# ELECTROCHEMICAL STUDY OF HEAT TREATED SS304 STAINLESS STEEL IN SIMULATED BODY FLUID ENVIRONMENT

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# ELECTROCHEMICAL STUDY OF HEAT TREATED SS304 STAINLESS STEEL IN SIMULATED BODY FLUID ENVIRONMENT

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Thesis submitted in fulfilment of the requirement for the award of the degree of Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering UNIVERSITI MALAYSIA PAHANG

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# UNIVERSITI MALAYSIA PAHANG FACULTY OF MECHANICAL ENGINEERING

We certify that the project entitled "*Electrochemical Study of Heat Treated SS304 Stainless Steel in Simulated Body Fluid Environment.*" is written by *Nik Mohd Nazairen Bin Nik Cob.* We have examined the final copy of this project and in our opinion; it is fully adequate in terms of scope and quality for the award of the degree of Bachelor of Engineering. We herewith recommend that it be accepted in partial fulfilment of the requirements for the degree of Bachelor of Mechanical Engineering.

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

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Dedicated, truthfully for supports, encouragements and always be there during hard times, my beloved family.

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#### ABSTRACT

This thesis is entitled about the electrochemical study of heat treated SS304 stainless steel in simulated body fluid environment. Metallic materials are generally used for biomedical devices for various parts of the human body. When a metal device is implanted into human body, it is continually exposed to extracellular tissue fluid. The exposed metal surface of the implant undergoes electrochemical dissolution of material at a finite rate due to the interaction with body environment. This interaction can lead to either failure of the implant or have an adverse effect on the patient resulting in the rejection of the implant by the surrounding tissue or both. In conjunction with that, it is important to improve the corrosion resistance and wear properties by conducting the heat treatment processes. The objectives of this study are, to study the corrosion behaviour of the heat treated SS304 stainless steel in phosphate buffered saline (PBS) and hank's balanced salt solution (HBSS) as simulated body environment and also to investigate the effect of heat treatment process to the corrosion rate of SS304 stainless steel in PBS and HBSS as simulated body environment. Heat treatment process, which is carburizing has been performed with different temperature and time. Carburizing is a process of adding Carbon to the surface. This is done by exposing the part to a carbon rich atmosphere at an elevated temperature and allows diffusion to transfer the carbon atoms into steel. The electrochemical study was conducted using a potentiostat WPG1000, interfaced to a computer in PBS and HBSS used for simulating human body fluid conditions. SS304 stainless steel show increased in corrosion rate after the carburizing process in simulated body fluid due to the resultant formation of chromium carbides.

## ABSTRAK

Tesis ini membentangkan tentang kajian elektrokimia terhadap keluli tahan karat 304 yang terawat haba di dalam larutan berair (elektrolit) yang digunakan untuk mensimulasikan cecair tubuh manusia. Bahan-bahan logam biasanya digunakan untuk peranti bioperubatan untuk pelbagai bahagian badan manusia. Peranti logam yang ditanamkan ke tubuh manusia, adalah terus-menerus terdedah cecair rangkaian ekstraseluler. Permukaan logam implan yang terdedah mengalami kakisan elektrokimia pada peringkat terhad kerana interaksi dengan pesekitarannya. Interaksi ini boleh menyebabkan kegagalan implan atau mempunyai kesan buruk pada pesakit yang mengakibatkan penolakan implan dengan rangkaian sekitarnya atau kedua-duanya. Rawatan haba adalah penting untuk meningkatkan ketahanan terhadap pengaratan dan penghausan. Tujuan kajian ini adalah, untuk mempelajari perilaku pengaratan selepas rawatan haba besi tahan karat 304 di dalam cecair phosphate buffered saline(PBS) dan cecair hank's(HBSS) sebagai persekitaran badan simulasi dan juga untuk menyiasat pengaruh dari rawatan haba terhadap pengaratan besi tahan karat 304 di dalam PBS dan HBSS sebagai simulasi badan persekitaran. Rawatan haba yang telah dilakukan adalah karburasi dengan suhu yang berbeza dan masa yang berbeza. Karburasi adalah proses penambahan karbon ke permukaan sesuatu bahan. Hal ini dilakukan dengan mendedahkan sesuatu bahagian pada karbon yang tinggi pada suhu tinggi dan membenarkan resapan atom karbon ke dalam bahan tersebut. Penyelidikan elektrokimia yang dijalankan menggunakan WPG100 potensiostat, dihubungkan ke komputer dalam larutan berair (elektrolit) yang digunakan untuk mensimulasikan cecair tubuh manusia. Keluli tahan karat 304 menunjukkan peningkatan kadar pengaratan selepas proses karburasi di dalam simulasi cecair tubuh manusia disebabkan oleh pembentukan karbida kromium

