

UNIVERSITI MALAYSIA PAHANG

BORANG PENGESAHAN STATUS TESIS ♦

JUDUL: EFFECT OF HEAT TREATMENT ON EROSION-CORROSION

SESI PENGAJIAN: 2011/2012

Saya,

AHMAD SYAZWAN BIN YAHYA (900709-11-5205)
(HURUF BESAR)

mengaku membenarkan tesis (Sarjana Muda / ~~Sarjana~~ / ~~Doktor Falsafah~~)* ini disimpan di perpustakaan dengan syarat-syarat kegunaan seperti berikut:

1. Tesis ini adalah hakmilik Universiti Malaysia Pahang (UMP).
2. Perpustakaan dibenarkan membuat salinan untuk tujuan pengajian sahaja.
3. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (✓)

☐

SULIT

(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia seperti yang termaktub di dalam AKTA RAHSIA RASMI 1972)

☐

TERHAD

(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi / badan di mana penyelidikan dijalankan)

☒

TIDAK TERHAD

Disahkan oleh:

(TANDATANGAN PENULIS)

(TANDATANGAN PENYELIA)

Alamat Tetap:

808-C Kg. Jeram, Manir,
21200 Kuala Terengganu,
Terengganu.

Pn. Nor Imrah binti Yusoff
(Nama Penyelia)

Tarikh:

Tarikh:

CATATAN: * Potong yang tidak berkenaan.

** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali tempoh tesis ini perlu dikelaskan sebagai SULIT atau TERHAD.

♦ Tesis dimaksudkan sebagai tesis bagi Ijazah Doktor Falsafah dan Sarjana secara Penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Laporan Projek Sarjana Muda (PSM).

EFFECT OF HEAT TREATMENT ON EROSION-CORROSION

AHMAD SYAZWAN BIN YAHYA

Report submitted in partial fulfillment of the requirements for the award of Bachelor of
Mechanical Engineering

Faculty of Mechanical Engineering
UNIVERSITI MALAYSIA PAHANG

JULY 2012

UNIVERSITI MALAYSIA PAHANG
FACULTY OF MECHANICAL ENGINEERING

I certify that the project entitled “Effect of Heat Treatment on Erosion-Corrosion” is written by Ahmad Syazwan bin Yahya. I have examined the final copy of this project and in my opinion; it is fully adequate in terms of scope and quality for the award of the degree of Bachelor of Engineering. I herewith recommend that it be accepted in partial fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering.

Mr. Luqman Hakim Bin Ahmad Shah

Examiner

Signature

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature :

Name : PN.NOR IMRAH BINTI YUSOFF

Position : LECTURER OF MANUFACTURING ENGINEERING

Date :

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature :

Name : AHMAD SYAZWAN BIN YAHYA

Matric Id : MA08064

Date :

DEDICATION

*I specially dedicate to my beloved parents, my siblings,
My supervisor and those who have guided
And motivated me for this project*

ACKNOWLEDGEMENT

In the name of Allah S.W.T the most gracious and merciful, first and foremost, after a year of struggle and hard work, with His will, this thesis is completed. Thanks to Allah for giving me the strength to complete this project and the strength to keep on living. I would like to convey heartiest appreciation to my supervisor, Pn. Nor Imrah binti Yusoff for his consistency, advising and giving ideas throughout this thesis. I appreciate his consistent support from the first day I start doing the thesis.

My sincere thanks go to all my lab mates and members of the staff of the Mechanical Engineering Department, UMP, who helped me in many ways and made my thesis work pleasant and unforgettable. Many special thanks go to instructor engineer and assistance instructor for their excellent co-operation, inspirations and supports during this study.

To my parents, thank you for all the support they gave me. Your sacrifice is too great to be measured. It will be never forgotten. Also to my siblings and friends who gives fully support in making these thesis.

Last but not least, to all people that involved directly nor indirectly in succeeded the thesis, I cannot find appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals. Hopefully, they will continue to support me and thanks for making this possible to happen.

ABSTRACT

Erosion-corrosion is complex phenomenon which involves the interaction between mechanical process of solid particle erosion and electrochemical process of corrosion. This thesis is investigates the effect of heat treatment on erosion-corrosion. The aims of this project are to investigate the effect of heat treatment on erosion-corrosion of stainless steel in aqueous slurries. Specimens of AISI Type 301 Stainless Steel been annealed heat treated to 1070 °C with slowly cooling type. Two types of specimens which are annealed and control specimens of AISI Type 301 Stainless Steel were prepared to evaluate the erosion-corrosion affected. Erosion-corrosion test then were carried out by immersing those specimens in aqueous 3.5% NaCl solution and presence of 10% sand particles. Velocity, temperature and concentration in aqueous were stated as fixes as we maintain to focus the effect of heat treatment. Erosion-corrosion test undergo specifically was electrochemical test and Potentiostat was choosing as the tools to analyzed corrosion potential rate. Annealed AISI Type 301 Stainless Steel show increased in corrosion rate (mmpy) than control AISI Type 301 Stainless Steel due to decreasing of steel hardness (HRC) after being annealed.

ABSTRAK

Hakisan-kakisan adalah fenomena kompleks yang melibatkan interaksi antara proses mekanikal hakisan zarah pepejal dan proses elektrokimia kakisan. Tesis ini adalah mengenai kesan rawatan haba terhadap proses hakisan-kakisan. Matlamat projek ini adalah untuk mengkaji kesan rawatan haba terhadap hakisan-kakisan keluli tahan karat dalam larutan akueus. Spesimen AISI 301 keluli tahan karat menjalani proses rawatan haba dengan dipanaskan kepada suhu 1070 ° C dan process penyejukan secara perlahan-lahan . Tujuan utama proses penyepuh lindapan adalah untuk melembutkan keluli. Dua jenis spesimen iaitu disepuh lindap dan spesimen kawalan AISI Jenis 301 tahan karat sedia untuk dianalisis terhadap kesan proses hakisan-kakisan . Ujian hakisan -kakisan kemudiannya telah dijalankan dengan merendamkannya spesimen tersebut dalam larutan akueus 3.5% NaCl dan dengan kehadiran 10% zarah pasir. Halaju, suhu dan kepekatan dalam akueus adalah tetap untuk memberi tumpuan kepada kesan rawatan haba. Ujian hakisan-kakisan menjalani ujian elektrokimia dan Potentiostat telah dipilih sebagai alat untuk menilai kadar potensi kakisan yang dianalisis. Jenis disepuh lindap AISI 301 keluli tahan karat menunjukkan peningkatan dalam kadar hakisan-kakisan (mm/tahun) daripada jenis kawalan AISI 301 keluli tahan karat disebabkan penurunan nilai kekerasan keluli (HRC) selepas disepuh lindap.

TABLE OF CONTENTS

	Page
EXAMINER DECLARATION	ii
SUPERVISOR'S DECLARATION	iii
STUDENT'S DECLARATION	iv
DEDICATION	v
ACKNOWLEDGEMENTS	vi
ABSTRACT	vii
ABSTRAK	viii
TABLE OF CONTENTS	ix
LIST OF TABLES	xiii
LIST OF FIGURES	xiv
LIST OF SYMBOLS	xvi
LIST OF ABBREVIATIONS	xvii

CHAPTER 1 INTRODUCTION

1.1	Introduction	1
1.2	Project background	2
1.3	Problem statement	3
1.4	Project objective	3
1.5	Scope of the project	3

CHAPTER 2 LITERATURE REVIEW

2.1	Introduction	4
2.2	Erosion-corrosion	5
	2.2.1 Erosion-corrosion process	5
	2.2.2 Erosion-corrosion mechanism	7
	2.2.3 Applications constraints and protecting approaches	7

2.3	Types of corrosion	8
2.3.1	Pitting corrosion	8
2.3.2	Intergranular corrosion	10
2.4	Corrosion in seawater	11
2.5	Heat treatment	12
2.5.1	Stages of heat treatment	12
2.5.2	Types of heat treatment	13
2.5.2.1	Annealing	13
2.5.3	Quenching media	14
2.6	Preparation and test mechanical properties for specimens	14
2.7	Electrochemical techniques in corrosion testing	16
2.7.1	Electrochemical polarization	17
2.7.2	Tafel extrapolation	17
2.8	Passivity	19
2.9	Austenitic stainless steels	20
2.10	Conclusion	21

CHAPTER 3 METHODOLOGY

3.1	Introduction	22
3.2	Preparation of specimens	26
3.2.1	Design of specimens	27
3.2.2	Reduce length and diameter	28
3.2.3	Surface finishing	28
3.2.4	Composition analysis	29
3.2.5	Cold mounting process	31
3.2.6	Surface polishing	32

3.3	Heat treatment process	33
3.4	Inspection of erosion-corrosion parameter	36
3.4.1	Microstructure analysis	36
3.4.2	Surface roughness test	37
3.4.3	Rockwell hardness test	38
3.5	Electrochemical test and measurement	39
3.5.1	Electrochemical cell set-up	40
3.5.2	General parameter	43
3.6	Corrosion rate analysis	44
3.6.1	Tafel plot	44

CHAPTER 4 RESULTS AND DISCUSSION

4.1	Introduction	46
4.2	Hardness test analysis	46
4.3	Corrosion rate analysis	48
4.4	Sample characterization	56
4.5	Surface roughness, Ra analysis	64

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

5.1	Introduction	66
5.2	Conclusion	66
5.3	Recommendation	67

REFERENCES

68

APPENDICES

70

A Gantt Chart /Project Schedule FYP 1

70

B Gantt Chart /Project Schedule FYP 2

71

LIST OF TABLES

No.	Title	Page
2.1	Passive region	19
3.1	Classified of specimens type	27
3.2	Composition of AISI Type 301 stainless steel	30
3.3	Approximate periods for annealing steel	34
4.1	Corrosion rates determine by Tafel Extrapolation Method	56
4.3	Specimens surface roughness value before and after electrochemical Erosion-corrosion test	65

LIST OF FIGURES

No.	Title	Page
2.1	Process of erosion-corrosion	6
2.2	(a) An example of general deep pitting corrosion with some pits joining to form larger pits and interconnected pitting , (b) Pitting corrosion in stainless steel backscattered SEM image	9
2.3	Intergranular Corrosion, Sensitized 304 stainless steel exhibiting intergranular attack, 100X magnification	11
2.4	(a) Detail of the home-built slurry wear testing machine used for corrosion–erosion experiments. (b) SEM image showing the morphology of SiO ₂ particles used in the tests.	15
2.5	Polarization diagram	18
2.6	Passivity diagram	20
3.1	Flow chart FYP	23
3.2	Experiment setup	25
3.3	Lathe turning machine to reduce and slice raw material to specimen	28
3.4	Precision surface grinder	29
3.5	Top surface finishing	29
3.6	Spark emission spectrometer	30
3.7	Cold mounting machine	31
3.8	Surface of specimen to undergoes erosion-corrosion test	32

3.9	Three grades of polish type	33
3.10	Furnace for heat treatment process	34
3.11	Program controller setting for furnace to heat and soaking specimens at 1070 ° C	35
3.12	Specimens undergoes heating process in furnace	35
3.13	Image analyser	36
3.14	Surface roughness Perthometers	37
3.15	Rockwell hardness test	38
3.16	Result for HRC appears on screen after load applied	39
3.17	Electrochemical test cell	40
3.18	Working electrodes connected to Wonatech Potentiostat (WPG100)	41
3.19	Wonatech Potentiostat (WPG100)	42
3.20	Electrochemical test set-up	42
3.21	General Potentio dynamic parameters from Potentiostat WPG100 for IVMan software	43
3.22	Tafel plot	44
3.23	Tafel analysis using IVMan software	45
4.1	Specimen A1 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel	50
4.2	Specimen A2 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel	51

4.3	Specimen A3 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel	52
4.4	Specimen B1 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless Steel	53
4.5	Specimen B2 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless Steel	54
4.6	Specimen B3 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless	55
4.7	Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A1; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	58
4.8	Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A2; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	59
4.9	Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A3; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	60
4.10	Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070° specimen B1; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	61
4.11	Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070°C specimen B2; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	62
4.12	Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070°C specimen B3; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test	63
4.13	Graph bar for increment of surface roughness ,Ra	64

LIST OF SYMBOLS

NaCl	Sodium Chloride
β_a	Anodic Slopes
β_c	Cathodic Slopes
Cr	Chromium
E_{corr}	Corrosion Potential
E_p	Primary Passivation Potential
I_c	Critical Current Density
I_p	Passive Current Density
LogI	Log Current
%	Percentage
g	Weight
s	Time
g/cm^3	Density
$^{\circ}C$	Temperature

LIST OF ABBREVIATIONS

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
SEM	Scanning electron microscopy
FYP	Final Year Project
UMP	University Malaysia Pahang
WE	Working electrode
RE	Reference electrode
RP	Corrosion resistance
CE	Counter electrode

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Erosion-corrosion is complex phenomenon which involves the interaction between mechanical process of solid particle erosion and electrochemical process of corrosion. A whole range of issues is faced by designer and engineer when trying to obtain relevant information on erosion-corrosion performance of a material.

Erosion-corrosion can be simplified as acceleration in the rate of corrosion attack in metal due to the relative motion of a corrosive fluid and a metal surface. The increase turbulence caused by pitting on the internal surface of a tube can result in rapidly increasing erosion rates. This erosion-corrosion is associated with a flow-induced mechanical removal of protective surface film that results in a subsequent corrosion rate increase via electrochemical process. The mechanical damage by the impacting fluid imposed disruptive shear stresses on the material surface. Erosion-corrosion may be enhanced by sand particles effect and impacted by multiphase flows. The morphology of surfaces affected by erosion-corrosion may be in the form of shallow pits related to the flow direction.

Wear caused by solid particle erosion-corrosion cause the many problem to components in service, resulting in material loss and subsequent equipment failure. Most erosion-corrosion related problems occur in power plants, oil and gas processing plants and chemical plants. The problem has been reported to affect equipment for examples pipelines, valves, heat exchangers, pressure vessels and various rotating equipment namely compressors, turbines and pumps.

To archive more wear resistant of erosion-corrosion, many works concern of annealing heat treatments were undergoes as they can change the morphology of the metals and alloys which effect the properties of these metal. As example, inter-critical annealing heat treatments for reinforcing steel have illustrated a good corrosion resistance.

1.2 PROJECT BACKGROUND

Effect of heat treatment to erosion-corrosion material behavior could be important thing to elaborate in further study which aqueous-slurries are the corrosive environment.

Erosion-corrosion is the acceleration or increase in deterioration or attack on a metal because of relative movement between a corrosive fluid and metal surface. Erosion-corrosion test were carried out after materials been heat treated by immersing the materials in the aqueous solution and presence of sand particles. In each case, pure corrosion and combined corrosion effects were studied by practically protecting the materials from the impact of solid particles during the test. Erosion-corrosion was determined by corrosion rate of Potensiostat WPG 100, erosion-corrosion parameter and microstructural studies by light microscopy.

1.3 PROBLEM STATEMENT

In recent years, study of erosion-corrosion has become significant. A lot of time and money were spent to study of this erosion-corrosion. All these study are aimed to explore the behavior of material due to effect that can evaluate the corrosion rate of material. Certain industries apply heat treatment to manipulate useful of material like high temperature welding, structural application, chemical tanks, pressure vessels and quite applications in industries are ready enough to wear erosion-corrosion environment that can costly to money and safety.

1.4 PROJECT OBJECTIVE

The objectives of this study are:

- (i) Study the erosion-corrosion behavior on stainless-steel.
- (ii) Investigate the effect of heat treatment on erosion-corrosion of stainless-steel in aqueous slurries.

1.5 SCOPE OF THE PROJECT

The scopes of this study include:

- (i) Specimens used was AISI Type 301 Stainless-Steel
- (ii) Investigation covers heat treatment process, electrochemical erosion-corrosion testing and inspection parameter involved.
- (iii) Annealed specimens at 1070 °C in furnace and slowly cooling rate by air.
- (iv) Electrochemical erosion-corrosion testing in aqueous slurries of 3.5% NaCl and presences of 10% sand particles with erosion by magnetic stirrer.
- (v) Result elaborated from hardness test, microstructural study, corrosion rate and surface roughness testing.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The single most important property of stainless steels, and the reason for their existence and widespread use, is their corrosion resistance. Before looking at the properties of the various stainless steels, a short introduction to corrosion phenomena is appropriate. In spite of their image, stainless steels can suffer both "rusting" and corrosion if they are used incorrectly.

When steel need to be bending or rolling, apply heat treatment to soften or change the structure can affect to the steel corrosion behaviours. Certain effect from welding also can affect to corrosion of steel. The effects of heat treatment study to determine how it's supposed to being effect to the corrosion rate in certain environment. As the infrastructure of industrialized countries continues to age, more and more failures due to corrosion are occurring. As examples, replacing all the bridge and pipeline would be prohibit expensive and the study of material involve maybe can come out with good outcome at the end.

2.2 EROSION-CORROSION

Erosion-corrosion is acceleration in the rate of corrosion attack in metal due to the relative motion of a corrosive fluid and a metal surface. The increased turbulence caused by pitting on the internal surfaces of a tube can result in rapidly increasing erosion rates and eventually a leak. Erosion-corrosion can also be aggravated by faulty workmanship. For example, burrs left at cut tube ends can upset smooth water flow, because localized turbulence and high flow velocities, resulting in erosion-corrosion. A combination of erosion and corrosion can lead to extremely high pitting rates.

In simply view state that erosion-corrosion is a general term that refers to a corrosion process enhanced by the action of flowing fluids. The process can also be characterized by whether the fluid contains solid particles, is in the form of impinging droplets, or is undergoing cavitation. Cavitation is the formation and sudden collapse of vapor bubbles in a liquid. (William F.Smith, 2006)

2.2.1 Erosion-corrosion process

Erosion-corrosion can result in general corrosion that occurs at a higher rate than would be expected under stagnant conditions. In this case the measure of erosion-corrosion would be the rate of surface waste or the rate of penetration. The more usual effect of erosion-corrosion, however, is localized attack, which can appear as grooves, gullies, waves, rounded holes and usually exhibits a directional pattern that correlates with the direction of flow. Figure 2.1 showed the process of erosion-corrosion mechanism.

