.5
Correlation	1.0	1.0	1.0	1.0
Range of Experimental Potential. E(I=0) $\pm \Delta$ mV	10	10	10	10

**Table 4.** Experimental results of a SS-321 sample immersed in an HCl solution medium environment with a variation of molar concentration or pH using the potentiodynamic method.

Molar Concentration	pH	Free Corrosion pot (mV)	Passivation Potential (mV)	Transpassive Potential (mV)
0.1	1.0	-198.06	+70	+300
0.2	0.7	-214.11	+100	+310
0.3	0.522	-465.69	-400	+175
0.4	0.4	-445.55	-400	+125

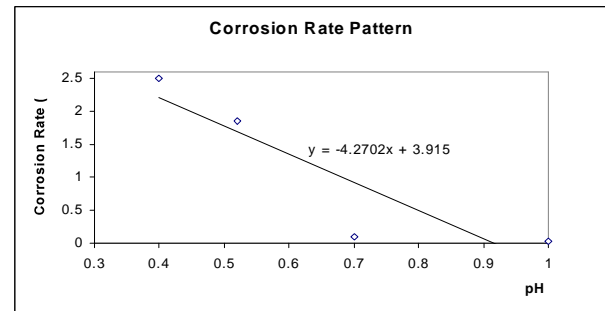
particular condition the alloy would experience deliberate corrosion without any external interference in the form of additional input of potential. After the free-corrosion potential has been reached, then to generate the corrosion process in the sample, the overall potential must be steadily increased, which in turn causes the corrosion current to increase to generate corrosion in the sample. The free corrosion-potential is an indicator whether a sample immersed in a corrosion-medium is easy or difficult to corrode; the higher the free corrosion potential, the more difficult it is for a sample to experience corrosion.

Experimental results of potentiodynamic technique measurements show that the lowest corrosion potential at pH equals to 0.522, which is equivalent to a 0.3 mol concentration chloride acid solution. has been recorded to be -465.69 mV. It is clear that for pH values equal to 0.522 and 0.4 the passivation potential is of the same magnitude which is about -400 mV; on the other hand for pH values equal to 0.522 and 0.4 the transpassive potential is measured to be +175 mV and +125 mV respectively. At pH equals to 0.4 the free corrosion-potential increases from -465.69 mV at pH 0.522 to a value of around -445 mV; the reason behind this is an increase of cathodic protection and also because of a stronger acid medium, the exchange current generation becomes more significant. forming more abundant hydrogen bubbles which tend to block the corrosion current increases [8].

Cathodic protection arises because SS-321 contains anti-corrosion alloying elements nickel (Ni) and chrome (Cr) each with a reduction potential of -0.254 volt and -0.744 volt respectively and comparably more negative with respect to the iron (Fe) reduction potential of -0.037 volt. Before attacking Fe, the corrosion solution must first overcome the negative potentials generated by Ni and Cr. If the external potential increases. energy formed by the external potential will surmount the potential barrier formed by Ni and Cr. Further, increment of the external working potential will induce the corrosion current to increase and the samples will be corroded until the potential reaches the reverse potential. namely the onset potential where the passivation process starts to occur. At this condition. if the working potential is being steadily incremented. current would begin to decrease. This is brought about by the alloy

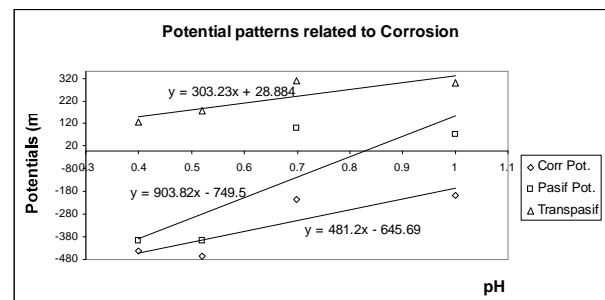
partial protection. for example corrosion product adhering to the samples' surface reducing further contact between electrolyte solution and the samples' surface.

The distribution pattern of the variation corrosion rate data versus corrosion-solution pH values is shown in Figure 3. The corrosion rate tends to decrease with respect to pH; this means that corrosion rate is proportional to the solution's acid concentration; The higher the concentration of chloride acid applied in the corrosion solution, the higher the corrosion rate is obtained.