# **TABLE OF CONTENTS**

	Page
EXAMINER'S 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	xii
LIST OF FIGURES	ixiii
LIST OF SYMBOLS	xvi
LIST OF ABBREVIATIONS	xvii

# CHAPTER 1 INTRODUCTION

1.1	Introduction	1
1.2	Problem Statement	2
1.3	Objectives of Study	3
1.4	Scopes Of Project	3

# CHAPTER 2 LITERATURE REVIEW

2.1	Introduction	4
2.2	Stainless Steel	4
2.3	Heat Treatment-Carburizing	6
2.4	Corrosion	7
	2.4.1 Passivity	7
2.5	Corrosion of Bio Implant	10
	2.5.1 Pitting Corrosion	12
	2.5.2 Crevice Corrosion	13
	2.5.3 Galvanic Corrosion	14

	2.5.4	Stress Corrosion Cracking (SCC)	14
	2.5.5	Fretting Corrosion	15
2.6	Human	Bodies As Corrosive Environment	16
2.7	Simulat	ed Body Fluid	19
	2.7.1	Phosphate buffered saline (PBS)	19
	2.7.2	Hank's balanced salt solution (HBSS)	20

# CHAPTER 3 METHADOLOGY

3.1	Introduction	21
3.2	General Experiment Procedure	22
3.3	Sample Preparation	23
3.4	Heat Treatment (Carburizing)	25
	3.4.1 Carburizing process	25
3.5	Surface Analysis	27
3.6	Electrochemical Test And Measurements	30
	<ul><li>3.6.1 Electrochemical Cell Set-Up</li><li>3.6.2 General Parameters</li></ul>	31 33
3.7	Corrosion Rate Analysis	34
	3.7.1 Tafel Plot	34

# CHAPTER 4 RESULTS AND DISCUSSION

4.1	Sample Characterization	36
4.2	Potentiodynamic Tests	43

# CHAPTER 5 CONCLUSION

5.1	Introduction	57
5.2	Conclusions	57
5.3	Recommendations	58

REFER	ENCES	59
APPEN	DICES	61
А	Gantt Chart /Project Schedule FYP 1	61
	Gantt Chart /Project Schedule FYP 1	62
		0_

# LIST OF TABLES

Table No	. Title	Page
2.1	SS304 stainless steel composition	5
2.2	Passivity region	8
2.3	A typical chemical composition of normal human blood plasma	16
2.4	Representative values for the primary ionic constituents of the major fluid compartments.	18
2.5	One of the common composition of PBS	19
2.6	HBSS and its components	20
2.7	Ion concentration of simulated body fluid	20
4.1	Types of specimens and process	55
4.2	Corrosion rates determined by Tafel extrapolation method in PBS	55
4.3	Corrosion rates determined by Tafel extrapolation method in HBSS	55

# LIST OF FIGURES

Figure	No. Title	Page
2.1	Phase Diagram of Stainless steel ternary	6
2.2	Case microstructure of stainless steel after air cooling from carburizing temperature	7
2.4	Passivity diagram	8
2.5	Passive surface illustration	9
2.6	Surface phenomenon of implant and cell biological fluid	11
2.7	Pitting corrosion mechanism	12
2.8	Crevice corrosion mechanism	13
2.9	Galvanic corrosion mechanism	14
2.10	Fretting scars on the taper neck	15
2.11	Human body compartment	17
3.1	General experiment procedure	22
3.2	SS304 stainless steel	23
3.3	Cutting machine band saw	23
3.4	SS304 stainless steel after cutting	24
3.5	SS304 stainless steel after drilling	24
3.6	Carburizing process configuration	25
3.7	Electric Furnace	26
3.8	Steel Container	26
3.9	Cold mounting process	27
3.10	Grinding process	28
3.11	Polishing process	28
3.12	Etching process	29