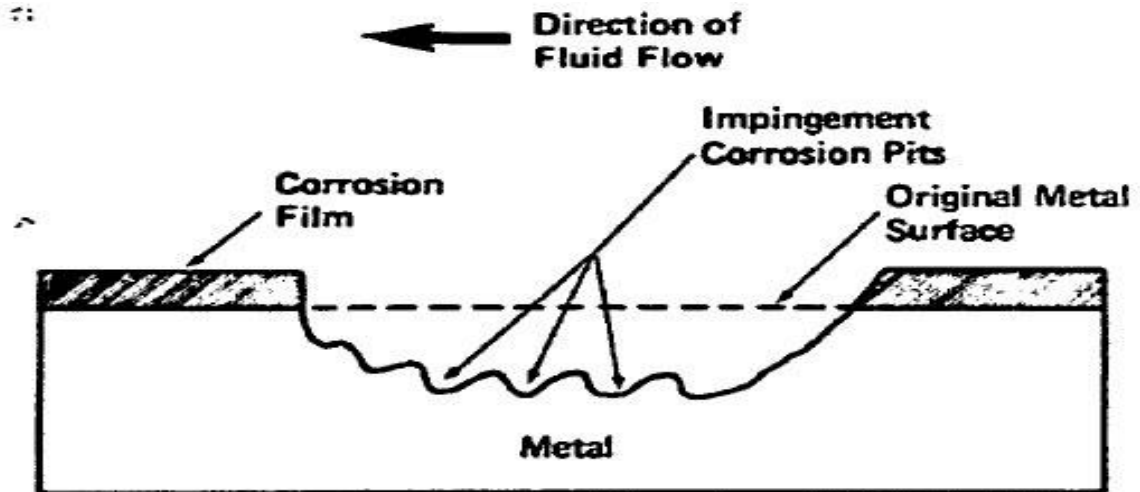


Figure 2.1: Process of erosion-corrosion

(Source: Pierre R. Roberge, Corrosion Testing Made Easy: Erosion-Corrosion)

The appearance of cavitation damage is similar to pitting. However, the pitted areas are closely spaced and the surface is usually considerably roughened. Severe cavitation damage can completely remove sections of metal. Cavitation damage that results from fluid movement relative to the metal surface can usually be correlated with the direction of fluid flow. A condition conducive to erosion-corrosion is the flow of corrosive fluids (gas or liquid) relative to a metal surface. The rate of corrosion depends upon the flow rate of the fluid. Turbulent flow results in much higher corrosion rates than Laminar flow. Hard particles entrained in the flowing fluid can enhance erosion-corrosion. Cavitation damage is caused by flow discontinuities that result in the formation and subsequent collapse of vapor bubbles on a metal surface. High-velocity drop impingement, such as raindrops on a helicopter rotor, can also result in rapid surface damage.

2.2.2 Erosion-corrosion mechanism

Erosion-corrosion implies that the fluid medium is potentially corrosive to the metal. Erosion facilitates the corrosion process. This fact distinguishes erosion-corrosion from pure erosion or mechanical wear. Erosion corrosion influences the rate of corrosion by changing the conditions of local cell action. The corrosion process is accelerated if the fluid speed is sufficient to remove weakly adhered corrosion products from the surface. Removal of these products reduces their polarizing or inhibitive effect.

At the breakaway speed the fluid begins to remove the corrosion film and the corrosion rate increases. A steady corrosion rate is achieved at the speed at which the film is completely removed. Fluid flow also maintains a uniform concentration of corrosion at the metal surfaces. Impingement of suspended hard particles can accelerate the damage to the protective film and can cause mechanical damage to the underlying metal.

2.2.3 Application Constraints and Protection Approaches

There are several methods for preventing or minimizing damage resulting from erosion-corrosion is available. Material selected can be the method which use or choose the material with better resistance to erosion-corrosion. Most of them were design consideration where we can conduct to streamline the flow, avoid design that create turbulence, minimize abrupt changes in flow direction. Introduce smooth aerodynamic or hydrodynamic flow channels which avoid roughly textured surfaces. For the application of pipeline be careful on align pipe sections. Avoid flow obstructions in design or obstructions that can arise under operations, increase the thickness of material in vulnerable areas, install renewable impingement plates or baffles, and design for easy repair by using interchangeable parts. Cathode protection also can be helpful.

2.3 TYPES OF CORROSION

All corrosive attacks begin on the surface of the metal making the classification of corrosion by physical appearance a convenient of identification. Erosion-corrosion depends on how pitting corrosion forms and also come with intergranular corrosion.

2.3.1 Pitting corrosion

Pitting corrosion is one of the most destructive and intense forms of corrosion. It can occur in any metal but is most common on metals that form protective oxide films, such as aluminum and magnesium alloys. It is first noticeable as a white or gray powdery deposit, similar to dust, which blotches the surface. When the deposit is cleaned away, tiny holes or pits can be seen in the surface. (See Figures 2.2(a) and 2.2(b).) These small surface openings may penetrate deeply into structural members and cause damage completely out of proportion to its surface appearance.

Localized attack in resistant surface produces pitting corrosion. Stainless steel and titanium alloys with chromium depend on a passive film for corrosion resistances are susceptible to pitting by local breakdown of the film at isolated sites. Pitting is a form of localized corrosion that proceeds because of local cell action which produces cavities beginning at the surface. Pitting usually occurs on metal surface immersed in a solution or moist environment. It can occur on a surface exposed to the atmosphere if there are droplets of moisture or a condensed moisture film present on the metal surface. Sometimes, pitting may associate with intergranular corrosion. Pits develop at weak spots in the surface film and at sites where the film is damaged mechanically under conditions where self-repair will not occur.



Figure 2.2 (a): An example of general deep pitting corrosion with some pits joining to form larger pits and interconnected pitting.

(Source: Pipeline Corrosion and Cracking and the Associated Calibration Considerations for Same Side Sizing Applications by Ginzel, R.K. & Kanter, W.A.)



Figure 2.2 (b): Pitting corrosion in stainless steel backscattered SEM image

(Source: ATC Labs Argo - Tech Materials Laboratory webmaster@atclabs.com)

The importance of pitting significantly depends on the nature of the surface layer or the film that has formed on the surface due to the interaction of the material with the environment. Thus a state of “passivity” is forced on the material, which safeguards the material from general corrosion by slowing down the dissolution process at the surface.

Pitting occurs when the anodic site becomes fixed on a small part of the surface due to a break in the passive layer, irregularities in the surface caused due to variations in the metal itself, incomplete coatings, scaling deposits that build up at selected areas, etc. The cathodic area can be anywhere outside the pit. This results in a high corrosion current density at the base of the pits. Movement of metal ions or HC ions from the bottom of the pit is restricted by the film covering the top of the pit (Fontana , 1987). The depletion of oxygen creates a difference in electrochemical potential between the pit and the surrounding metal.

2.3.2 Intergranular corrosion

Intergranular corrosion is an attack on the grain boundaries of a metal. A highly-magnified cross section of any commercial alloy shows the granular structure of the metal. The grain boundary and the grain center can react with each other as anode and cathode when in contact with an electrolyte. (See Figure 2.3.) In other way to define Intergranular corrosion is the preferential corrosion of material at the grain (crystal) boundaries. Though the loss material will be small, intergranular corrosion can cause the catastrophic failure of equipment. Intergranular corrosion is a common form of attack on alloys but occurs rarely with pure metals. The attack is usually caused by a differential couple being set up between impurities existing at the grain boundary. The classic example of intergranular corrosion in chemical plant is the weld decay of unstabilised stainless steel.

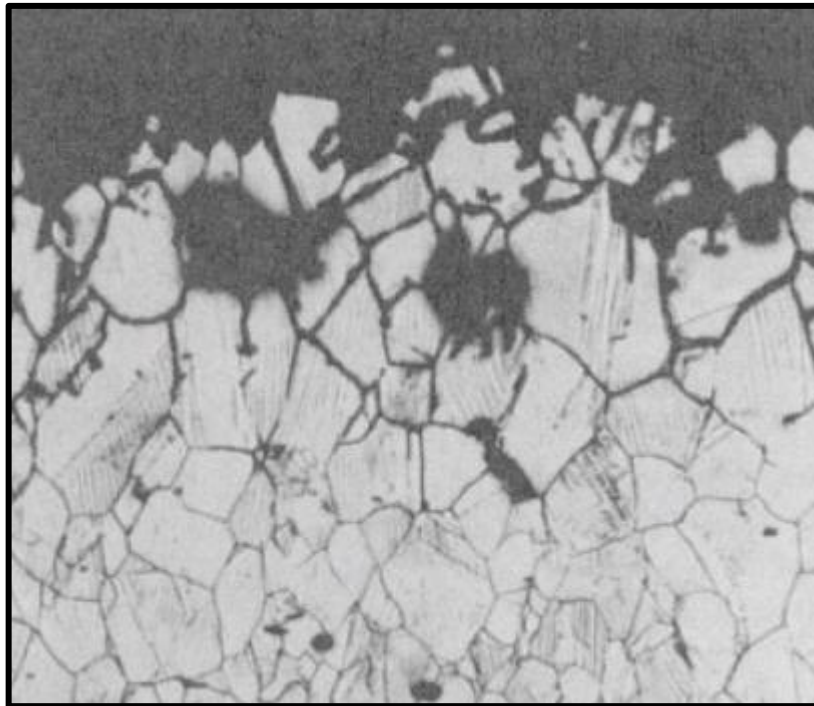


Figure 2.3: Intergranular Corrosion, Sensitized 304 stainless steel exhibiting intergranular attack, 100X magnification

(Source: Corrosion Morphology, <http://abduh137.wordpress.com>)

2.4 CORROSION IN SEAWATER

As aqueous slurries used in these experiment was 3.5% of NaCl where it's relate to the environment seawater or solution relate to it. Seawater is used by many industries, such as shipping, offshore oil and gas production, power plants and coastal industrial plants. Most of the elements that can found on earth are present in seawater. However, 11 of the constituents account for 99.95 percent of the total solutes, as indicated with chlorine ions being the largest constituent. Seawater typically contains about 3.5 percent sodium chloride, although the salinity may be weakened in some area by dilution with fresh water or concentrated by solar evaporation. (Zaki Ahmad, 2006)

Seawater is normally more corrosive than fresh water because of the higher conductivity and penetrating power of the chlorine ion through surface films on a metal. The rate of the corrosion is controlled by the chlorine content, oxygen availability and the temperature.

2.5 HEAT TREATMENT

The process of heat treating is the method by which metals are heated and cooled in a series of specific operations that never allow the metal to reach the molten state. The purpose of heat treating is to make a metal more useful by changing or restoring its mechanical properties. Through heat treating, we can make a metal harder, stronger, and more resistant to impact. Also, heat treating can make a metal softer and more ductile.

The various types of heat-treating processes are similar because they all involve the heating and cooling of metals; they differ in the heating temperatures and the cooling rates used and the final results. The usual methods of heat-treating ferrous metals (metals with iron) are annealing, normalizing, hardening, and tempering. Most nonferrous metals can be annealed, but never tempered, normalized, or case-hardened. (Heat Treatment and Inspection of Metals, ATC Manual 52-5, Air Training Command, Scott Air Force Base, Ill., 1963)

2.5.1 Stages of heat treatment

Heat treating is accomplished in three major stages which stage 1 start heating the metal slowly to ensure a uniform temperature. Continue with the stage 2 which soaking or holding the metal at a given temperature for a given time. Lastly, stage 3 consists of cooling the metal to room temperature.

The primary objective in the heating stage is to maintain uniform temperatures. If uneven heating occurs, one section of a part can expand faster than another and result in distortion or cracking. Uniform temperatures are attained by slow heating.

The heating rate of a part depends on several factors. One important factor is the heat conductivity of the metal. A metal with a high-heat conductivity heats at a faster rate than one with a low conductivity. Also, the condition of the metal determines the rate at which it may be heated. The heating rate for hardened tools and parts should be slower than unstressed or untreated metals. Finally, size and cross section figure into the heating rate. Parts with a large cross section require slower heating rates to allow the interior temperature to remain close to the surface temperature that prevents warping or cracking. Parts with uneven cross sections experience uneven heating; however, such parts are less apt to be cracked or excessively warped when the heating rate is kept slow.

2.5.2 Types of heat treatment

There are four basic types of heat treatment are used today. They are annealing, normalizing, hardening, and tempering. While annealing selected as a purposed for heat treatment process for stainless steel, the study were elaborated of annealing procedure.

2.5.2.1 Annealing

In general, annealing is the opposite of hardening, you anneal metals to relieve internal stresses, soften them, make them more ductile, and refine their grain structures. Annealing consists of heating a metal to a specific temperature, holding it at that temperature for a set length of time, and then cooling the metal to room temperature. The cooling method depends on the metal and the properties desired. Some metals are furnace-cooled, and others are cooled by burying them in ashes, lime, or other insulating materials.

Welding produces areas that have molten metal next to other areas that are at room temperature. As the weld cools, internal stresses occur along with hard spot and brittleness. Welding can actually weaken the metal. Annealing is just one of the methods for correcting these problems.

Due to the study of (Firas M.F, 2010) on effect of intermediate annealing on mechanical properties, they found that the hardness of the material increase and decrease depends on annealing temperature and holding time. The study shows an increase in the corrosion resistance of the material due mainly to annealing process.

2.5.3 Quenching media

The cooling rate of an object depends on many things. The size, composition, and initial temperature of the part and final properties are the deciding factors in selecting the quenching medium. A quenching medium must cool the metal at a rate rapid enough to produce the desired results.

Mass affects quenching in that as the mass increases, the time required for complete cooling also increases. Even though parts are the same size, those containing holes or recesses cool more rapidly than solid objects. The composition of the metal determines the maximum cooling rate possible without the danger of cracking or warping. This critical cooling rate, in turn, influences the choice of the quenching medium.

2.6 PREPARATION AND TEST MECHANICAL PROPERTIES FOR SPECIMENS

The experiment set up consists of preparation of specimens, corrosion inspection, and due to experimental study by D.Lopez, 2005 (Effect of particle velocity and impact angle on the erosion corrosion of AISI 304 and AISI 420 stainless steels) where the corrosion-erosion tests were performed in slurry wear testing machine. The slurries were composed of 3.5% NaCl solution and 30% of SiO₂ particles with mean diameter between 0.21 and 0.30 mm. The sample was placed in a container where the impact angle of the slurries against the surface could be adjusted to 30° or 90°. (see Figure 2.4)

Mean impact velocities of 4.5, 6.9 and 8.5 m/s were used and the temperature slurries were between 25 and 28° C. simultaneously with the slurry wear tests,

polarization curves were obtained in a Potentiostat by using a Pt counter electrode and Ag/AgCl reference electrode with a potential scan of 1m V/s and starting potential of – 600 m V. The entire sample cylinder with 9.5mm in diameter and 5mm height were polished and cleaned. From this experimental study, I can elaborate the parameters that I can use in the progress experiment.

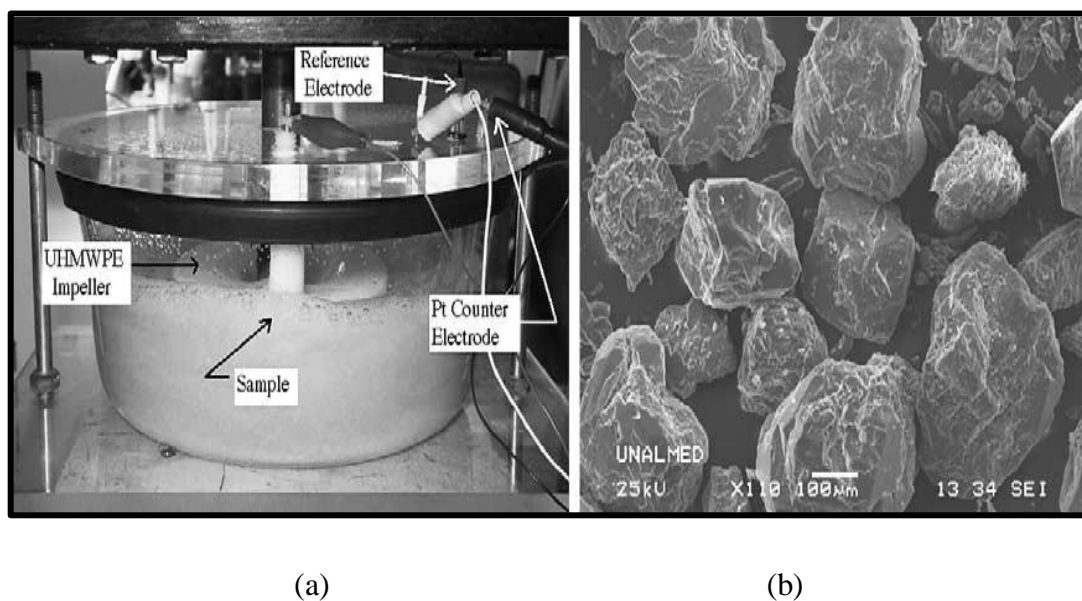


Figure 2.4 : (a) Detail of the home-built slurry wear testing machine used for corrosion–erosion experiments. (b) SEM image showing the morphology of SiO₂ particles used in the tests.

(Source: Effect of particle velocity and impact angle on the erosion corrosion of AISI 304 and AISI 420 stainless steels by D.Lopez, J.P Congote)

2.7 ELECTROCHEMICAL TECHNIQUES IN CORROSION TESTING

Electrochemical methods in corrosion testing have been used ever since the electrochemical nature of corrosion process was discovered. These methods are used both for corrosion monitoring and as laboratory techniques. One of the most important applications has been the estimation of corrosion rate instantaneously by means of polarization resistance. Usually electrochemical techniques have been employed to both speed data development and to better understand corrosion mechanism. ASTM G5 defines polarization as “the change from the open-circuit electrode potential as a result of the passage current”. Polarization method involves changing the potential of the working electrode and monitoring the current which is produce as a function of time or potential.

The important fact why the using electrochemical technique for corrosion measurement is needed because of corrosion is an electrochemical process. Corrosion is the chemical or electrochemical reaction between a material, usually a metal and its environment produces a deterioration of the material and its properties. (ASTM G15: Standard terminology related to corrosion). Fundamentally, corrosion is an electrochemical process, so using electrochemical techniques is obvious. A broad range of electrochemical techniques have been developed specifically for corrosion measurement. While electrochemical techniques are fast than the oldest technics and electrochemical techniques are very sensitive.