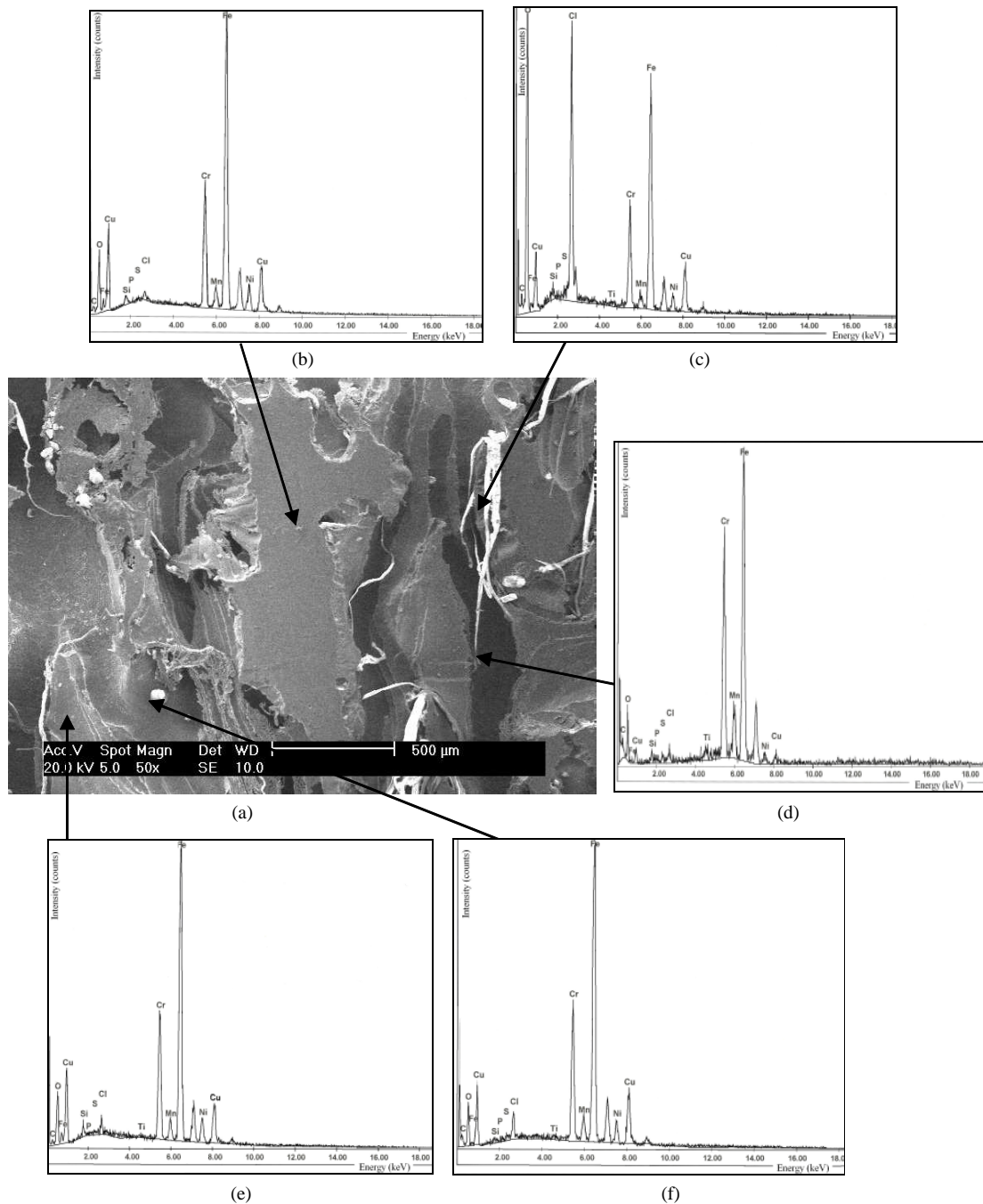


**Figure 3.** Plot of corrosion rate versus corrosion solution pH pattern.

The data distribution pattern for corrosion potential, passivation potential and transpassivation potential variation versus corrosion solution pH values is shown in Figure 4. It is clearly visible that corrosion potential, passivation potential and transpassivation potential variation tends to increase with pH values. This means that corrosion potential, passivation potential and transpassivation potential are inversely proportional to the solution-acid concentration.



**Figure 4.** Data plots for corrosion potential, passivation potential, and transpassivation potential versus corrosion solution pH values.



**Figure 5.** SEM Micrograms of a corrosion sample immersed in a pH = 0.4 chloride acid medium : a. SEM microgram of the microstructure. The microgram gives a clear indication of surface erosion caused by the corrosion medium. b. EDS results taken over a spot on the sample's surface. EDS pattern shows that the sample's surface still seems to be flat overall; there is a possibility that chrome oxide or iron oxide are present on the spot c. This spot reveals the presence of a significant amount of chrome oxide, iron oxide and iron chloride. d. EDS pattern showing the possibility of the presence of chrome oxide, iron oxide, iron chloride, and a minor amount of nickel hydride oxide. e. EDS pattern showing the possibility of the presence of chrome oxide, iron oxide or iron chloride. f. EDS pattern showing the possibility of the presence of chrome oxide, iron oxide and a small amount of iron chloride.