3.13	Inverted Microscope IM7000	29
3.14	Types of simulated body fluid solution	30
3.15	Electrochemical Test Cell	31
3.16	Type of Electrode	32
3.17	Interpreting Data and Result by using WPG Software Connected to WPG100 Potentiostat.	32
3.18	Sample parameters of Potentiodynamic Polarization	33
3.19	Tafel Plot	34
3.20	Tafel analysis using IVMAN software	34
4.1	Microstructure of SS304 stainless steel	37
4.2	Microstructure of SS304 stainless steel after carburizing for 6 housr at 900°C	38
4.3	Microstructure of SS304 stainless steel after carburizing for 6 hours at 950°C	39
4.4	Microstructure of SS304 stainless steel after carburizing for 8 hours at 900°C	40
4.5	Microstructure of SS304 stainless steel after carburizing for 8 hours at 950°C	41
4.6	Potentiodynamic polarization curves and Tafel extrapolation plot obtained in PBS at pH 7.4 and $37\pm2^{\circ}$ C for SS304 Stainless Steel (aerburising for 8 hours at 050°C) in contact	45
4.5	Microstructure of SS304 stainless steel after carburizing for 8 hours at 950°C	41
4.6	Potentiodynamic polarization curves and Tafel extrapolation plot obtained in PBS at pH 7.4 and $37\pm2^{\circ}$ C for SS304 Stainless Steel (carburizing for 8 hours at 950°C) in contact	45
4.7	Potentiodynamic polarization curves and Tafel extrapolation plot obtained in PBS at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 900°C) in contact	46

- 4.8 Potentiodynamic polarization curves and Tafel extrapolation plot 47 obtained in PBS at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 950°C) in contact
- 4.9 Potentiodynamic polarization curves and (b) Tafel extrapolation 48 plot obtained in PBS at pH 7.4 and 37±2°C for SS304 stainless steel(carburizing for 6 hours at 900°C) in contact
- 4.10 Potentiodynamic polarization curves and Tafel extrapolation plot 49 obtained in PBS at pH 7.4 and 37±2°C for SS304 stainless steel in contact
- 4.11 Potentiodynamic polarization curves and Tafel extrapolation plot 50 obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 950°C) in contact
- 4.12 Potentiodynamic polarization curves and Tafel extrapolation plot 51 obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 900°C) in contact
- 4.13 Potentiodynamic polarization curves and Tafel extrapolation plot 52 obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 950°C) in contact
- 4.14 Potentiodynamic polarization curves and Tafel extrapolation plot obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 53 stainless steel (carburizing for 6 hours at 900°C) in contact
- 4.15 Potentiodynamic polarization curves and Tafel extrapolation plot 54 obtained in HBSS at pH 7.4 and 37±2°C for SS304 stainless steel in contact
- 4.16 Graph of SS304 stainless steel corrosion rate in PBS and HBSS 56

# LIST OF SYMBOLS

$eta_a$	Anodic Slopes			
$eta_c$	Cathodic Slopes			
BaCO <sub>3</sub>	Barium Carbonate			
CaCl <sub>2</sub>	Calcium Chloride			
$CO_2$	Carbon Dioxide			
СО	Carbon Monoxide			
Cr	Chromium			
$E_{corr}$	Corrosion Potential			
$E_p$	Primary Passivation Potential			
Fe	Ferum			
HCl	Hydrochloric acid			
HNO <sub>3</sub>	Nitric Acid			
$I_c$	Critical Current Density			
$I_p$	Passive Current Density			
LogI	Log Current			
KCl	Kalium Chloride			
KH2PO4	Potassium dihydrogen			
MgSO <sub>4</sub>	Magnesium Sulphate			
NaCl	Natrium Chloride			
Na <sub>2</sub> HPO <sub>4</sub>	Disodium phosphate			
NaHCO <sub>3</sub>	Sodium Bicarbonate			

# LIST OF ABBREVIATIONS

ASTM	American Standard Testing Method		
Ca	Calcium		
CE	Counter electrode		
CoCrMo	Cobalt-chromium-molybdenum		
FYP	Final year project		
HBSS	Hank's balanced salt solution		
PBS	Phosphate buffered saline		
RE	Reference electrode		
RP	Corrosion resistance		
SCC	Stress corrosion cracking		
WE	Working electrode		

#### **CHAPTER 1**

## **INTRODUCTION**

## 1.1 BACKGROUND OF STUDY

Nowadays, foreign materials have been routinely implanted in human body. The majority of these materials are made from metal alloys, such as stainless steels, cobalt chromium based alloys and titanium alloys. Major application of these materials are used as dental implants, orthopedic fracture plates, spinal rods, cranial plates, joint replacement prostheses and others. As the global population increases in age, the application of implants has increased rapidly.

The human body is not an environment that one would consider hospitable for an implanted metal alloys. Body fluid consists of aerated solution containing approximately 1% sodium chloride, together with minor amounts of other salts and organic compounds such as platelets, fat globules and chemical substances at a pH of 7.4. While it is well known that chloride solutions are among the most aggressive and corrosive to metals and ionic composition, protein concentration in body complicate the biomedical corrosion. When a metal device is implanted into human body, it is continually exposed to extracellular tissue fluid. The exposed metal surface of the implant undergoes and electrochemical dissolution of material at a finite rate due to the interaction with surrounding environment. This interaction can lead to either failure of the implant or have an adverse effect on the patient resulting in the rejection of the implant by the surrounding tissue or both.