Electrochemical techniques are fast while corrosion is an inherently slow process, this technic is important. A typical corrosion rate is 10 mpy or 0.254 mmpy. The best corrosion tests are weigh loss measurements after exposure but these kind of technique was slow take days, weeks or month. Electrochemical instruments polarize the sample to accelerate the corrosion process and make the measurement in minutes or hours.

Electrochemical techniques are sensitive. These techniques can measure very low corrosion rates. Corrosion scientists may use corrosion rate and current interchangeably. The best measure of specific potentiostat's ability to measure low corrosion rates is the lowest current range. Then corrosion measurements are reasonably reproducible. (J.Genesca, 2002)

2.7.1 Electrochemical Polarization

Electrochemical polarization test methods are close enough related for understanding and studying corrosion resistance of materials hence the effect of changes in corrosive environment. The basic tool of dc electrochemical such as cyclic voltammetric, potentiodynamic, potentiostatic, and galvanostatic was the method of investigation. They can perform the criteria for protection of anodic and cathodic protection and susceptibility to several forms of corrosion. By using the methods mentioned above as conjunction, the pitting, crevice, and active-passive of alloy can be easily exhibited. Here, it can describe the performance of the stainless steel with different alloying elements.

By referring to mixed electrodes system where the reactions take place simultaneously at a metal-electrolyte interface, polarization ζ expresses the different between the potential of anodic or cathodic polarization and its corrosion potential.

$$\zeta = E - E_{\text{corr.}} \quad (2.1)$$

If the polarization of $\zeta > 0$, it is anodic current flow, while $\zeta < 0$, it is cathodic current flow.

2.7.2 Tafel Extrapolation

In experimentally creating the polarization diagram, the first measurement is corrosion potential, ϕ_{corr} , when the applied current, I_{appl} is zero. The working electrode has been polarized anodically to establish one of the dash lines in Figure 2.5. The polarization was repeated, but with I_{appl} reversed to obtain the second dash line. The graph of ϕ versus I_{appl} has been plotted on the nobler and the more active sides of the corrosion potential.

The anodic tafel region has been extrapolated from to the equilibrium anode potential, ϕ_A to determine exchange current density, i_{oa} . Similarly, from the tafel region that has been extrapolated to the equilibrium potential, ϕ_c to determine the exchange current density i_{oc} . Hence, the i_{corr} can be determine by extrapolating from either to anodic or cathodic Tafel region to the corrosion potential ϕ_{corr} , where $i_c=i_a$ for condition that $A_a=A_c$ (anode-cathode area ratio=1).

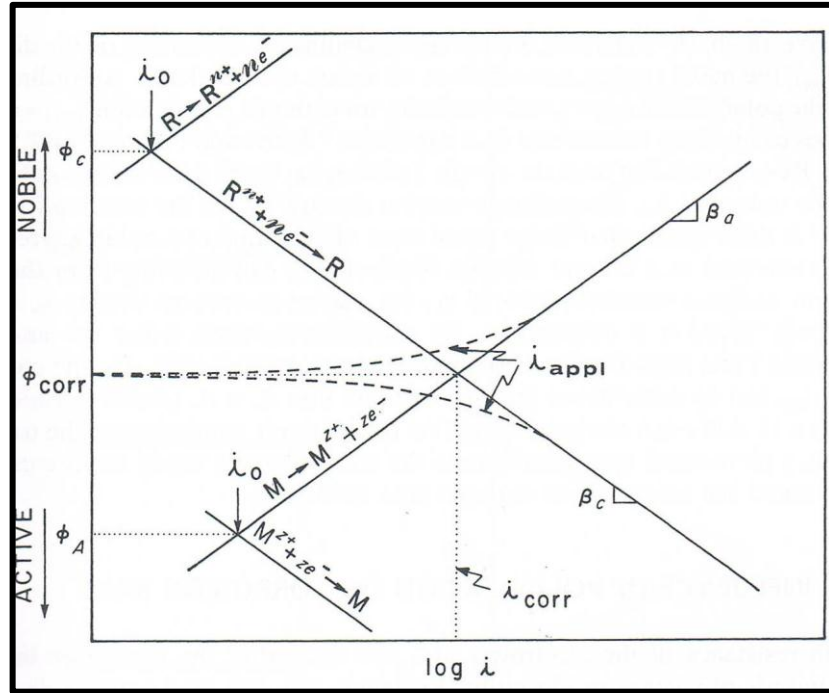


Figure 2.5: Polarization diagram

(Source: Winston, 2008)

The metal can be say in active state when exposed to the new material certain media condition. It tends to be dissolution or attack since it is not in stable naturally or thermodynamically. But, there is also certain tendency to return to its stable state. Therefore, the polarization test is where the corrosion potential and current would be determined by turn specimens into active state. According to ASTM G5, the plots of E_{appl} versus $\log I$ are called Tafel plot. Hence, the current density is determined by using these two equations.

$$E_{appl} = E_{corr} + \beta_a \log \frac{i}{i_{corr}} \quad (2.2)$$

$$E_{appl} = E_{corr} - \beta_c \log \frac{i}{i_{corr}} \quad (2.3)$$

where β_a and β_c are positive constant.

2.8 PASSIVITY

Passivity is refers to the loss of chemical reactivity experienced by certain metals and alloys under particular conditions. It occurs when the spontaneous formation of a hard non-reactive surface film that prevent further corrosion. This layer is usually an oxide or nitride that is a few atoms thick. Table 2.1 and Figure 2.6 show the passivity region. Passivity can be conveniently divided into three regions: active, passive, and trans-passive.

Table 2.1: Passivity region

Regions	Behavior
Active	Identical as normal metal
Passive	Slight increase in the oxidizing power of the solution cause a corresponding rapid increase in the corrosion rate. If more oxidizing agent is added, the corrosion rate shows a sudden decrease
Transpassive	Very high concentrations of oxidizers or in the presence of very powerful oxidizers, corrosion rate again increased

Source: Fontana (1986)

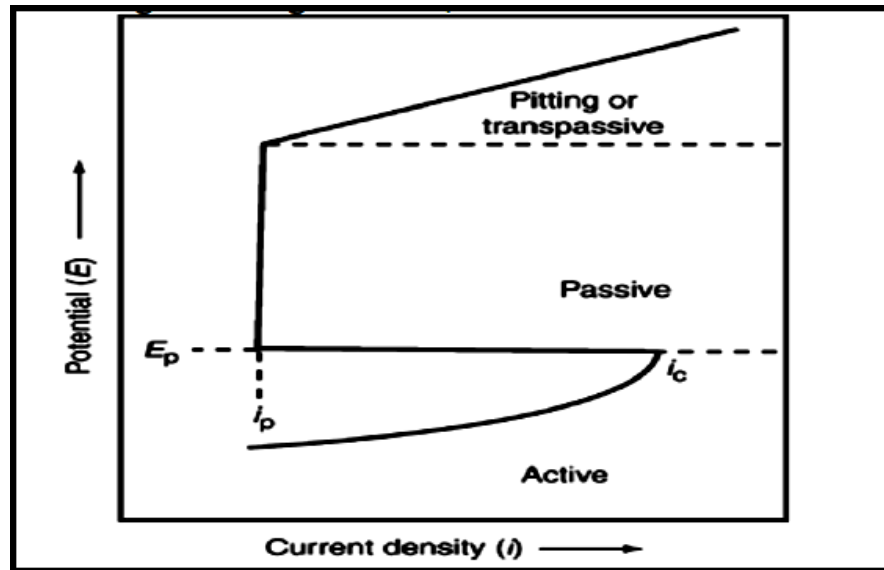


Figure 2.6: Passivity diagram

Source: Thomas (1991)

Figure show the idealized anodic polarization curve for an iron-water system exhibiting passivity. Three different potential regions are shown the active, passive and pitting or Transpassive regions. E_p is potential above which the system become passive and exhibits the passive current density i_c .

Although the thickness of these passive films is typically only a few nanometers, they act as a highly protective barrier between the metal surface and the aggressive biological environment. Consequently, the passive film kinetically retards the rate of dissolution by many orders of magnitude.

2.9 AUSTENITIC STAINLESS STEELS

Stainless –steel are selected as engineering materials mainly because of their excellent corrosion resistance in many environment. The corrosion resistance of stainless steel is due to their high chromium contents. The stainless steel used was classified by AISI the American Iron and Steel Institute has established standards for steel compositions.

Austenitic stainless-steels are essential iron-chromium-nickel ternary alloys containing about 16 to 25 percent Cr and 7 to 20 percent Ni. These alloys are called austenitic since their structure remains austenitic at all normal heat treatment. Austenitic stainless steels normally have better corrosion resistance than ferritic and martensitic ones because carbides can retain solid solution by rapid cooling from high temperature. However if these alloys are to welded or slowly cooled from high temperature through the 870- 600 °C range, they can become susceptible to higher pitting and Intergranular corrosion because chromium contains carbides precipitate at the grain boundaries. This difficulty can be circumvented to some degree either by lowering the maximum carbon content in the alloy to about 0.03 percent C (type 304L alloy) or by adding an alloying element such as columbium to combine with the carbon in alloy.

The maximum temperature to which austenite stainless steel like type 301 can be exposed continuously without appreciable scaling is about 871°C as oxidation resistance.

2.10 CONCLUSION

In conclusion, this chapter has been described briefly about the process needed for this project as well as the useful information to guide the researcher once experiment is carried out. It is very important to know the fundamental of the experiment base on previous research and text book. All information is gathering through this chapter to convenient the investigation of the study and hence, helps the reader to gain understanding toward this project.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

The serious understanding of heat treatment principal and erosion-corrosion law were undergoes while these project were aim to investigate the effect of heat treatment to erosion-corrosion behavior of AISI Type 301 Stainless Steel. Corrosion laboratory has been provided which consist to provide erosion-corrosion environment during these project were conduct. The key of these project was erosion-corrosion testing where the inspection of erosion-corrosion parameter were taken before and after the process. Erosion corrosion test were carried out by immersing the specimen in an aqueous 3.5% NaCl solution and presence of sand particles. This project work on where the data of heat treatment and erosion-corrosion were control. Specimens been prepared as its design follow the ASTM G31-72 (2004) and ASTM G5-94(2004) criteria and then carry out the heat treatment process. Specimen was divided to two parts where parts one, carry on with heat treatment and second part as the control data where no heat treatment apply. Parts one specimen undergoes quenched annealed heat treatment. Then both parts were undergoing erosion-corrosion testing. Flow chart in Figure 3.1 and 3.2 show how these experiment processes start till the end of analysis the results. Certain factors which can manipulate corrosion behavior were fixed as we maintain to focus the effect of heat treatment. Velocity in aqueous, temperature on aqueous and concentration of aqueous were stated as fixed. Those corrosion test undergone specifically was electrochemical test because corrosion is an electrochemical process. Potentiostat was choosing as the tools to analyzed corrosion potential when electrochemical test was happen.

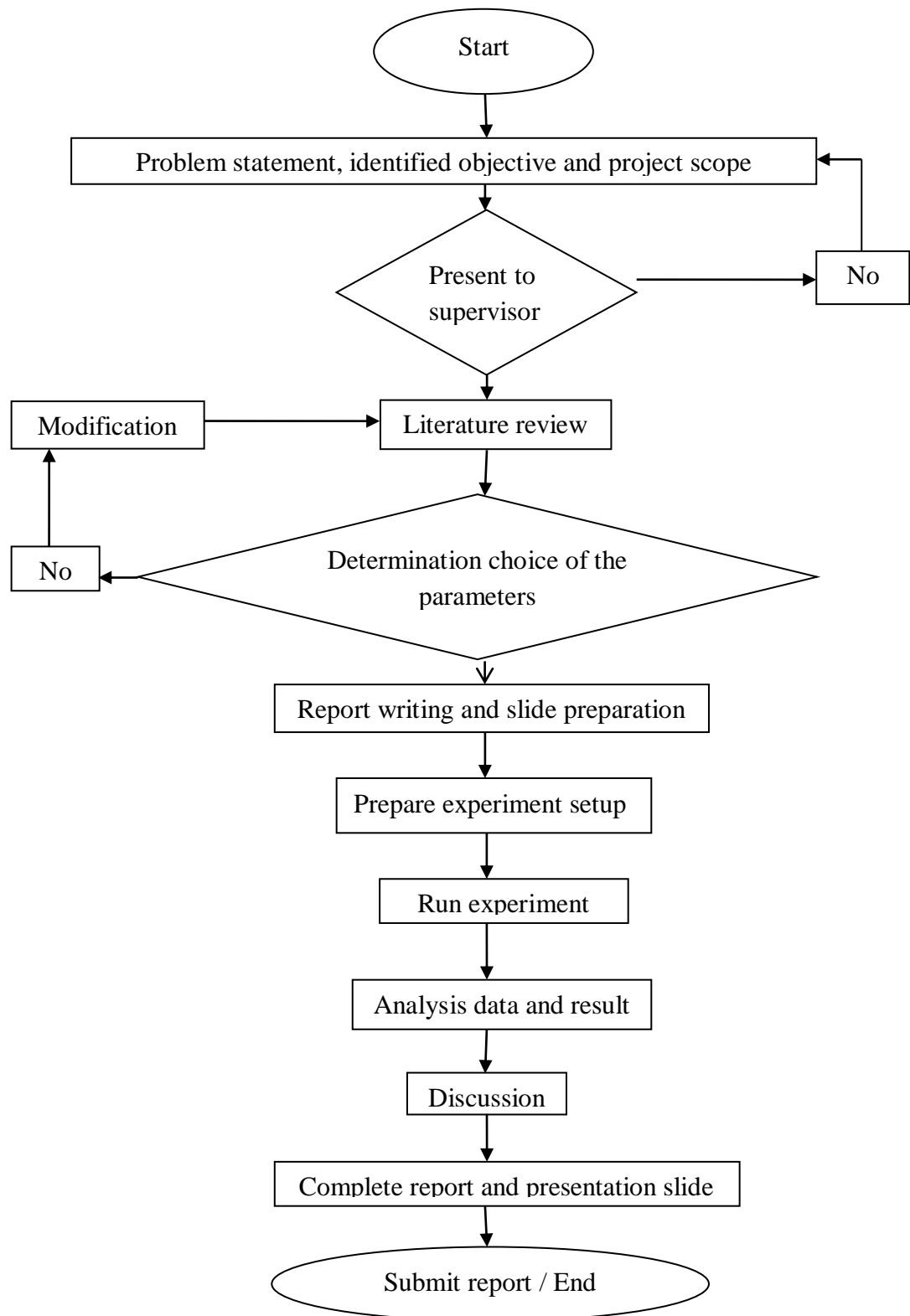
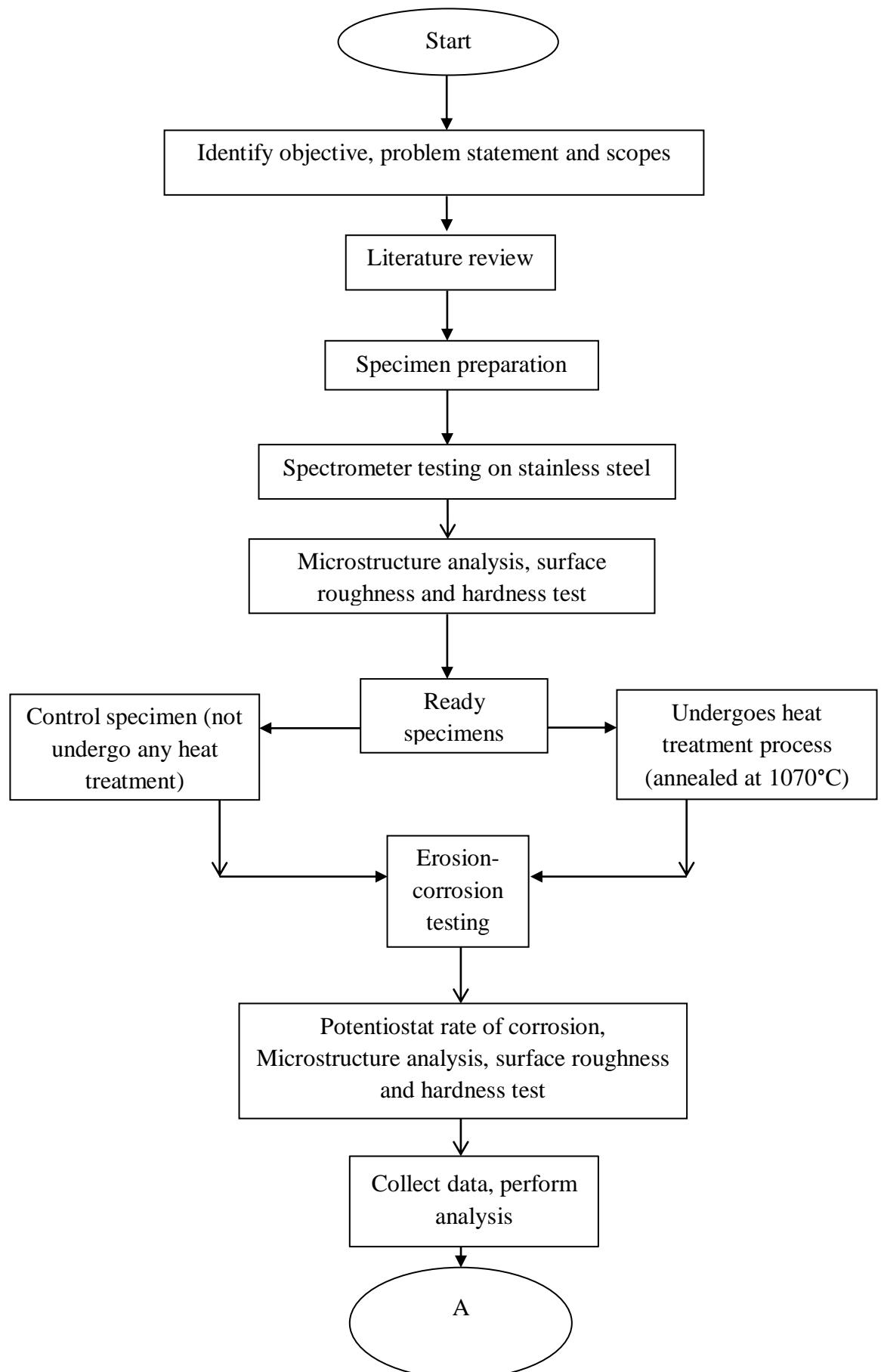


Figure 3.1: Flow chart FYP



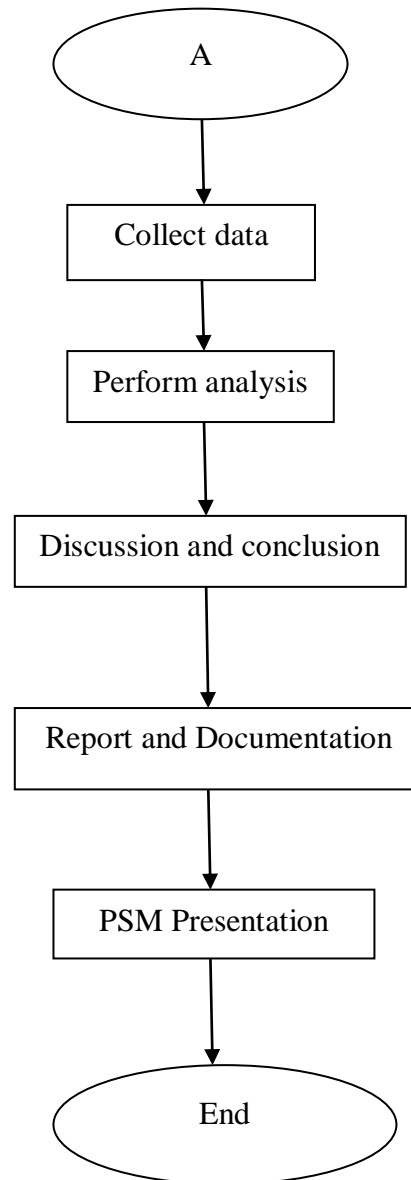


Figure 3.2: Experiment setup

Results output comes from corrosion potential and support to these data output, we conclude to take due to inspection of corrosion parameter which roughness test, hardness test and microstructure analysis before and after erosion-corrosion test were conducts. There is flow chart as shown in figure where represent the planning for the whole project.