Results of microstructure measurements on a corrosion sample in chloride acid medium with pH equal to 0.4 is shown in Figure 5: micrograms taken with SEM. Here it is obvious that the sample's surface has been eroded by the corrosion medium. consequently showing corrosion erosion. EDS pattern (Figure 5.b) on a selected

spot in the surface reveals the possibility of chrome oxide ( $Cr_2O_3$ ) or iron oxide ( $Fe_2O_3$ ) being present in the sample. On another selected spot (Figure 5.c). EDS pattern reveals the possibility of chrome oxide, iron oxide, and iron chloride. EDS pattern on still another surface spot (Figure 5.d) also reveals the possibility of chrome oxide,

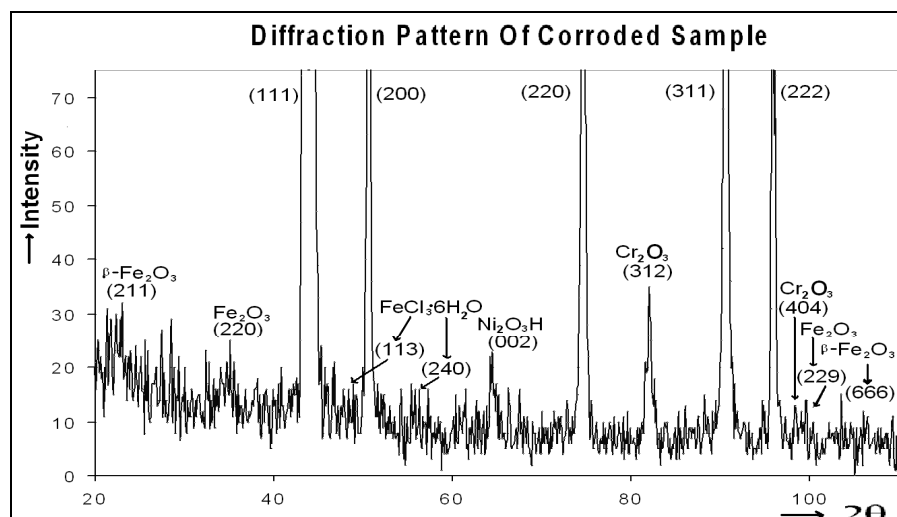


Figure 6. X-Ray diffraction intensity pattern of one corrosion sample after corrosion in a pH = 0.4 chloride acid medium.

iron oxide, iron chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) and nickel hydride oxide ( $\text{Ni}_2\text{O}_3\text{H}$ ) in a minor amount being present in the sample. Another EDS pattern for yet another spot (Figure 5.e) also strongly indicates the possibility of chrome oxide, iron oxide, and iron chloride being present in the sample. Finally the same could be concluded for yet another selected spot (Figure 5.f), namely that chrome oxide, iron oxide, and iron chloride possibly coexist in the sample. Based on both microstructural investigation supplemented by EDS data it could be claimed that in a chloric acid medium, pitting/erosion corrosion have certainly occur. This is brought about by chlorine assault on the sample's bulk; chlorine is able to penetrate and enter the bulk, and forming porosity, which later on will be filled with corrosion solution causing abrasion in the sample's bulk. The ensuing corrosion byproducts are chrome oxide, iron oxide, and iron chloride. This finding has been confirmed by x-ray diffraction data.

X-ray diffraction pattern on one particular pH = 0.4 chloric-acid corrosion sample is shown in Figure 6. Diffraction intensity shows the appearance of foreign peaks besides the regular peaks, indicating the presence of corrosion byproducts inside the sample; Analysis using the computer code JCPDS (Joint Committee On Powder Diffraction Standard) available in the CD-ROM format, points to the possible presence of solid corrosion byproducts such as iron oxide ( $\beta\text{-Fe}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ), chrome oxide ( $\text{Cr}_2\text{O}_3$ ), iron chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) and nickel hydride oxide ( $\text{Ni}_2\text{O}_3\text{H}$ ).

## CONCLUSION

Concluding from the above discussion and analysis, it could be argued that for commercial AISI-321 alloys undergoing corrosion in an chloride-acid medium with a pH variation of 1.0, 0.7, 0.523, and 0.4 the corrosion effect with measured corrosion values of only 0.0312, 0.0907, 1.85, and 2.5

mpy respectively is small according to Fontana's criteria. Therefore, judging by the Fontana's criteria these test-specimens have been proven to exhibit an excellent resistance toward corrosion when immersed in an chloride acid environment. The ensuing type of corrosion is erosion corrosion preceded by pitting corrosion. Corrosion rate will continue to decrease with respect to corrosion medium pH values; On the other hand, corrosion potential and passivation potential tend to increase with respect to the corrosion medium pH values. Potentiodynamic measurements yield the following potentials for pH value of 1.0, 0.7, 0.522, and 0.4 are -198, -214, -465, and -445 mV respectively. The passivation potential for pH value of 1.0, 0.7, 0.522, and 0.4 are +70, +100, -400, and -400 mV respectively. EDS and X-ray diffraction results reveal that the corrosion byproducts are possibly chrome oxide, iron oxide, iron chloride, and nickel hydride oxide.

## ACKNOWLEDGEMENT

The authors would like to express their gratitude to Mr. Gunandjar, the former head of P3IB-BATAN, for his valuable financial support for this research project fiscal year 2005, so that this programme could be carried out. Gratitude is also due to Dr. Mohammad Dani, the former head of BBI-P3IB. Finally, the authors would also like to express their gratitude to Dr. Ridwan, the head of PTBIN-BATAN, for his continuing support and a conducive environment, so that this research project could be continued into the 2006 fiscal year.

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