The fundamental requirement for choosing a metallic implant material is that it can be biocompatible, that is not exhibiting any toxicity to the surrounding biological system. Biocompatible materials play an important and a critical role in manufacturing a variety of prosthetic devices in a modern world. Prosthetic devices are artificial replacements that are used in a biological system, such as the human body in an effort to provide the function of the original part.

For any material to act as a biomaterial, it must satisfy two essential characteristics that is bio-functionality and biocompatibility. Bio-functionality is related to a set of properties which allow a device to perform a required function, while biocompatibility refers to the ability of the device to continue to perform that function, effectively and as long as necessary, in or on the body. The biocompatibility of a material is affected by many factors, one of which and the most important one, is its corrosion resistance in a highly aggressive, high chloride containing body fluid environment.

The material must withstand the body environment and not degrade to a point that it cannot function in the body as intended. Materials used in the human body must not be treated or used in a configuration that would degrade the corrosion behavior. The implantation of biomaterials into the human body allows it to increase the quality of life and should have biological and chemical stability to improve functions of the human body.

## **1.2 PROBLEM STATEMENT**

Metallic materials are generally used for biomedical devices for various parts of the human body. The interaction between the surrounding physiological environment and the surface of the implant materials can lead to bio-implant corrosion such as Pitting and Galvanic Corrosion. It is important to improve the corrosion resistance and wear properties by conduct the heat treatments process.

# **1.3 OBJECTIVES OF STUDY**

The objectives of this study are:

- (i) To study corrosion behavior of the heat treated SS304 stainless steel in PBS and HBSS as simulated body environment.
- (ii) To investigate the effect of heat treatment process to the corrosion rate of SS304 stainless steel in PBS and HBSS as simulated body environment.

# **1.4 SCOPES OF PROJECT**

The scopes of this study include:

- (i) Sample preparation for electrochemical test.
- (ii) Heat treatment process (carburizing).
- (iii) Electrochemical test using potentiodynamic polarization technique.
- (iv) Surface analysis of specimen by using Inverted Microscope.
- (v) Corrosion rate analysis using IVMan software.

### **CHAPTER 2**

## LITERATURE REVIEW

## 2.1 INTRODUCTION

The use of materials as part of surgical implant is not new. It has been reported that substitution of bone parts in the body has been done since the late 18<sup>th</sup> to 19<sup>th</sup> century. In this era, bronze or copper were the main materials utilized. The implants used were successful but they have the problem of copper ion poisoning effect in the body. There were no other suitable materials for implantation were studied other than the copper and bronze until the mid-nineteenth century (Williams, 1987).

By the mid-nineteenth century, the medical sciences have already progressed in serious attempts to repair body parts with foreign materials. Following the introduction of stainless steels and cobalt chromium alloys in 1930s, greater success was achieved in fracture fixation and the first joint replacement surgeries were performed.

Biomaterial can be defined as a nonviable material used in a medical device, intended to interact with biological systems (Williams, 1987). Metallic components are generally used for orthopedics applications, since high strength and corrosion resistance are needed for bone repair and replacement.

## 2.2 STAINLESS STEEL

Metals are used as biomaterials due to have high wear and corrosion resistance. The most popular metallic biomaterials such as stainless steel, cobalt base alloys such as cast CoCrMo and titanium alloys. The first stainless steel utilized for implant fabrication was the 18-8 (type 302 in modern classification) which is stronger and more resistance to corrosion than vanadium steel (first metal alloys developed).

Stainless steels, in particular type SS304, are the most familiar and most frequently used alloy in the stainless steel family. Table 2.1 shows the SS304 stainless steel composition.

These alloys considered for a wide variety of applications because of the following properties:

- (i) Resistance to corrosion
- (ii) Ease of fabrication
- (iii) Excellent formability
- (iv) Beauty of appearance
- (v) Ease of cleaning
- (vi) High strength with low weight

 Table 2.1: SS304 stainless steel composition

Element	Composition
carbon	0.08 max.
manganese	2.00 max.
phosphorus	0.045 max.
sulfur	0.030 max.
silicon	0.75 max.
chromium	18.00-20.00
nickel	8.00-12.00
nitrogen	0.10 max.
iron	Balance

Source: Thomas (1991)

Stainless steel implants are used as temporary implants to help bone healing, as well as fixed implants such as for artificial joints. Typical temporary applications are cranial plates, orthopedic fracture plates, dental implants, spinal rods, joint replacement prostheses, stents, and catheters. Figure 2.1 shows the phase diagram of stainless steel ternary. The main composition is chromium and nickel.