3.2 PREPARATION OF SPECIMENS

This corrosion laboratory was prepared to analyze surface properties and change in microstructure of stainless steel after being quench annealed and corrosion test. As the stainless steel has been choose as tested material in this project, preparation of stainless-steel has be work on based criteria-criteria needed. There are step from first raw material been collected until the specimen can be used in erosion-corrosion test. The designing of specimen comes as the specification of small area of corrosion environment could happen and there were be agree to design small type of specimen. There is flow during preparation of specimen

Collected raw stainless steel —————> decreased to needed diameter and slice into no of specimen needed using lathe turning machine —————> surface finishing using precision surface grinder —————> composition analysis —————> cold mounting process —————> surface polishing after been mounting

3.2.1 Design of specimen

Specimen as mention been design to 15mm in diameter and 5 mm in length. There would be 6 piece of specimens will used consist of differ type of control data as in shown in Table 3.1. Raw stainless-steel were collected and was cut using a band saw machine. As the raw material shape come as hollow bar so we agree to going-trough to that shape for specimen to making it easier in step.

Table 3.1: Classified of specimens type

Specimens	Specification
A1	Specimen 1 for control the data taken (AISI Type 301 Stainless Steel)
A2	Specimen 2 for control the data taken (AISI Type 301 Stainless Steel)
A3	Specimen 3 for control the data taken (AISI Type 301 Stainless Steel)
B1	Specimen 1 for effect of heat treatment (annealed AISI Type 301 Stainless Steel)
B2	Specimen 2 for effect of heat treatment (annealed AISI Type 301 Stainless Steel)
B3	Specimen 3 for effect of heat treatment (annealed AISI Type 301 Stainless Steel)

3.2.2 Reduce length and diameter

Raw stainless-steel cutting was in 30 mm diameter while to reduce as become 15 mm and slice into 5mm length each it could be practically way using lathe turning machine (Figure 3.3). Working on turning and facing need set cutting speed to suitable value cause of small specimen need. So we set to low speed which 425 rpm to make sure the specimen not bending while reducing diameter.



Figure 3.3: Lathe turning machine to reduce and slice raw material to specimen

3.2.3 Surface finishing

Provided a fine surface of specimens was a needed while working on corrosion test. After reducing and slice into a shape of specimens, we undergo the specimen surface finishing. While we just needed top fine surface, they were only grind only on top of specimen surface. Precision surface grinder machine was used to make sure we have fine surface finishing and the use of coolant protect the specimen from heat treated effect. (See Figure 3.4 and 3.5)



Figure 3.4: Precision surface grinder



Figure 3.5: Top surface finishing

3.2.4 Composition analysis

A spark emission spectrometer was used to analysis the composition of the stainless-steel before further on to the testing method. The analysis to analyze and check the composition of stainless-steel weather it was martensitic, austenite or ferrite stainless steel. Then when all those composition been analyze there were be to agree which stainless-steel group comes and we can follow all criteria of the ASTM guideline

of these material. Before undergo the testing composition analysis process, the surface of the specimen must be flat enough. There are two possible that the specimen is not flat which is the grinding error and the other reason is other element on the surface of specimen such as corrosion. The sparking emission light reflected out from the specimen and this cause the reading taken by spectrometer in Figure 3.6 is not correctly or not accurate. Therefore the entire specimen must undergo the surface finishing before the metal analysis.



Figure 3.6: Spark emission spectrometer

Table 3.2: Composition of AISI Type 301 stainless steel

Fe	Cr	C	Ma	Ni	S	P
71.1 %	17.5%	0.03%	1.77%	8.07%	0.05%	0.041%

Results shown in Table 3.2 was the composition of AISI Type 301 stainless steel based on reference by American Iron and Steel Institute (AISI) steel type provided.

3.2.5 Cold mounting process

As the procedure to control the heat treatment data then divided of specimen who did not been heat treated was be cold mounting first then then the specimens that need to be heat treated will be undergoes those process first before been cold mounting. Where we just need a top specimen surface only for corrosion test then cold mounting was the best way to make it happen which to avoid another surface open to the slurry and to mount on the connection wire to the specimen. Cold mounting is especially suited for mounting specimens that are sensitive to heat or pressure. Additionally, cold mounting does not require an investment in a mounting press and is therefore good for infrequent mounting tasks. Cold mounting is the general term used to describe multi-component systems such as epoxies, acrylics, and polyesters. This is mixed together and cast in cold mounting machine in Figure 3.7 press by 2 bar air pressure in 10 minutes. Then as shown in Figure 3.8, specimens is ready to been tested for erosion-corrosion test.



Figure 3.7: Cold mounting machine

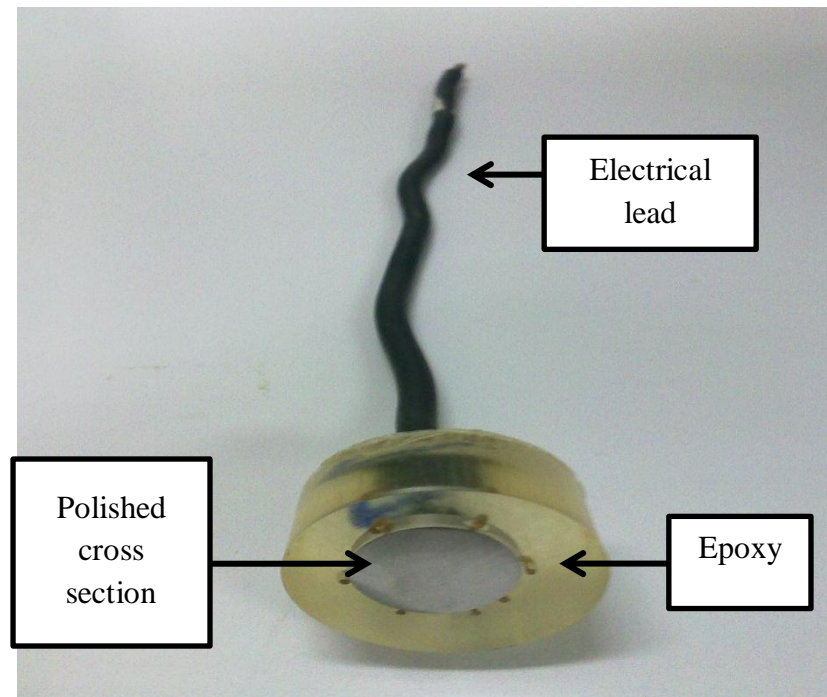
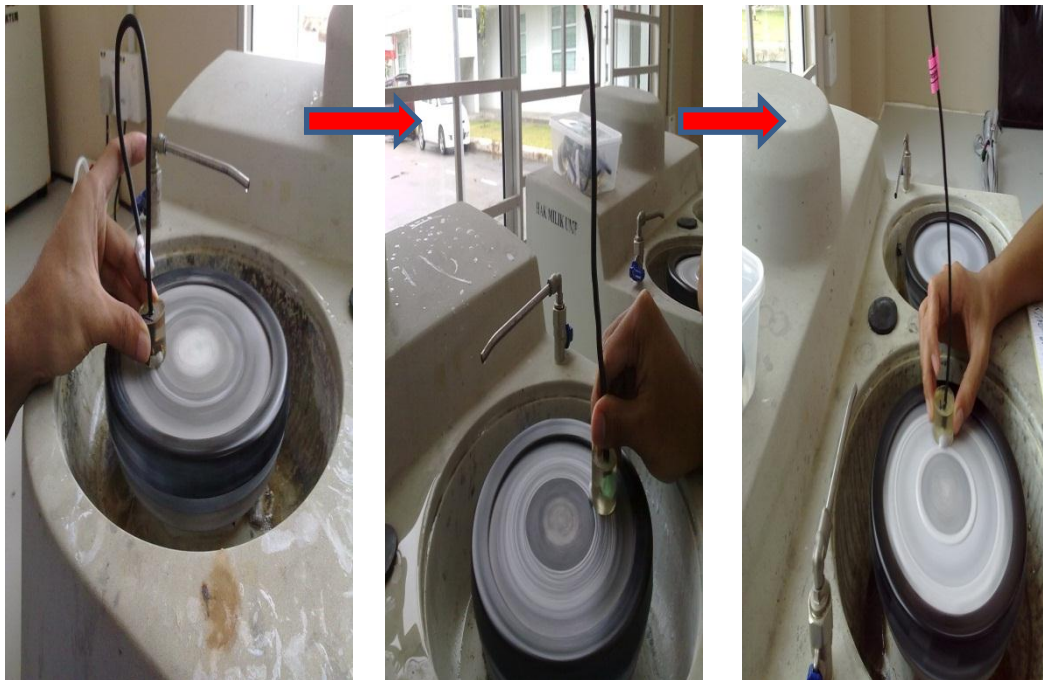


Figure 3.8: Surface of specimen to undergoes erosion-corrosion test

3.2.6 Surface polishing

Specimens after been cold mounting with wire need to be surface polished by three types of polish grade. Figure 3.9 shows there are 1 micron, 0.1 micron and 0.05 micron type of polish grade and each specimen undergoes those grades of polish around 10 minutes each. Then specimens ready to been image analyzer after getting fine surface and erosion-corrosion testing.



(a) grades 1 micron

(b) grade 0.1 micron

(c) grade 0.05 micron

Figure 3.9: Three grade of polish type

3.3 HEAT TREATMENT PROCESS

Heat treatment effect was the manipulate data in this project where specimen were undergoes some effect of heat treated. Stainless steel was quenching annealed and cooling in furnace and cooling to the open air. The specimens were heat to 1070 °C for 1 hours 45 minute and open air to the exposure in the carbide precipitation region. Time undergoes the annealing process based on the thickness of the specimens as shown in Table 3.3.

Table 3.3: Approximate periods for annealing steel

Thickness of metal (inch)	Time of heating to required temperature (hour)	Soaking time (hour)
Up to 1	3/4	1/2
1-2	1 1/4	1/2
2-3	1 3/4	3/4

Specimens were heating to required temperature from 0-1070 °C for 1 and ¼ hours in furnace as shown in Figure 3.10 and 3.12 then were soaking for ½ hours at 1070 °C in Figure 3.11. After that specimen were cooling to the open air till it cool at room temperature.

**Figure 3.10:** Furnace for heat treatment process



Figure 3.11: Program controller setting for furnace to heat and soaking specimens at 1070 °C

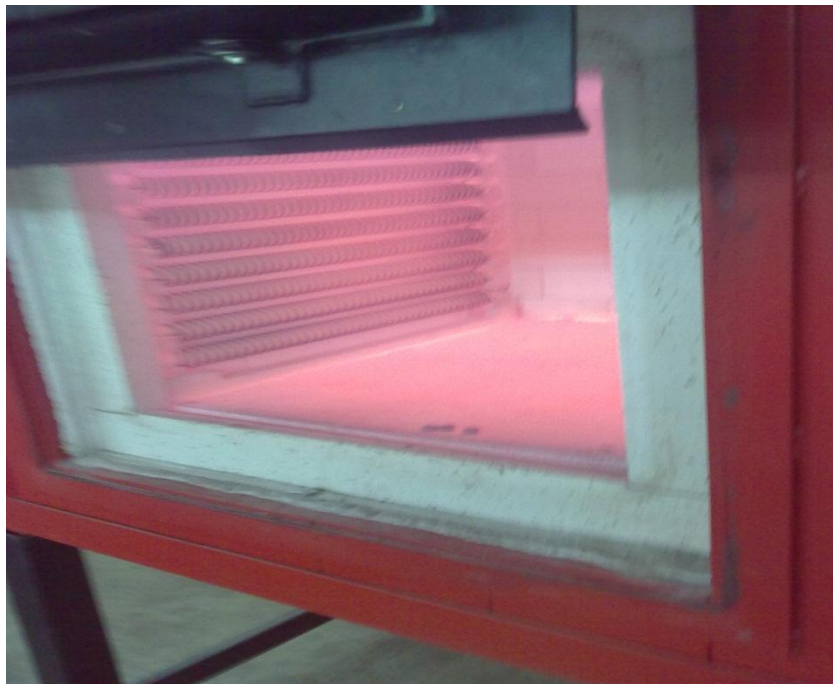


Figure 3.12: Specimens undergoes heating process in furnace

3.4 INSPECTION OF EROSION-CORROSION PARAMETER

Inspection of erosion corrosion parameter was prepared to observe and determine the material behavior and mechanical properties corresponding to each of the specimen. There were two time of inspection to be done which are before and after the material undergo erosion corrosion test. The test of preparation to check the microstructure inspection, Rockwell hardness test, and surface roughness test.

3.4.1 Surface microstructure analysis

Corrosion effect can be observed by microstructure as pitting corrosion will straightly can be shown. After the specimen is dried, the microstructure of the specimen is observed by using microscope knows as Image Analyzer as shown in Figure 3.13.



Figure 3.13: Image analyzer

3.4.2 Surface roughness test

After done with the measuring specimen weight, the surface roughness (Figure 3.14) was checked to know the Ra value of the specimen. This test was do before and after at every single experiment or test. Surface roughness, often shortened to roughness, is a measure of the texture of a surface. It is quantified by the vertical deviations of a real surface from its ideal form. If these deviations are large, the surface is rough if they are small the surface is smooth. Roughness plays an important role in determining how a real object will interact with its environment. Rough surfaces usually wear more quickly and have higher friction coefficients than smooth surfaces. Roughness is often a good predictor of the performance of a mechanical component, since irregularities in the surface may form nucleation sites for cracks or corrosion.



Figure 3.14: Surface roughness Perthometers

3.4.3 Rockwell Hardness test

Lastly, the hardness of the specimen was test using the Rockwell hardness test. The Rockwell hardness test shown in Figure 3.15 and 3.16 is the most common method for determines the hardness of ferrous and many nonferrous metals. In this test, a standard minor load is applied to set a hardness steel ball or a diamond cone in the surface of the metal, followed by the application of a standard major load. The hardness is measured by depth of penetration. Rockwell superficial hardness a made using lights minor and major load and a more sensitive system for measuring depth of indentation. The indenter is force into the test material under a preliminary minor load and after equilibrium is achieves an indicating device, which follows the movement of the indenter, is set to the datum position. An additional major load is then applied with resulting increase in penetration. The conditions are then allowed to stabiles and then the additional major load is removed, leaving the minor load in places. In this test, a hard steel ball or diamond cone indenter is pressed into the surface of the material being test. The result of the test is read directly from machine.



Figure 3.15: Rockwell hardness test



Figure 3.16: Result for HRC appears on screen after load applied.

3.5 ELECTROCHEMICAL TEST AND MEASUREMENT

The electrochemical study was conducted using a Potentiostat WPG100 interfaced to a computer. The basic instrument need for the electrochemical test includes:

- (i) Working Electrode (W.E)
- (ii) Counter Electrode (C.E)
- (iii) References Electrode (R.E.)
- (iv) Potentiostat WPG100
- (v) Computer with software program
- (vi) Test cell

The test (Figure 3.17) was conducted in aqueous slurries adding with sand particles. All of the solution had been stored at the required temperature to prevent from any condition which might affect the solution.