Figure 2.1: Phase diagram of stainless steel ternary.

Source: Adler (1991)

#### 2.3 HEAT TREATMENT-CARBURIZING

Carburizing is a process of adding carbon to the surface. This is done by exposing the part to a carbon rich atmosphere at an elevated temperature and allows diffusion to transfer the carbon atoms into steel. This diffusion will work only if the steel has low carbon content, because diffusion works on the differential of concentration principle.

There are three well-known methods of carburizing. These methods introduce carbon by the use of solid compounds (pack carburizing), liquids (salt bath carburizing) and gas (atmospheric gas, plasma and vacuum carburizing). Figure 2.2 shows the case microstructure of stainless steel after air cooling from carburizing temperature.



Figure 2.2: Case microstructure of stainless steel after air cooling from carburizing temperature

Source: Iskander (1981)

# 2.4 CORROSION

Corrosion is defined as the destruction or deterioration of a material because of reaction with its environment. We have all seen corrosion and know that the process produces a new and less desirable material from the original metal and can result in a loss of function of the component or system.

The iron atom can lose some electrons and become a positively charged ion. This allows it to bond to other groups of atoms that are negatively charged. Oxygen, in the presence of water, accepts electrons to form hydroxyl ions. Oxygen dissolves quite readily in water and because there is usually an excess of it, reacts with the iron hydroxide.

# 2.41 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 2.2 shows the passivity region. Passivity can be conveniently divided into three regions: active, passive, and transpassive.

Regions	Behavior
Active	Identical as normal metal.
Passive	Slight increases 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.

	<b>A A</b>	D '	••.	•
Table	2.2:	Passi	ivity	region

Source: Fontana (1986)



Figure 2.3: Passivity diagram

Source: Thomas (1991)

Figure 2.3 shows 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 becomes passive and exhibits the passive current density  $i_p$ . The critical current density for passivation is  $i_c$ .

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 bio-logical environment. Consequently, the passive film kinetically retards the rate of dissolution by many orders of magnitude. Figure 2.4 shows the protectiveness of the passive film is determined by the rate of ion transfer through the film, as well as the stability of the film against dissolution. A variety of factors can influence ion transport through the film, such as the film's chemical composition, structure, thickness and presence of defects. Principally, the nature and stability of a passive film on a particular metal or alloy depend on the environmental conditions, such as the composition of the electrolyte, the redox conditions, the exposure time and temperature.



Figure 2.4: Passive surface illustration

Source: Virtanen (2007)

#### 2.5 CORROSION OF BIO IMPLANT

Corrosion is the degradation of a material properties or mass over time due to environmental effects. In biomaterials, corrosion is one of the major processes that cause problems when metals and alloys are used as implants in the body. The most fundamentally important is the interaction between the surrounding physiological environment and the surface of the implant itself. This interaction can lead to either the failure of the implant to function as it was intended, or have an adverse effect on the patient resulting in the rejection of the implant by the surrounding tissue, or both (Williams, 1976)

Corrosion of implants in the aqueous medium of body fluids takes place via electrochemical reactions. When a metal device is implanted into the human body, it is continually exposed to extracellular tissue fluid. The exposed metal surface of the implant undergoes an electrochemical dissolution, due to interactions with the surrounding environment. In the case of the human body, this environment can contain water, complex organic compounds, dissolved oxygen, sodium, chloride, bicarbonate, potassium, calcium, magnesium, phosphate, amino acids, proteins, plasma, lymph, saliva (Williams, 1976)

Upon implantation, the tissue environment is disturbed, disrupting blood supply to the surrounding tissue and the ionic equilibrium. The initiation of corrosion can be the result of various conditions existing along the implant surface, whether it is the formation of localized electrochemical cells resulting in pitting attack, or crevice corrosion.



Figure 2.5: Surface phenomenon of implant and cell biological fluid

# Source: Konttinen(1992)

The physiology of the human body functions to prevent and remove any foreign substances that are present in the body. When foreign substances, in this case metallic implants are detected, the body secretes antibodies (proteins) which act as powerful oxidizing agents (Williams, 1976). Figure 2.5 shows these proteins concentrate around the implant material and absorb into the biomaterial. This attacking mechanism results in corrosion rate enhancement of the metallic implants. Besides the influence of the environment, the rate of corrosion is also dependent on the type of materials.

#### 2.5.1 Pitting Corrosion

Pitting corrosion is a type of localized corrosion caused by local dissolution of the passive film and the formation of cavities surrounded by an intact passivated surface. Pitting usually occurs in halide containing solutions, of which, for most metals, chloride ion is the most aggressive. Surgical stainless steel shows higher susceptibility than Co- and Ti-based alloys to pitting and crevice corrosion in halide solutions. Corrosion damage is frequently observed at screw holes after removal of temporary stainless steel plates (Milosev,2007). The detection and meaningful monitoring of pitting corrosion usually represents a major challenge. Pitting failures can occur unexpectedly, and with minimal overall metal loss. Furthermore, the pits may be hidden under surface deposits or corrosion products. Figure 2.6 shows the pitting corrosion mechanism.



Figure 2.6: Pitting Corrosion mechanism

Source: Alison (2008)

#### 2.5.2 Crevice Corrosion

Crevice corrosion is a type of localized corrosion closely related to pitting corrosion. It occurs preferentially in regions on the metal surface where mass transfer is limited. At these occluded areas the concentration of aggressive chloride ions, decrease in pH value and depletion of oxygen can rapidly lead to activation of the surface. Stainless steel is the most susceptible of the three alloy groups discussed to crevice-induced localized corrosion (Virtanen,2007) .Therefore crevice corrosion may lead to sudden devastating failure of the metal in service. Crevices make a chemical environment which is different from that of freely exposed surfaces and therefore accelerate corrosion. This environment keeps moisture, traps pollutants, concentrates corrosion products and meanwhile excludes oxygen. Most cases of crevice corrosion occur in near-neutral solutions in which dissolved oxygen is the cathode reactant. Figure 2.7 shows the crevice corrosion mechanism that occurs to metal.



Figure 2.7: Crevice corrosion mechanism

Source: Crevice corrosion mechanism & prevention (2009)

#### 2.5.3 Galvanic Corrosion

Galvanic corrosion occurs when dissimilar metals are in direct electrical contact in corrosive solutions or atmospheres. Enhanced corrosion of the less noble metal takes place, whereas the corrosion rate of the more noble metal is reduced or even completely suppressed. However, once the metal or alloy is covered by a protective passive layer, the corrosion potential is typically that of a more noble metal than that of the bare metal surface (Barrena,2007). Therefore, judging solely by the standard potential values, one would overestimate the danger of galvanic corrosion. Since passive films act as very efficient barriers to corrosion, the danger of galvanic corrosion is lower for passive materials than for the coupling of actively corroding metals. Relative movement between the implant and the tissue, for example, at a bearing surface or on a cyclically loaded implant, will cause mixing at the interface and will modify the composition of the electrolyte and may modify the surface of the alloy. Figure 2.8 shows the galvanic corrosion mechanism.



Figure 2.8: Galvanic corrosion mechanism

Source: Roberge (1999)

## 2.5.4 Stress Corrosion Cracking (SCC)

SCC is the cracking induced from the combined influence of tensile stress and a corrosive environment. The impact of SCC on a material usually falls between dry cracking and the fatigue threshold of that material. The required tensile stresses may be in the form of directly applied stresses or in the form of residual stresses.

## 2.5.5 Fretting Corrosion

Fatigue occurs under conditions of large number of cyclic loading. Surface fatigue wear is due to subsurface microcracking followed by breaking off of large pieces of material under repeated loading and sliding cycles. Abrasive wear is a process in which particles are pulled off from one surface and adhere to the other during sliding. This effect can be minimized in biomaterials by using good surface finish of the materials. Fretting fatigue occurs under conditions of cyclic loading and cyclic friction loading, as for example in the event of repeated microscopic motion between screws and plates (Trebse, 2007).

Fretting corrosion is a form of damage which occurs at the interface of two close fitting surfaces when they are subjected to slight oscillatory slip and joint corrosion action. The damage is mostly restricted to local site and the debris will accumulated locally and lead to increase in stress. Most implants usually fail due to corrosion fatigue and fretting corrosion. Figure 2.9 shows the fretting corrosion scars on the tapper neck.



Figure 2.9: Optical micrograph of fretting scars on the taper neck

Source: Salo(2007)

#### 2.6 HUMAN BODIES AS CORROSIVE ENVIRONMENT

The corrosive environment of body fluids can generally be simulated by a 0.9% NaCl solution containing small amounts of other inorganic salts at a temperature of 37°C. The chemical environment of blood plasma is highly aggressive for many metals and alloys, due especially to the presence of high concentration of chloride ions and their ability to induce localized corrosion (Nakamura, 2003). Other ions may also contribute to the corrosion process, either as accelerators or inhibitors. For instance, layers of Ca-phosphates precipitate on titanium and titanium-based alloys in simulated body fluids, as well as in vivo, but the effect of such deposit layers on the corrosion process has scarcely been considered. Table 2.3 shows the chemical composition of normal human blood plasma.