3.5.1 Electrochemical cell set-up

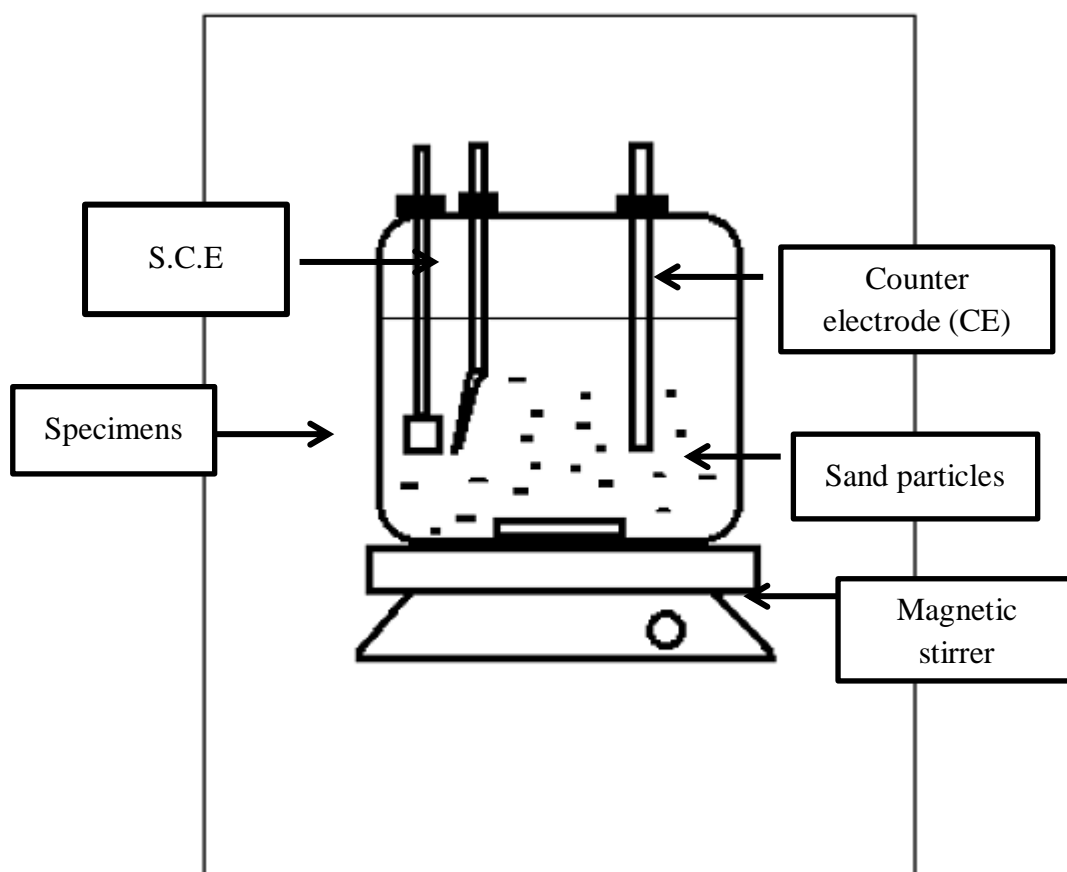


Figure 3.17: Electrochemical test cell

The working electrodes were connected to the specimens which is AISI Type 301 Stainless Steel, a graphite wire as counter electrode, CE (red cable) and saturated calomel electrode S.C.E. as the reference electrode, RE (blue cable) as shown in figure. Saturated calomel electrode S.C.E. was used as reference electrode as it provides stable references against which the applied potential may be accurately measured.

The counter electrode was used to provide the applied current and as such should be composed of highly corrosion resistance material. Counter electrode that were used in this cell is graphite rod. (See Figure 3.18)

The lugging capillary connected as references electrode that allows sensing of the solution potential close to the working electrode without the adverse effect that occur when the large reference electrode is placed near the working electrode. Erosion corrosion test were carried out by immersing the specimen in an aqueous 3.5% NaCl solution and in apply of erosion-corrosion mechanism, 10% of sand particles were placed and magnetic stirrer gives the velocity to the aqueous around 100 rpm. The effect of sand particles were taken as the corrosion reacts to specimens. The constant electrolyte temperature as room temperature used maintains in each specimens testing. All the potential measurement had been analyzed by using Potentiostat (WPG100) as shown in Figure 3.19 and Figure 3.20.

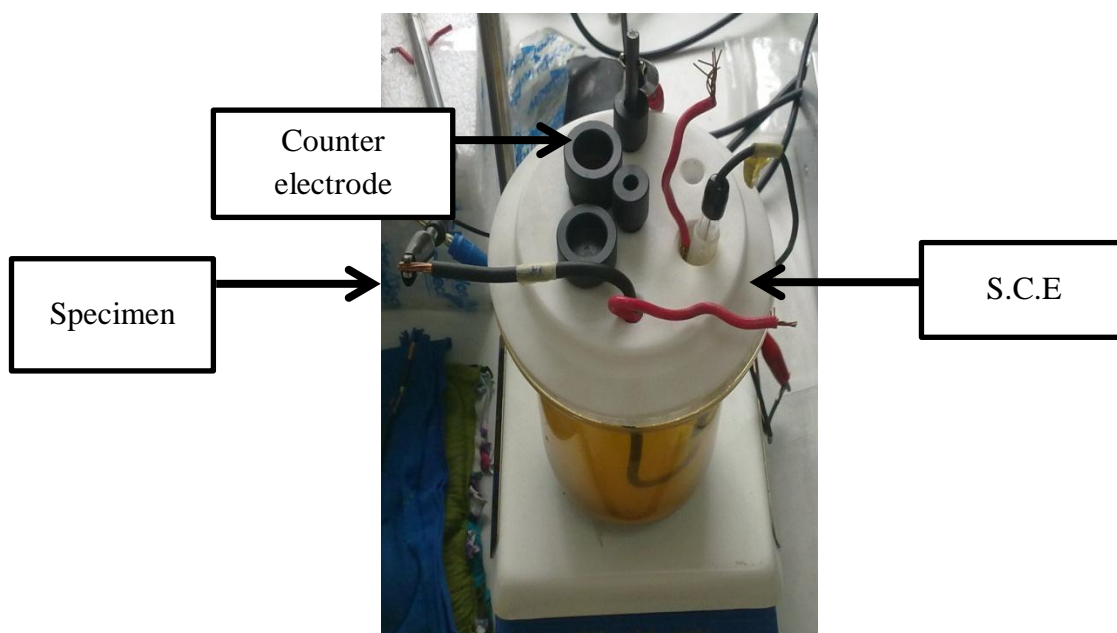


Figure 3.18: Working electrodes connected to Potentiostat (WPG100)



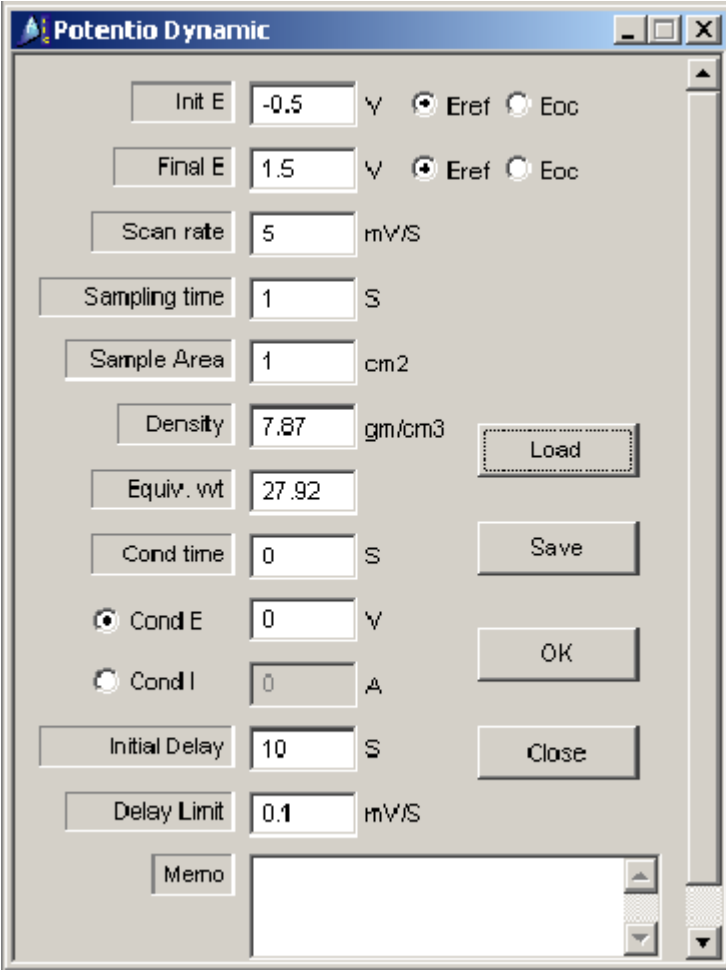
Figure 3.19: Potentiostat (WPG100)



Figure 3.20: Electrochemical test set-up

3.5.2 General parameter

There are number of parameters which must be specified and can be controlled by IVMan software. The first is the initial is the initial and final potentials which the scan will take. In this software the default value for initial and final potential is -50 mV to 150 mV. The value of the scan rate used is 5 mV/s several parameters such as temperature, sampling time, voltage, density and area of sample will be set with desire value for use in the test. The density of was set up to 7.87 g/cm³ sample area 1.767 cm² and equivalent weight is 27.92 g. Figure 3.21 show general parameter from Potentiostat.



The screenshot shows the 'Potentio Dynamic' window with the following parameters and controls:

- Init E:** -0.5 V (Radio button selected for Eref, Eoc is unselected)
- Final E:** 1.5 V (Radio button selected for Eref, Eoc is unselected)
- Scan rate:** 5 mV/S
- Sampling time:** 1 S
- Sample Area:** 1 cm²
- Density:** 7.87 gm/cm³
- Equiv. wt:** 27.92
- Cond time:** 0 S
- Cond E:** 0 V (Radio button selected, Cond I is unselected)
- Cond I:** 0 A
- Initial Delay:** 10 S
- Delay Limit:** 0.1 mV/S
- Memo:** A text area for notes.
- Buttons:** Load, Save, OK, Close.

Figure 3.21: General Potentio dynamic parameters from Potentiostat WPG100 for IVMan software

3.6 CORROSION RATE ANALYSIS

3.6.1 Tafel plot

Tafel plot allow the direct measurement of the corrosion current from which the corrosion rate can be quickly calculated in the IVMAN software.

Figure 3.22 show the example of Tafel plot. Vertical axis is potential and the horizontal axis is the logarithm of absolute current. Theoretical current for the anodic and cathodic reaction was shown is straight lines. The curved line is the total current which is the sum of the anodic and cathodic currents. Sharp points in the curve as shown in figure is actually the point where the current changes signs as the reaction changes from anodic to cathodic or vice versus. The sharp point is due to the use of a logarithmic axis and the use of a log axis is necessary because of the wide range of current values that must be displayed during a corrosion experiment.

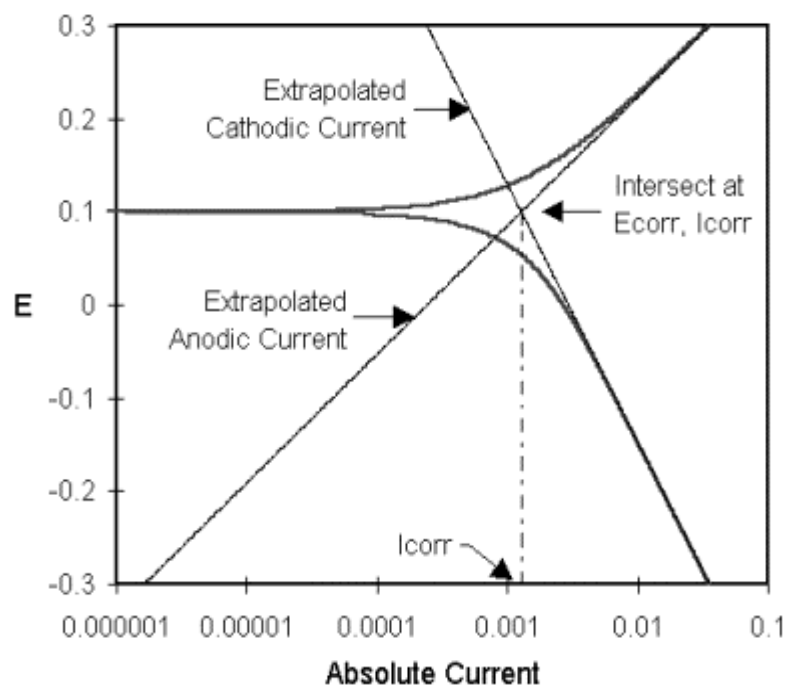


Figure 3.22: Tafel plot

Source: Hench (1985)

The potential of the metal is the means by which the anodic and cathodic reactions are kept in balance. Notice that the current from each half reaction depends on the electrochemical potential of the metal. Excess electrons shift the potential of metal more negative which slows the anodic reaction and speed up the cathodic reaction. The equilibrium potential assumed by the metal in the absence of the electric connections to the metal is called the open circuit potential E_{oc} . In electrochemical corrosion experiment, the first step is the measure of E_{oc} . The value of either the anodic or the cathodic current at the intersection is I_{corr} and E_{corr} is the value of E_{oc} before experiment. The value of I_{corr} was used to calculate corrosion rate of the specimen using IVMan software. Figure 3.23 shows the Tafel analysis using IVMan software to get the corrosion rate value.

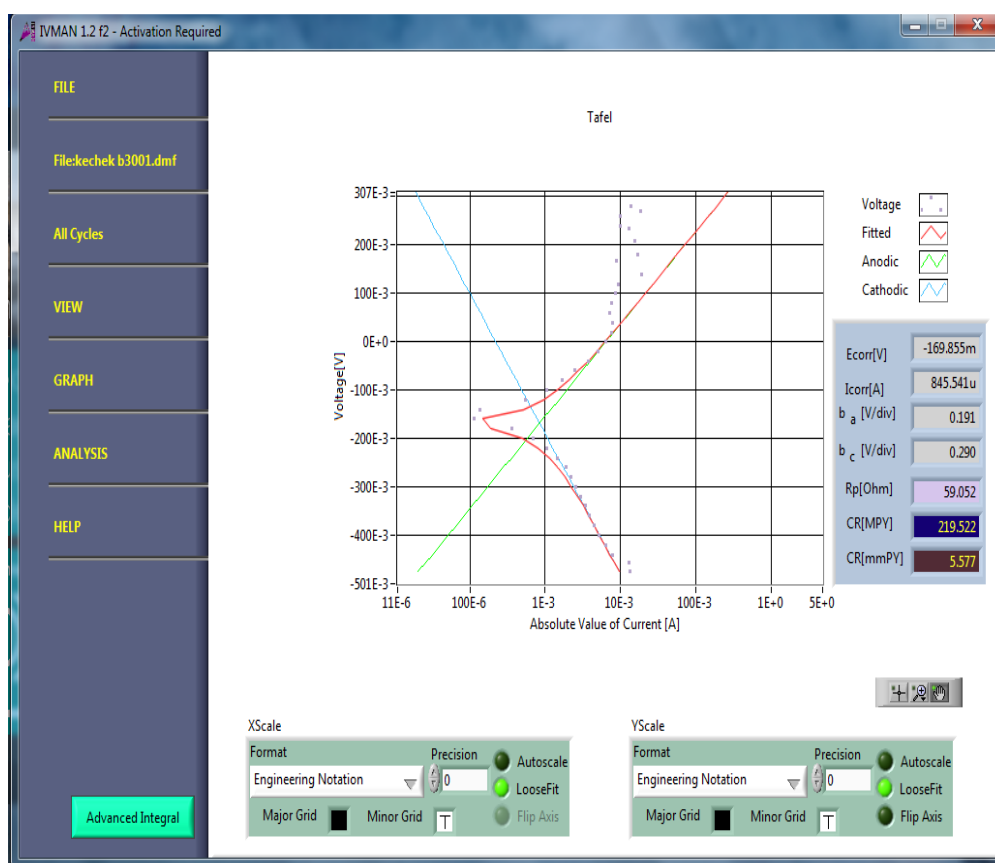


Figure 3.23: Tafel analysis using IVMan software

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter is to discuss the results that have been obtained from the experiment. The experiments that have been done were annealing process of stainless steel and undergo the electrochemical erosion-corrosion test by specified parameters. The objective of this chapter is to determine the effects of heat treatment on erosion-corrosion behavior of annealed AISI Type 301 Stainless Steel in aqueous slurries environment. The outcome of the experiment had been discussed in detail.

4.2 HARDNESS TEST ANALYSIS

The major purpose of annealing is to reduce hardness where the other purpose was improving machinability, facilitate cold-working, produce a desired microstructure. Full annealing is the process of softening steel by a heating and cooling cycle, so that it may be bent or cut easily. Table 4.1 showed that effect of annealing on AISI Type 301 Stainless Steel can reduce the hardness of the steel. As the data come out in Table 4.1, AISI Type 301 Stainless Steel reduces about 43% of hardness after being annealing. Effect of steel reducing hardness will cause the higher corrosion implement.

Table 4.1: Rockwell hardness test of specimens before electrochemical erosion-corrosion test

Specimens	Rockwell hardness test data	Average data (HRC)
AISI Type 301 Stainless Steel as control specimens	C 28.9 C 29.2 C 29.6	C 29.23
AISI Type 301 Stainless Steel annealing specimens	C 20.0 C 18.7 C 22.5	C 20.40

4.3 CORROSION RATE ANALYSIS

There were at least two electrochemical reactions which are an oxidation and a reduction reaction occurs during corrosion at a metal-electrolyte interface. It happens because the corrosion is due to an electrochemical mechanism, it is clear that electrochemical techniques can be used to study corrosion reactions and mechanisms. This behavior noted for all samples that were tested.

Figure 4.1 to 4.6 (a) illustrated a Potentiodynamic polarization curve obtain during erosion-corrosion test and Figure 4.1 to 4.6 (b) show the Tafel extrapolation for the Potentiodynamic polarization curve of the sample which can determine the corrosion rate of the sample during test.

Corrosion normally occurs at a rate determine by equilibrium between opposing electrochemical reactions. The first is anodic reaction in which a metal is oxidized, releasing electrons into the metal. The other is the cathodic reaction in which a solution species H^+ is reduced, removing electrons from the metal. When these two reactions are in equilibrium, the flow of electrons from each reaction is balanced, and no net electron flow (electric current) occurs.

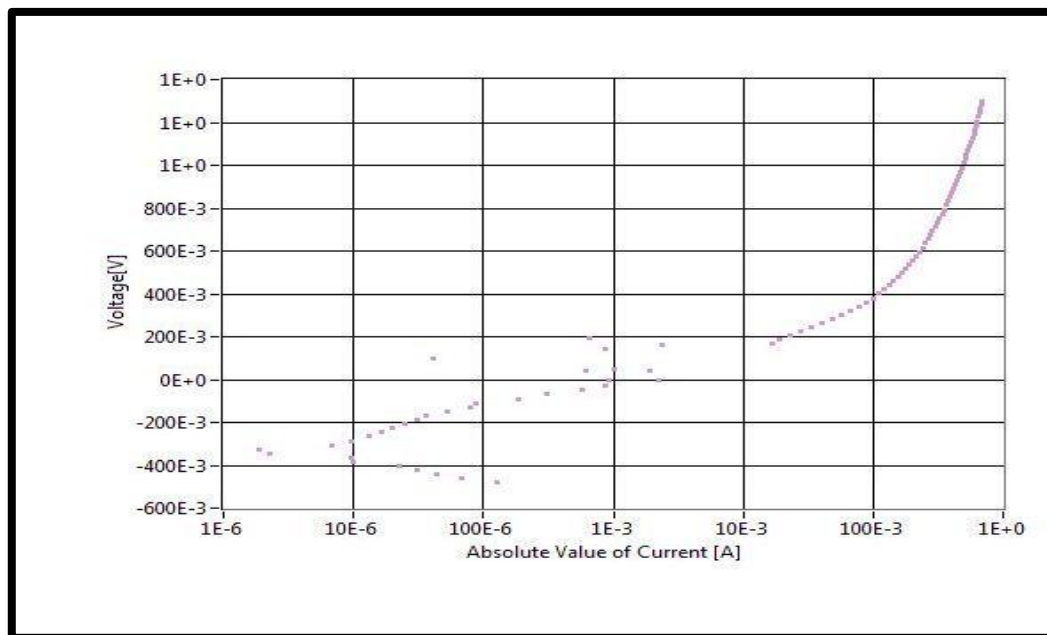
From Figure 4.1 to 4.6(b) Tafel exploration plot, the vertical axis is potential and the horizontal axis is the logarithm of absolute current. The theoretical current for the anodic and cathodic reaction are shown as strapline. The curved line is the total current which is the sum of the anodic and cathodic currents. This is the current were measured sweep of the potential of the metal with Potentiostat.