 Table 2.3: A typical chemical composition of normal human blood plasma

Ion	Concentration (mmol/l)
Na <sup>+</sup>	142.0
$\mathbf{K}^+$	5.0
$\mathrm{Mg}^{2+}$	1.5
$\tilde{Ca^{2+}}$	2.5
Cl	103.0
HCO <sub>3</sub>	27.0
$HPO_4$	1.0
$\mathbf{SO_4}^{2-}$	0.5

Source: Virtannen(2007)

The body temperature of 37°C can accelerate electrochemical reactions and even change the mechanism of corrosion from that occurring at room temperature. Studies conducted at room temperature may lead to underestimation of the risk of corrosion and direct extrapolation to body temperature is not straightforward. For example, the above mentioned precipitation of Ca-phosphates is much more likely to occur at 37°C than at room temperature.

In addition to inorganic species, body fluids contain different types of biomolecules and cells, which may attach to the biomaterial surface and affect the surface reactions. Proteins are a primary constituent of the synovial fluid in total joints with other organic components such as hyaluronic acid and lubricin. The formation of a protein-containing biofilm on the metal surface enhanced the corrosion process of the base alloy (Neville and Dowson, 2007). Although the biofilms also lubricate the surface, the total material degradation was increased due to increased corrosion (Neville and Dowson, 2007). Another significant factor determining the corrosion behavior of metals is the pH of the environment. Typically, changes in the pH value in the body fluids are relatively small since the fluids are buffered. On implantation, the pH of the tissue surrounding the implant may decrease to values around 5, and then recovers to 7.4 within weeks (Ethridge, 1975). Figure 2.10 shows the human body compartment.



Figure 2.10: Human body compartment

#### Source:Krider(2006)

The concentration of released Fe, Cr and Ni ions from stainless steel immersed in Hank's physiological solution increased in the presence of various proteins in the following order: fibrinogen < globulin < transferin < albumin (Milos, 2003) .The enhancement of dissolution rate in the presence of proteins can be explained by the formation of complexes between metal ions and proteins (Jacobs, 1990). These complexes can be transported away from the immediate vicinity. To retain the equilibrium the dissolution rate of a base metal increases and, consequently, suppresses the formation of the passive layer (Milos, 2003). Table 2.4 shows the representative values for the primary ionic constituents of the major fluid compartments.

**Table 2.4:** Representative values for the primary ionic constituents of the major fluid compartments.

Electrolyte	Plasma, (mEq/L) [molarity]	Plasma Water (mEq/L) [molality]	Interstitial Fluid (mEq/L)	Intracellular Fluid (mEq/L)
cations:				
sodium	142	153	145	10
potassium	4	4.3	4	160
calcium	5	5.4	5	2
magnesium	2	2.2	2	26
Total cations:	153	165	156	198
anions:				
chloride	101	108.5	114	3
bicarbonate	27	29	31	10
phosphate	2	2.2	2	100
sulphate	1	1	1	20
organic acid	6	6.5	7	
protein	16	17	1	65
Total anions:	153	165	156	198

Source: Joe Patlak(1990)
### 2.7 SIMULATED BODY FLUID

#### 2.7.1 Phosphate buffered saline (PBS)

PBS is often used in cell biology experiments to maintain the osmolarity of the cells. It contains salt ions, which balances the amount of salt ions inside the cell. If the cells are immersed into a solution that has too many salt ions, water will leak out from the cell, causing the cell to shrink. Conversely, if the cells are immersed into a solution that has too few salt ions, water will enter the cell, causing the cell to burst. Therefore, it is critical when performing cell biology experiments to maintain the cells at a certain osmolarity. PBS is at the correct osmolarity to keep the cells in an isotonic state.

PBS is also often used as a buffer in biochemistry experiments to maintain the pH of proteins. It can be used to dissolve peptide or protein samples directly, and to store the protein or peptide in that solution. Proteins require a certain pH range to maintain neutrality or charges on certain amino acids, which allow the structure of the protein to be maintained in its native state. Otherwise, the structure of the protein may become denatured. Besides using PBS to directly dissolve proteins, PBS is also highly useful as a buffer to maintain the pH in biochemical assays such as Western blots. Table 2.5 shows the composition of PBS.

(mmol/L)	(g/L)
137	8.00
2.7	0.20
10	1.44
1.76	0.24
	2.7 10 1.76

 Table 2.5: One of the common composition of PBS

Source: Medicago (2009)

# 2.7.2 Hank's Balanced Salt Solution (HBSS)

HBSS can be used as a temporary diluting, washing, irrigating or transporting solution for cell or tissue culture. It provides a buffering system to maintain the physiological pH range and osmotic balance of the culture media, and provides cells with a source of water and essential inorganic ions, and a carbohydrate as an energy source. Table 2.6 shows the HBSS components.