This experiment corresponds to Potentiostatic tests but instead of maintaining a well-defined potential, the latter is swept at constant rate using a function generator to drive the Potentiostat. The entire Potentiodynamic tests were conducted after stabilization of the free corrosion potential. The nature of stabilization of free corrosion potential with time was same because tests were conducted between same materials which are Type 301 Stainless Steel.

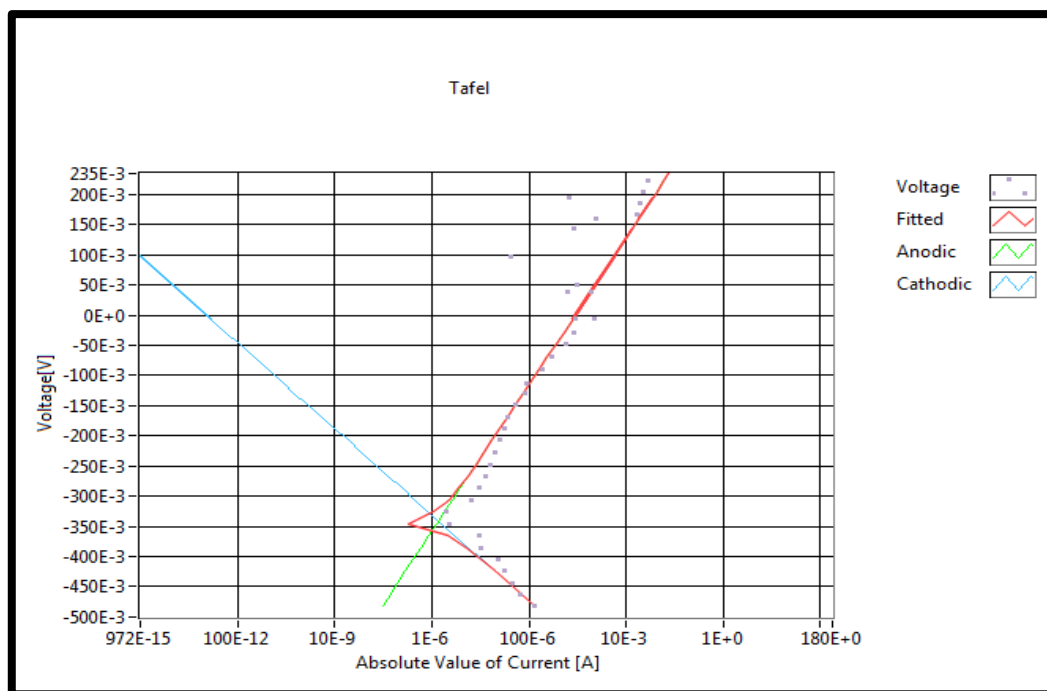
The experimental Tafel extrapolation plots were analyzed. The cathodic (β_c) and anodic (β_a) Tafel slopes, the estimates corrosion current density (i_{corr}) and estimated corrosion potential (e_{corr}), corrosion resistance (R_p) and corrosion rate are tabulated in Table 4.2. The corrosion rates were fairly reproducible and the corrosion rates of the specimen's type were comparable.

From the Table 4.2, annealed AISI Type 301 stainless steel specimens (B1, B2, B3) shown higher corrosion rate than the control specimens (A1, A2, A3). Effect of heat treatment annealed of AISI Type 301 stainless steel give corrosion rate 3 times more corrode than the actual specimens where average of annealed specimens was 3.8380 mm/year than 0.0132 mm/year of control specimens.

Figure 4.5 (b) Tafel extrapolation plots did not get in exact way because there is no interpolation axis between cathodic and anodic slopes but the data come out give the expected results of corrosion rate. The effect of Potentiostat condition and others factor maybe the way about the plot did not come as well as needed.

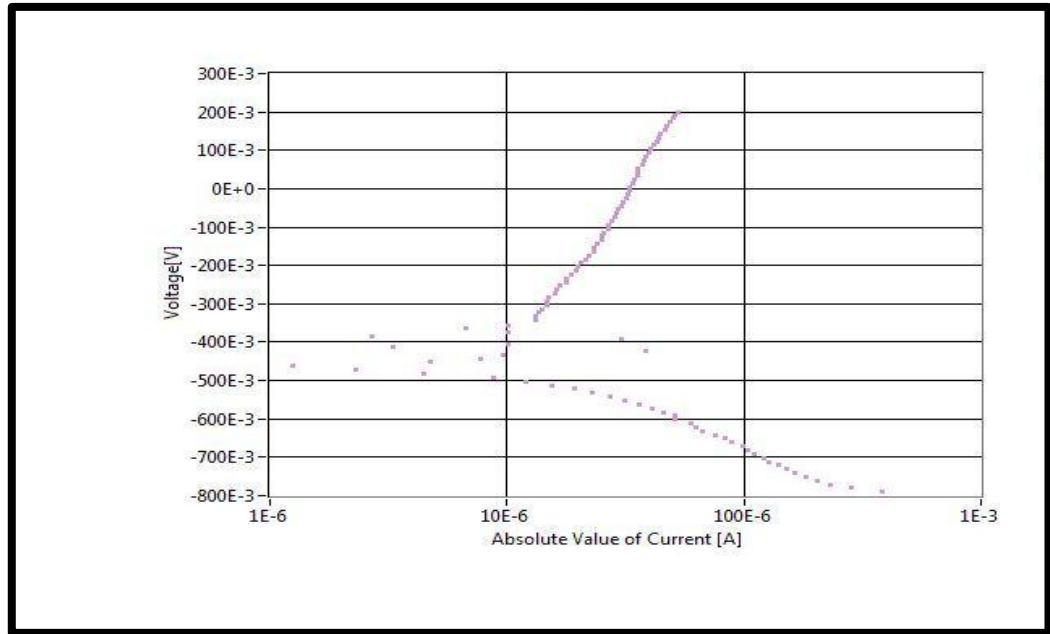


(a)

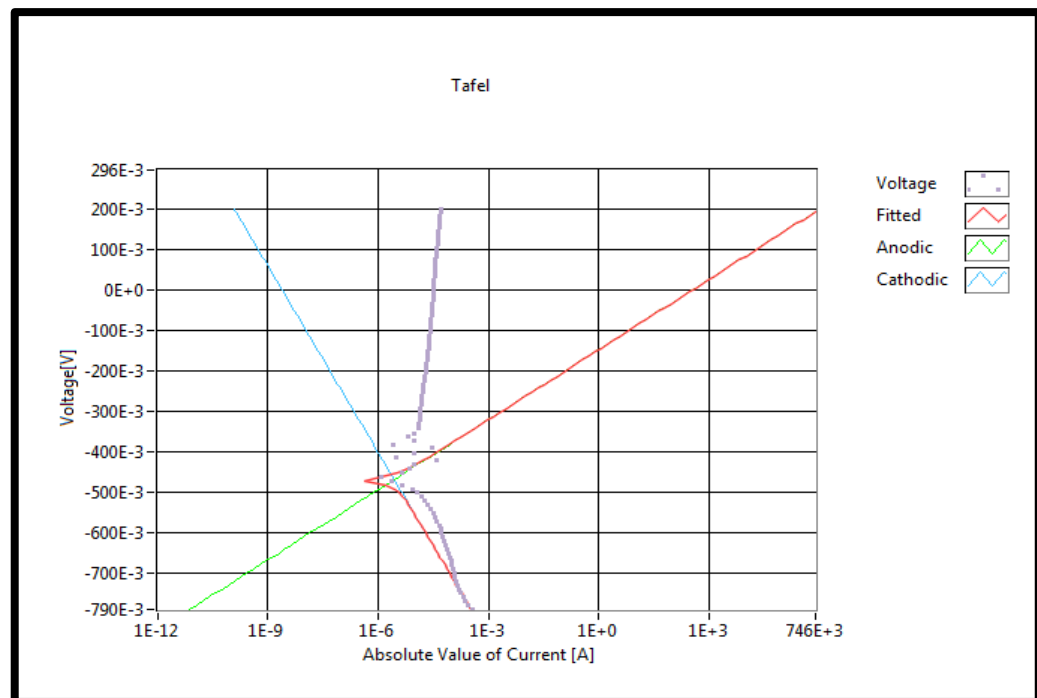


(b)

Figure 4.1: Specimen A1 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel

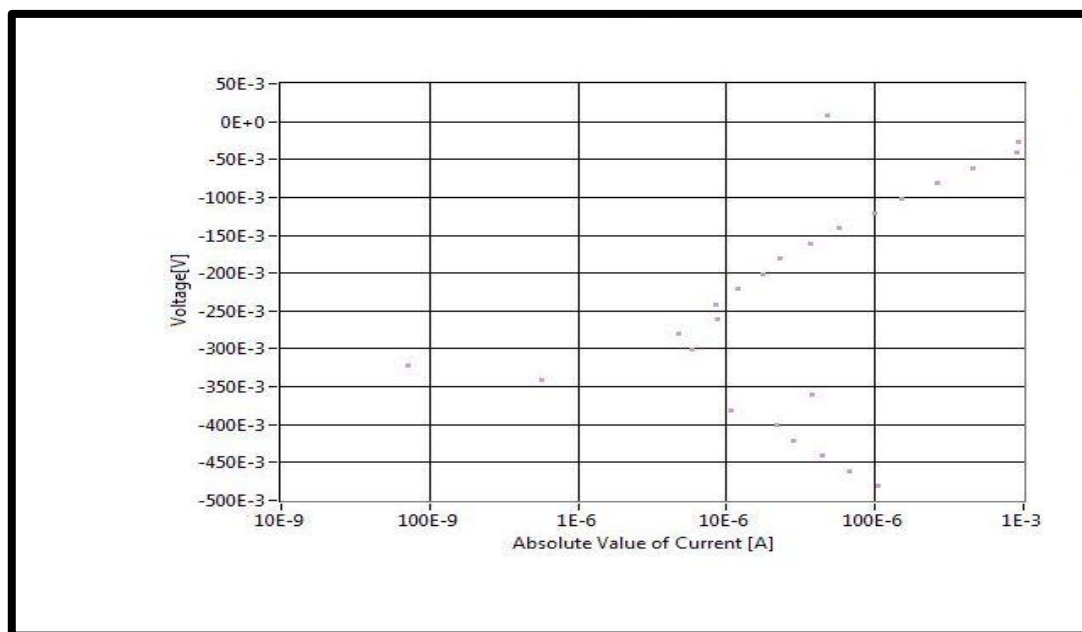


(a)

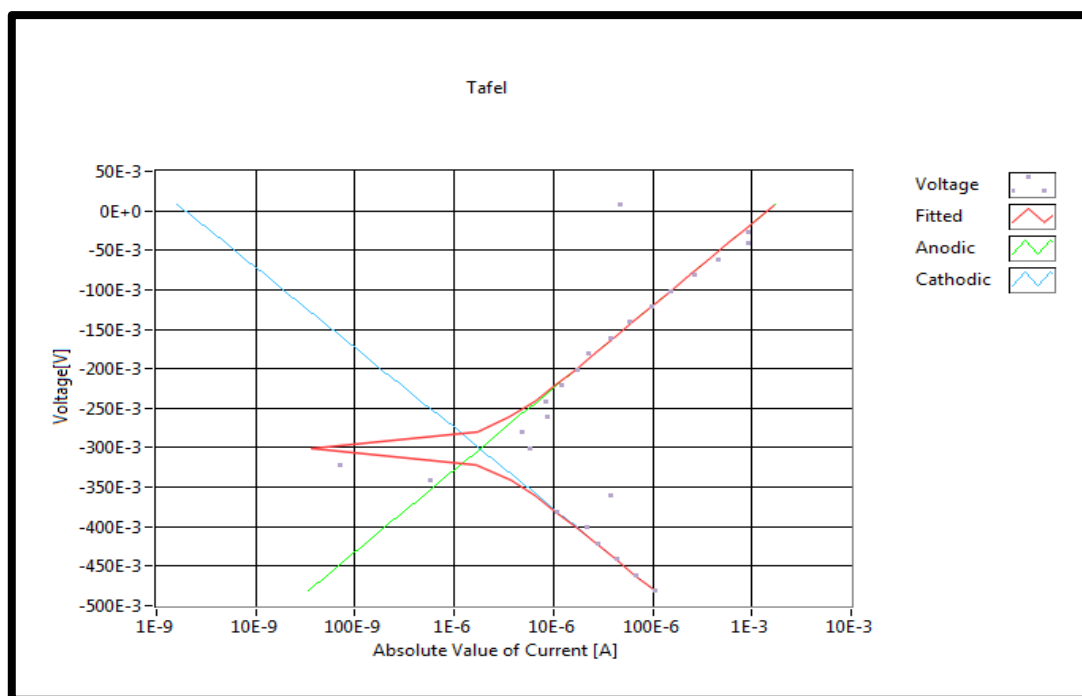


(b)

Figure 4.2: Specimen A2 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel

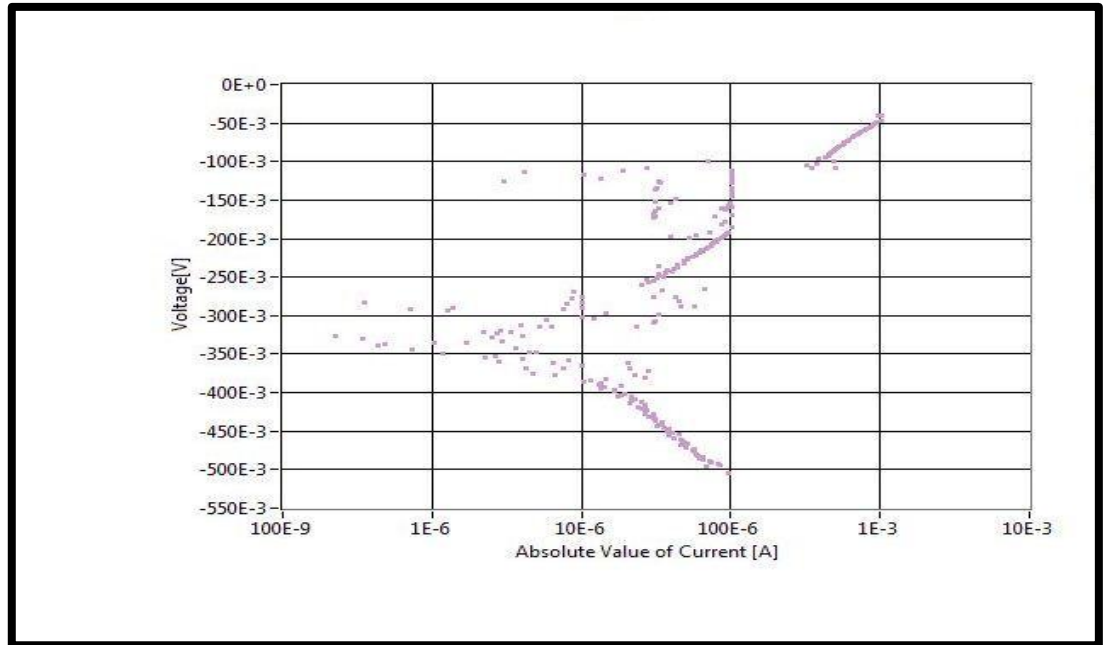


(a)

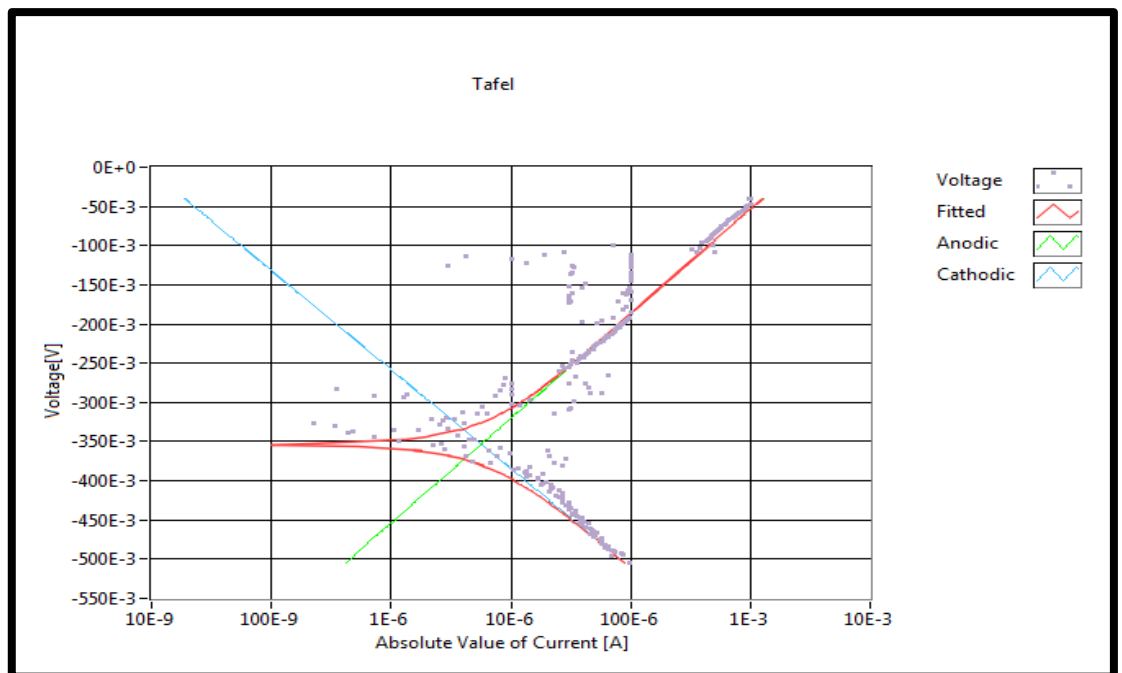


(b)

Figure 4.3: Specimen A3 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for control specimens AISI Type 301 Stainless Steel

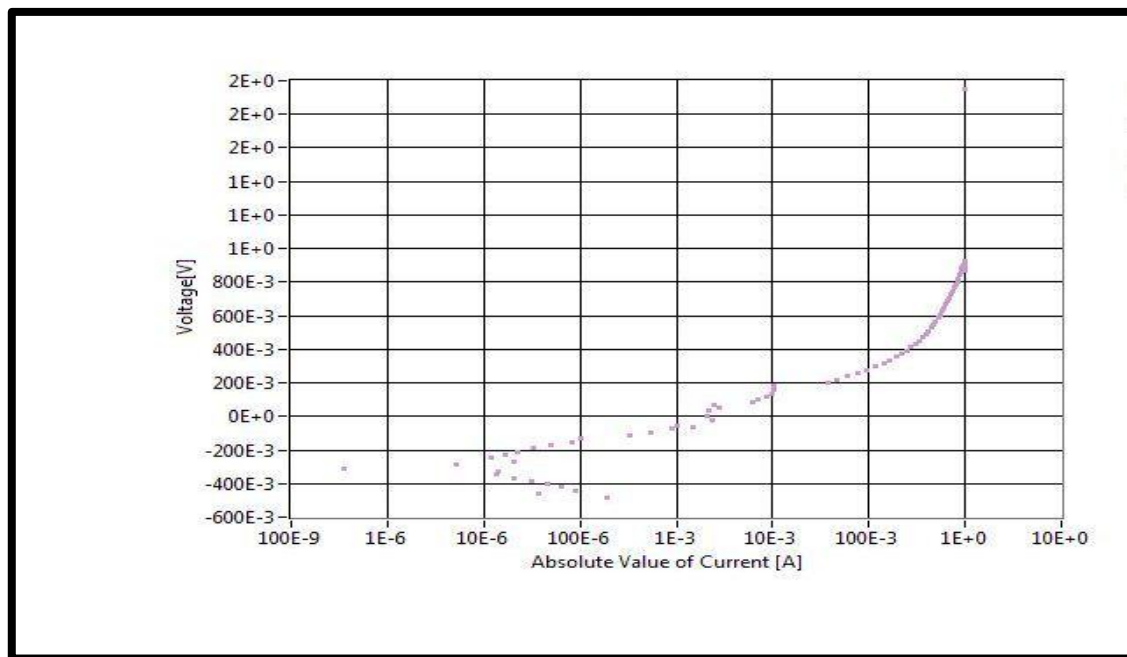


(a)

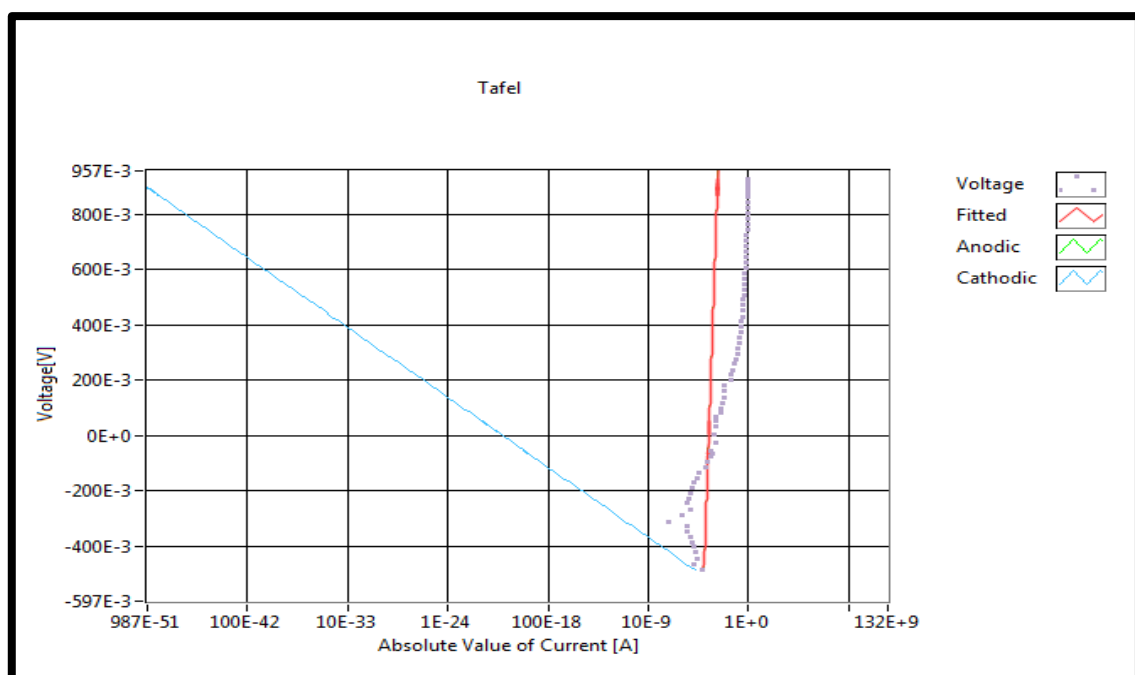


(b)

Figure 4.4: Specimen B1 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless Steel

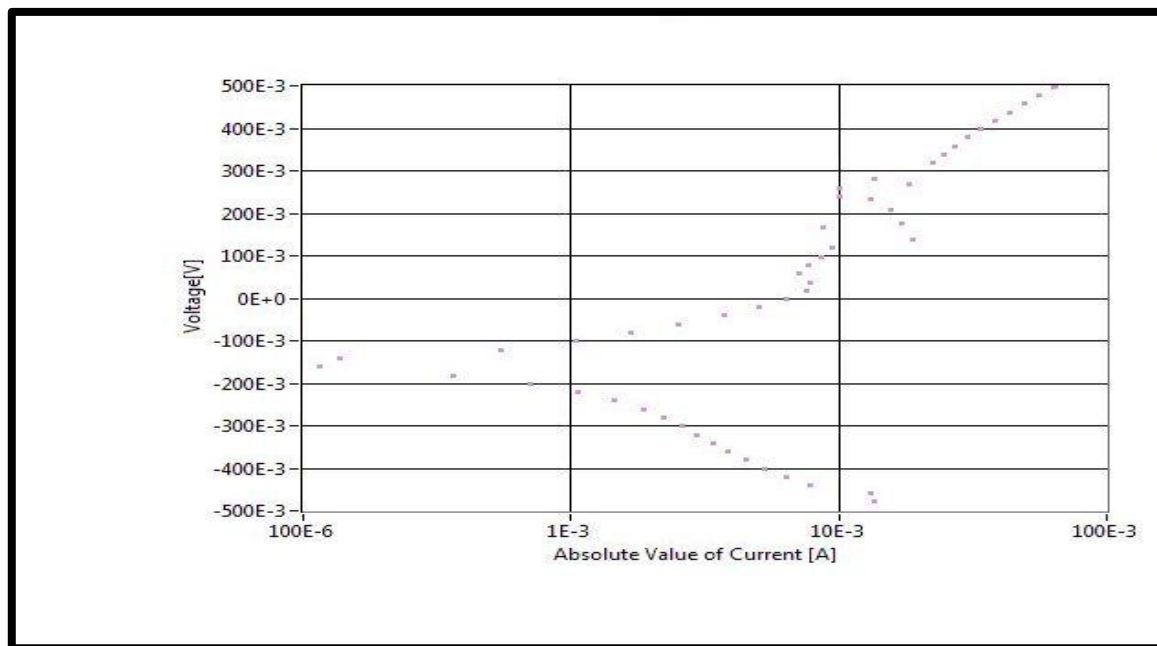


(a)

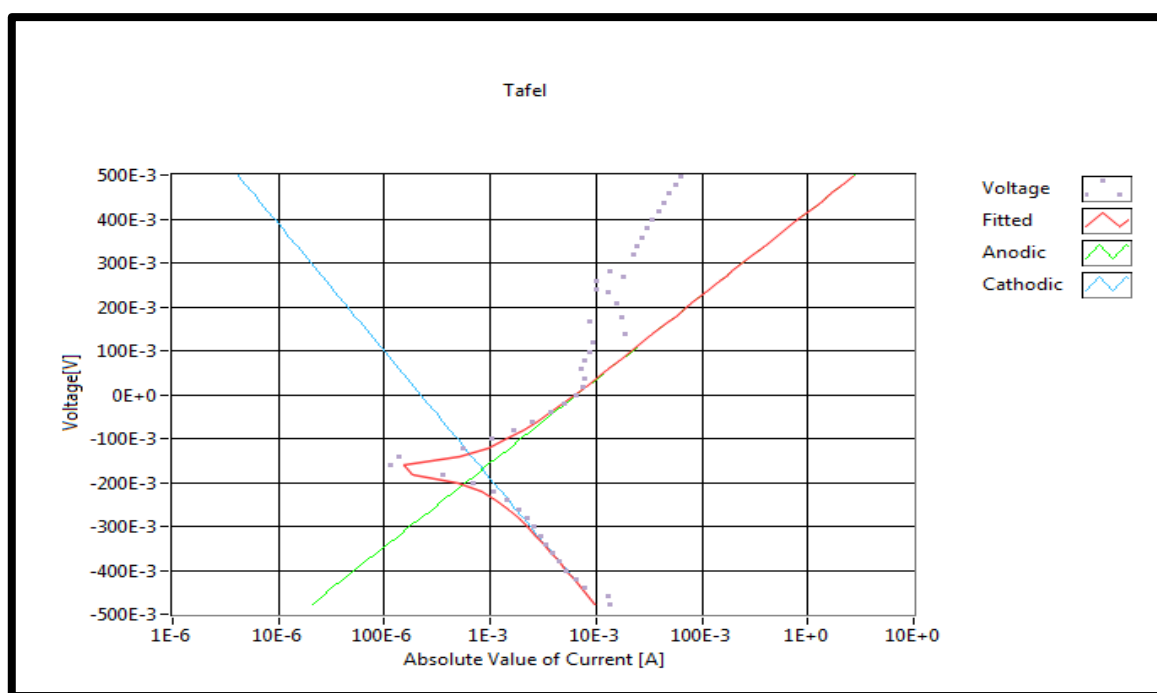


(b)

Figure 4.5: Specimen B2 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless Steel



(a)



(b)

Figure 4.6: Specimen B3 (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot for annealed specimens AISI Type 301 Stainless

Table 4.2: Corrosion rates determine by Tafel Extrapolation Method

Specimen	e_{corr} (V vs SCE)	i_{corr} (A/cm ²)	β_a (V/dec)	β_c (V/dec)	R _p (ohm)	Corr Rate (mm/year)	Average Corr Rate (mm/year)
A1	-0.3411	0.00000138	0.1210	0.071	14709.0	0.0092	0.0132
A2	-0.4696	0.00000283	0.0580	0.154	6468.0	0.0187	
A3	-0.3014	0.00000181	0.1040	0.102	2079.0	0.0119	
B1	-0.3531	0.0006320	0.1340	0.126	44.61	4.1680	3.8380
B2	-0.5071	0.0002682	1.2880	0.032	49.95	1.7690	
B3	-0.1698	0.0008455	0.1910	0.290	59.02	5.5770	

4.4 SAMPLE CHARACTERIZATION

The surface image analyze of the sample had been revealed by using inverted microscope before and after electrochemical erosion-corrosion test.

The following figure shows the surface image of AISI Type 301 Stainless Steel using Inverted microscope at magnification 50X. Six samples had been selected which 3 samples were the control specimens, which non heat-treated AISI Type 301 Stainless Steel and the other 3 specimens who have been annealed Type 301 Stainless Steel.

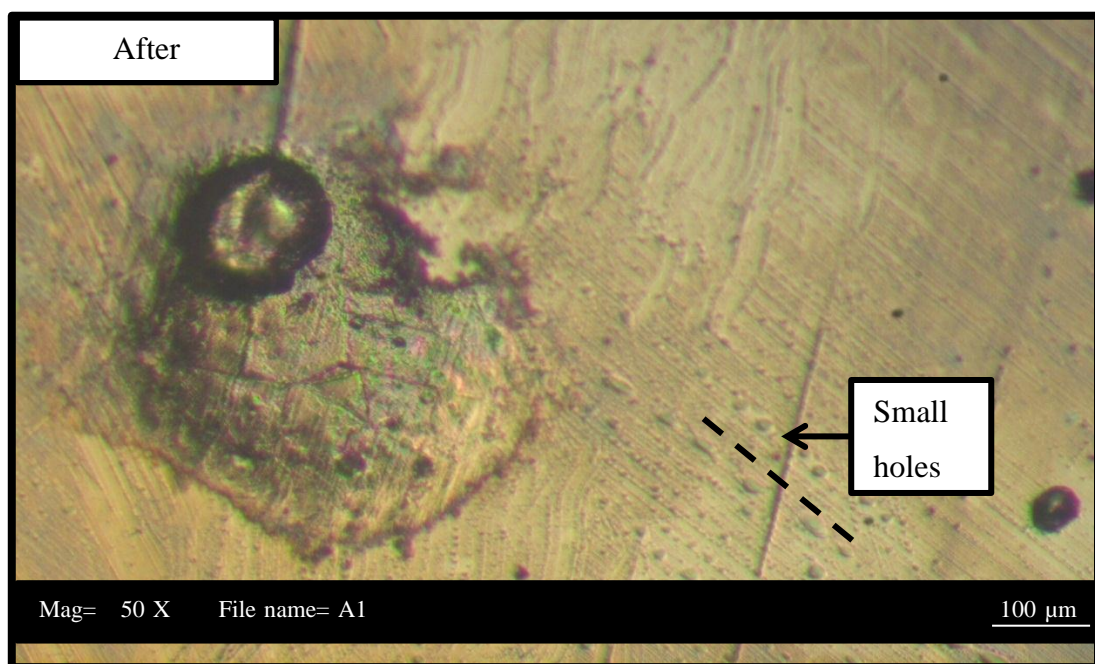
Figure 4.7 till 4.12 showed image of surface revealed by inverted microscope before and after the electrochemical erosion-corrosion for each specimens. These effects of electrochemical erosion-corrosion test can be analyzed by formation of pitting corrosion. The pits built up after erosion-corrosion test on Figure 4.10 till 4.12 shows more and larger than Figure 4.7 till 4.9. This happen because the annealed specimens B1, B2 and B3 more soft than control specimens A1, A2 and A3. It is first noticeable rough surface which blotches the surface. After the cleaning process holes and pits can be seen in the surface.

Pitting corrosion is one of the most destructive and intense forms of corrosion. It is first noticeable as a white or gray powdery deposit, similar to dust, which blotches the surface. When the deposit is cleaned away, tiny holes or pits can be seen in the surface. Stainless steel alloys with chromium depend on a passive film for corrosion resistances are susceptible to pitting by local breakdown of the film at isolated sites. Pitting is a form of localized corrosion that proceeds because of local cell action which produces cavities beginning at the surface. Pitting usually occurs on metal surface immersed in aqueous slurries. (Fontana, 1987)

The effect of velocity and sand particles to simulate erosion-corrosion test also give effect to the surface. The annealed specimens cause the high effect of sand impact where the surface showed the scratch and holes that also can cause pitting happen due to the reducing their hardness. The collision between the surface and particle sand by giving velocity of aqueous, accelerates the mechanical removal rate of the passive film thus accelerating the erosion-corrosion process. The sand particles did not cause much on surface of control specimens where the hardness of AISI Type 301 Stainless Steel still in protection of passive film. Results shown by (S.S. Rajahram, 2009) in erosion-corrosion resistance of materials in various test condition also given results as sand particle and solution given effect to corrosion rate and corrosion pitting on the material testing surface.



(a)

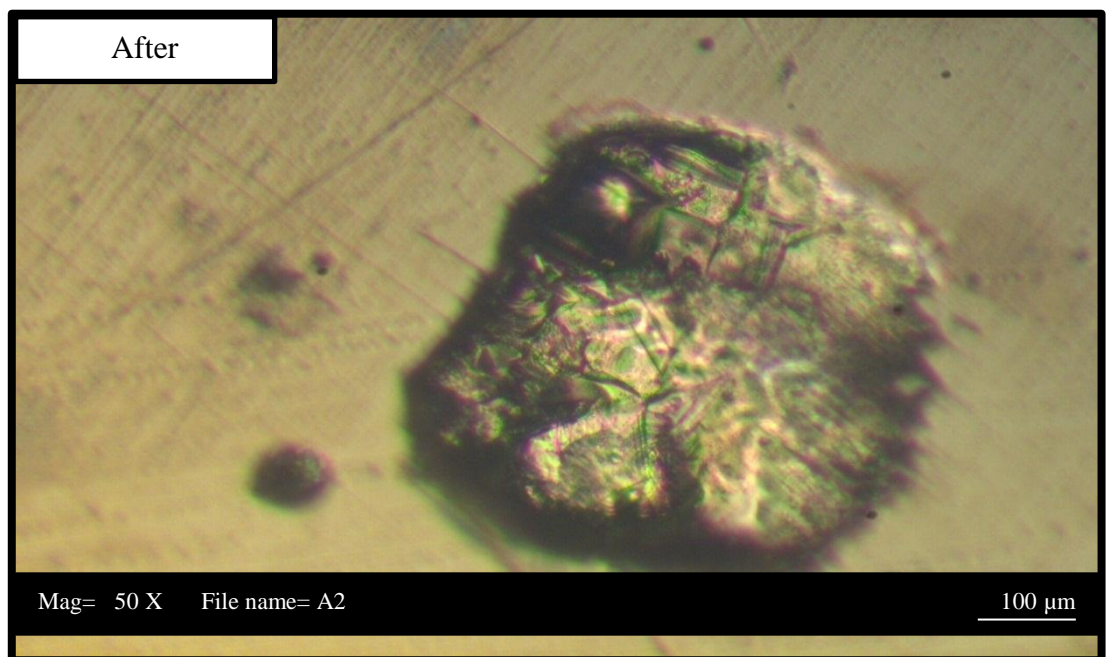


(b)

Figure 4.7: Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A1; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test



(a)

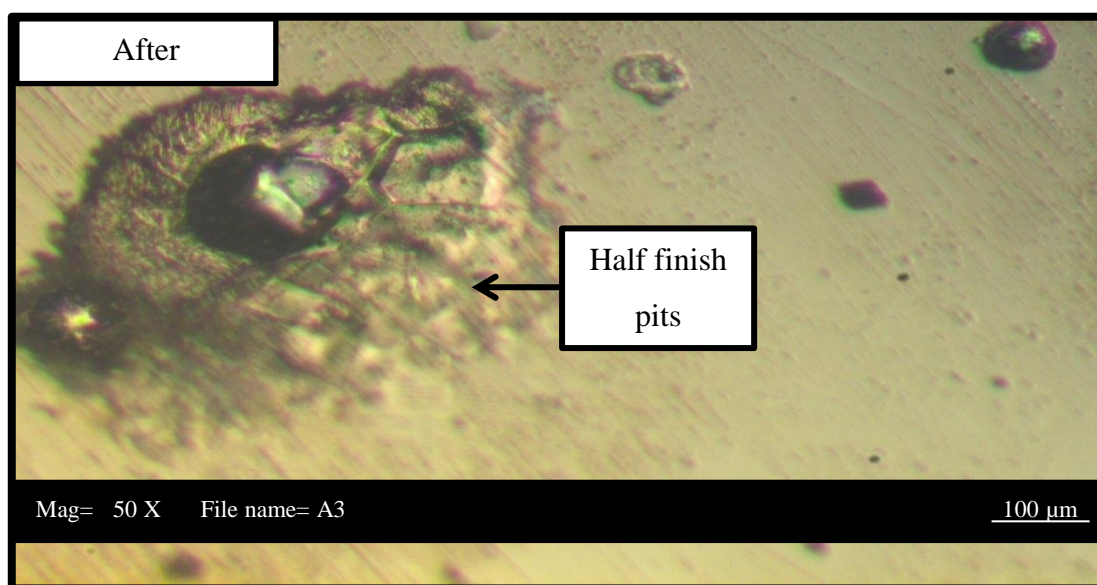


(b)

Figure 4.8: Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A2; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test



(a)



(b)

Figure 4.9: Surface structure at magnification 50X of AISI Type 301 Stainless Steel as control specimen A3; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test



(a)

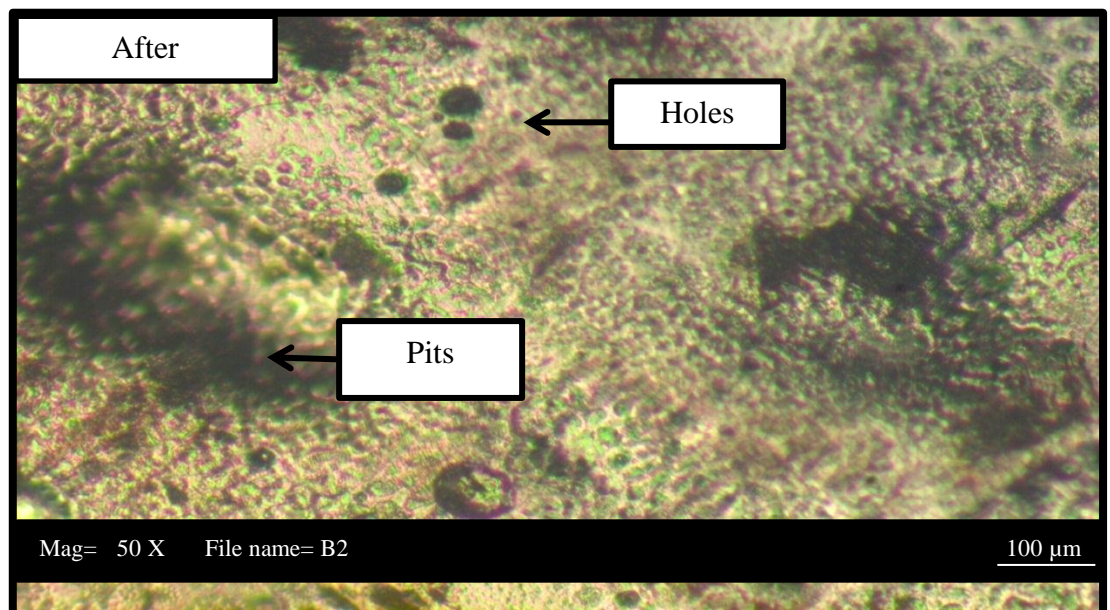


(b)

Figure 4.10: Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070° specimen B1; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test



(a)

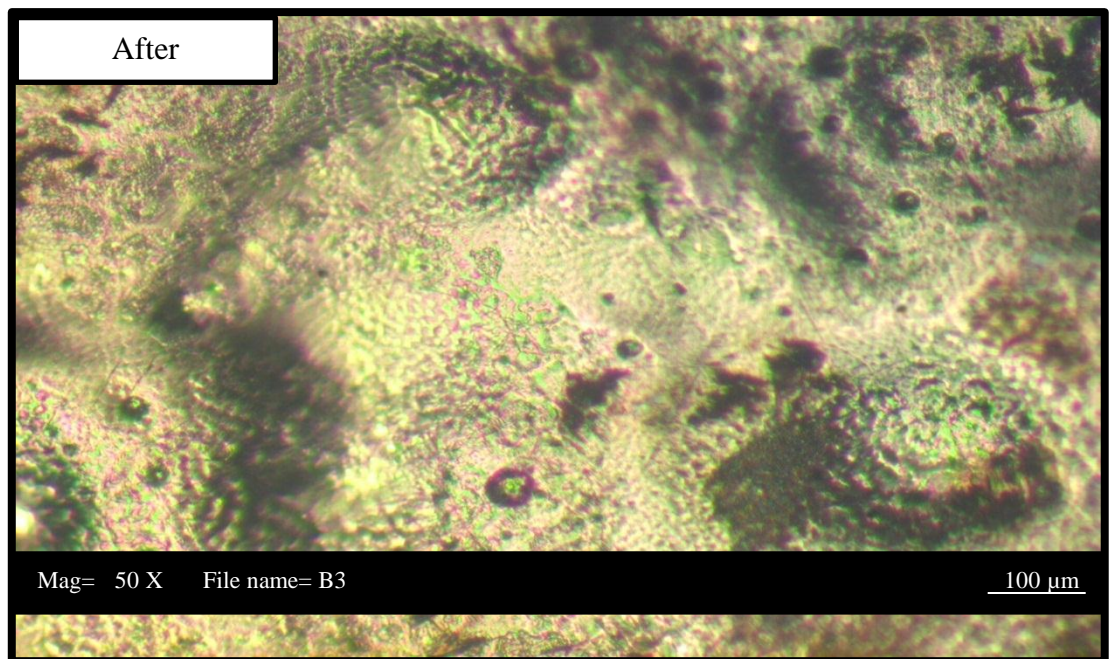


(b)

Figure 4.11: Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070°C specimen B2; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test



(a)



(b)

Figure 4.12: Surface structure at magnification 50X of annealed AISI Type 301 Stainless Steel annealing at 1070°C specimen B3; (a) before electrochemical erosion-corrosion test (b) after electrochemical erosion-corrosion test

4.5 SURFACE ROUGHNESS, Ra ANALYSIS

Third way to define how the corrosion rate can be approving by the test was the surface roughness analysis. Each specimen was taken their surface roughness, Ra value before and after electrochemical erosion-corrosion test were conduct.

Based on Figure 4.13 where graph bar plotted shows from results of surface roughness, Ra analysis where annealed specimens show higher increasing of average Ra value than control specimens $Ra\ 3.823 > Ra\ 1.301$.

Graph from Figure 4.13 based from data taken in Table 4.3, average of control data surface roughness, Ra before erosion-corrosion test was $0.023\ \mu m$ and after corrosion was $1.324\ \mu m$ which calculated as increasing just around $Ra\ 1.301\ \mu m$. Different to effect of heat treatment specimens where their surface having higher pits, holes and scratch and data of surface roughness evaluated that there is increasing of $Ra\ 3.823\ \mu m$.

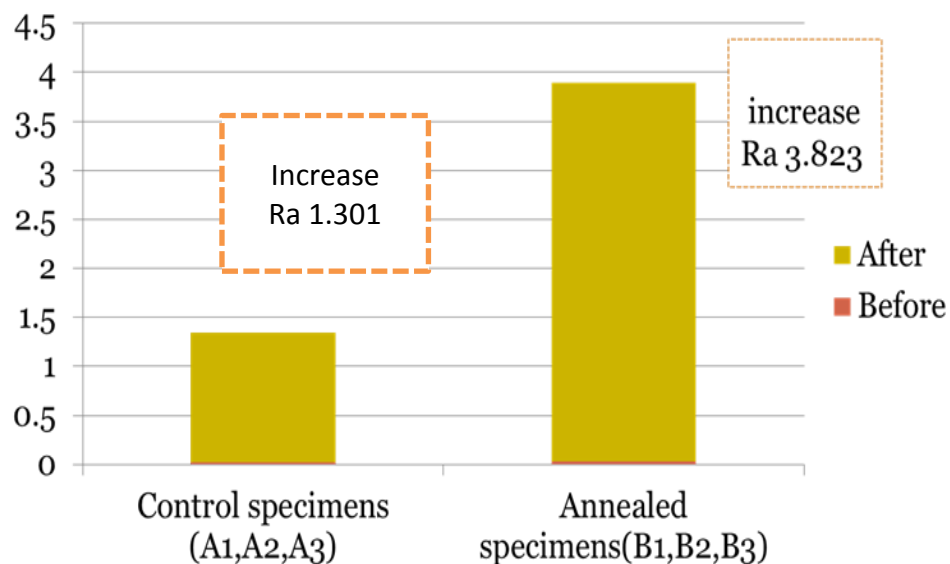


Figure 4.13: Graph bar for increment of surface roughness, Ra

Table 4.3: Specimens surface roughness value before and after electrochemical Erosion-corrosion test

Specimens		Surface roughness value (Ra)		
Control Data	before (μm)	after (μm)	Increment	
	A1	0.023	1.321	
	A2	0.026	1.206	1.301
	A3	0.020	1.446	
	Average	0.023	1.324	
Heat Treatment Effect	B1	0.031	3.760	
	B2	0.028	5.656	3.823
	B3	0.040	2.154	
	Average	0.033	3.856	

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 INTRODUCTION

For this project, effect of heat treatment has been evaluated on erosion-corrosion test of AISI Type 301 stainless steel. The result analysis comes from corrosion rate data, surface microstructure analysis and surface roughness analysis.

5.2 CONCLUSION

The following conclusions can be drawn from this study:

- (i) Annealed AISI Type 301 stainless steel have a higher corrosion rate than control AISI Type 301 stainless steel which decreasing of steel hardness contributed major factor to the increasing of corrosion rate.
- (ii) Analysis of surface microstructure show higher pitting and holes build on surface of annealed AISI Type 301 stainless steel than control AISI Type 301 stainless steel after undergo electrochemical erosion-corrosion test.
- (iii) Analysis of surface roughness, Ra test for annealed AISI Type 301 stainless steel having higher Ra value than control AISI Type 301 stainless steel due to corrosion built of pitting, holes and scratch.

- (iv) Aqueous slurries test environment also give effect to erosion-corrosion mechanism where solution of 3.5% NaCl, sand particles and velocity of solution can contribute to the increasing of corrosion rate.
- (v) Erosion-corrosion is complex phenomenon which involves the interaction between mechanical process of solid particle erosion and electrochemical process of corrosion was fully archive by undergone these experiment.

5.3 RECOMMENDATION

The investigation and study about the effect of heat treatment AISI Type 301 stainless steel on erosion corrosion behavior of steel in aqueous slurries environment should been continued.

- (i) Set up new industrial environment like erosion-corrosion at the piping bend and impeller of the pump.
- (ii) Using the others heat treatment process to give variety effects of heat treatment on erosion corrosion stainless steel.
- (iii) Using Scanning Electron Microscope (SEM) to analysts the effect of erosion corrosion at the grain boundaries of stainless steel.
- (iv) Set up various parameters of erosion corrosion test which cover the velocity of aqueous, temperature, sand particle size and solution pH.

REFERENCES

This guide is prepared based on the following references;

D.Lopez, J.P. Congote . . *Effect of particle velocity and impact angle on the corrosion-erosion of AISI 304 and AISI 420 stainless steels. Metallurgical and Materials Engineering Department, University of Sao Paulo*. Elsevier Academic Press: 2005.

Firas M.F.Al-Quran, Hossam Ibrahim Al-Itawi, . *Effect of the Heat Treatment on Corrosion Resistance and Microhardness of Alloy Steel. Department of Mechanical Engineering University of Jordan*. Eurojournals publishing: 2010

Fontana, M.G.1987. *Corrosion Engineering*, 3rd ed. USA: McGraw-Hill.

G-31-72, Standart practice for laboratory immersion corrosion testing of metals, Annual Book of ASTM Standarts, ASTM International, Philadelphia, PA, 2000,p.1.,Vol.3.0.2.

G5-94, Standart reference test method for making Potentiostatic and Potentiodynamic anodic polarization measurement, Annual Book of ASTM Standarts, ASTM International, Philadelphia, PA, 2004, p.1., Vol.3.0.2.

Oberg. E.F. and Ryffel, H, *1989 Machinery's Handbook 23rd Edition*, New York: Industrial Press Inc.

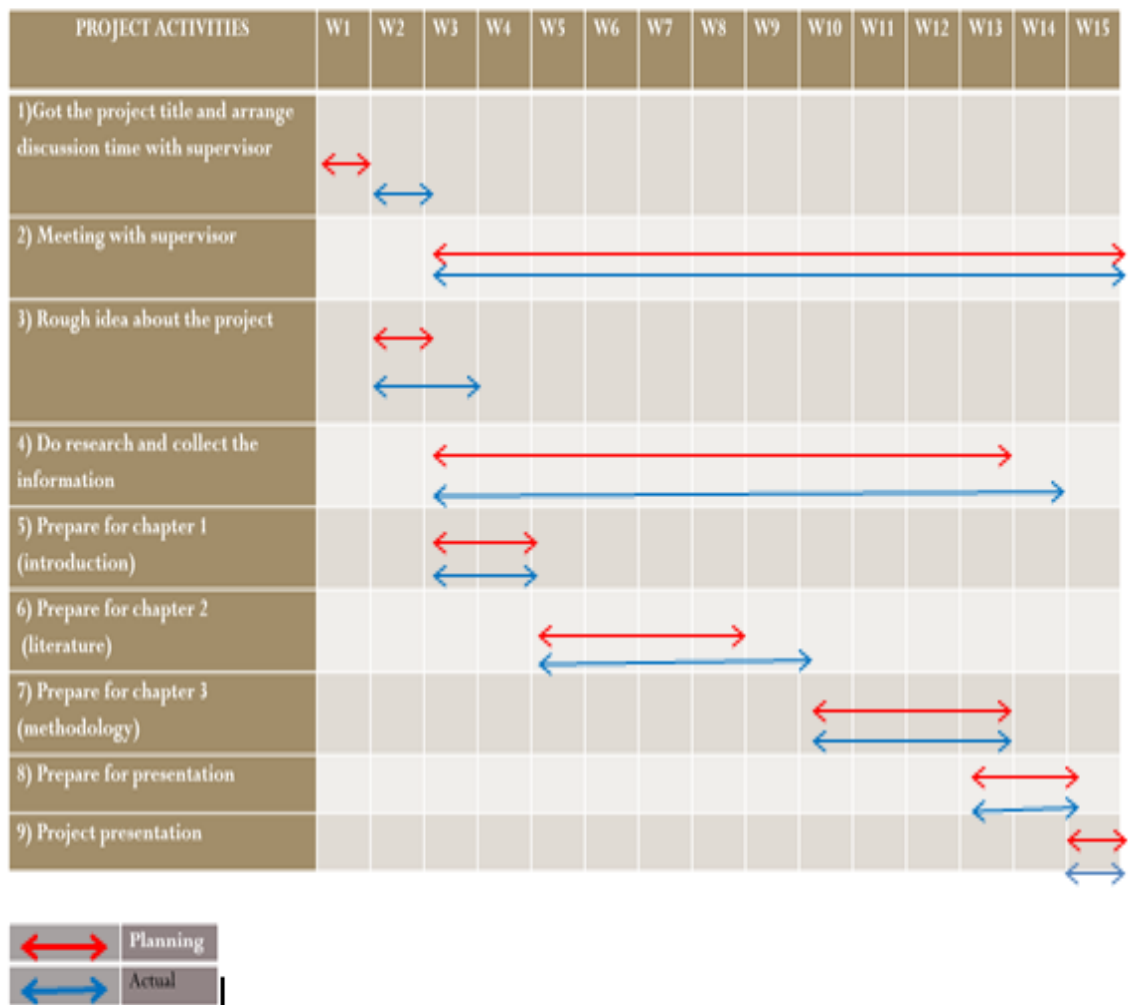
Pierre, R.R. 1999, *Handbook of Corrosion Engineering*, Mc Graw-Hill series in Material Science and Engineering.

S.S. Rajahram, T.J Harvey and R.J.K Wood. *Erosion-corrosion resistance of engineering materials in various test conditions. School of Engineering Sciences, University of Southampton*. Elsevier Academic Press: 2009.

- Sastri, V.S, Edward, G., and Mimoun, E. 2007. *Corrosion Prevention and Protection*. The Atrium: John Wiley & Sons, Ltd.
- Williams, D.F 1976. *Annu. Rev. Material Science*, 6, 237.
- Winston, R.R. and Herbert, H.U. 2008. *Corrosion and Corrosion Control*. Fourth ed. Canada: John Wiley & Sons, Inc.
- William, F.S. and Javad, H. 2006. *Foundation of Materials Science and Engineering*. Fourth ed. Boston Burr Ridge: McGraw-Hill.
- Williams, D.F. and Ann, R, 1976 *Material Science*.

APPENDICES (A)

Gantt chart /Project Schedule FYP 1



APPENDICES (B)

Gantt chart /Project Schedule FYP 2