 Salt
 0.185 CaCl <sub>2</sub> .2H <sub>2</sub> O
0.06 KH <sub>2</sub> PO <sub>4</sub> (anhyd.)
0.04788 Na <sub>2</sub> HPO <sub>4</sub> (anhyd.)
0.40 KCl
8.00 NaCl
0.09767 MgSO4 (Anhyd.)
0.35 NaHCO <sub>3</sub>
1.00 D-Glucose

Table 2.6: HBSS and its components

Source: Medicago (2009)

Table 2.7: Ion concentration of simulated body fluid

Ion	HBSS	PBS	
Na <sup>+</sup>	142.77	157.17	
$\mathbf{K}^+$	5.85	4.17	
$Mg^{2+}$	0.81	-	
$Ca^{2+}$	2.5	-	
Cl	145.87	140.70	
HCO <sub>3</sub> <sup>2</sup>	4.17	-	
HPO4 <sup>2</sup>	1.0	9.58	
$H_2PO_4^{2}$	0.44	1.47	
$\mathrm{SO}_4^{2}$	0.81	-	

Source: Diener (2010)

### **CHAPTER 3**

## METHODOLOGY

## 3.1 INTRODUCTION

Failure of artificial joints and bone fixation devices such as fracture and loosening becomes notable after 5-10 years of implantation(Steinemann,1996). These mechanical degradations are often caused by fatigue, fretting fatigue, and wear accelerated by corrosion. This fact suggests that the corrosion progresses little by little for many years on the surface of material attaching to the surrounding tissue. Thus, the corrosion factors in the tissue, which will attach to the implanted material, should be clarified to improve and evaluate the corrosion resistance of metallic biomaterials.

By referring to the literature review, corrosion is due to an electrochemical mechanism, so it is clear that electrochemical techniques can be used to study corrosion reactions and mechanisms. Electrochemical methods of corrosion testing had been carried out to study corrosion phenomena of heat treated SS304 stainless steel in simulated body environment.

# 3.2 GENERAL EXPERIMENT PROCEDURE



Figure 3.1: General experiment procedure

### **3.3 SAMPLE PREPARATION**

In this project, SS304 stainless steel as shown in Figure 3.2 is the main material had been used as a specimen. Ten specimens of SS304 stainless steel were been cut in the form of cylinder which is 15 mm width and 10 mm height. Figure 3.4 shows the specimen after cutting process. Each sample then had been immersed in two simulated body fluids; PBS and HBSS.

The entire specimens had been prepared from raw material. For stainless steel, the samples had been cut by using band saw as shown in Figure 3.3. After cutting process, the specimen were been drilled at the centre as shown in Figure 3.5.



Figure 3.2: SS304 stainless steel



Figure 3.3: Cutting machine band saw



Figure 3.4: SS304 stainless steel after cutting



Figure 3.5: SS304 stainless steel after drilling

### **3.4** Heat Treatment (Carburizing)

Carburizing is a process of adding carbon to the surface. This is done by exposing the part to a carbon rich atmosphere at an elevated temperature and allows diffusion to transfer the carbon atoms into steel. This diffusion will work only if the steel has low carbon content, because diffusion works on the differential of concentration principle.

### 3.4.1 Carburizing process

Some amounts of carburizing compound were placed inside steel container as shown in Figure 3.8. The specimens were placed above the compound, in the middle of the container, with the longitudinal direction parallel to top and bottom of the container. The rest of the container was placed with carburizing compound and container was tapped to ensure that the compound is densely packed to prevent air trap within it. The containers were closed with its top cover and electric furnaces were heated up to 900°C. The specimen was placed in the furnace using tongs and heat resisting gloves as shown in Figure 3.7. Specimens were heated for 6 hours at 900°C. Then, the specimens were left to cool outside the furnace. For other specimens carburizing process time and temperature, we used the Figure 3.6.



Figure 3.6: Carburizing process configuration



Figure 3.7: Electric Furnace



Figure 3.8: Steel Container

### 3.5 SURFACE ANALYSIS

Surface analysis had been carried out after heat treatment process. Observation on the surface microstructure and morphology of the specimen for determine the type of corrosion had been observed by using Inverted Microscope IM7000 series in the UMP mechanical laboratory at Pekan campus as shown in Figure 3.13. Before the surface analysis process, the specimen underwent cold mounted, grinded, polished and etched process.

Mounting of specimens is needed to allow them to be handled easily. First, copper wired was placed inside the hole, at the centre of the specimens. After that, the specimens were placed inside the mounting cup. The correct amounts of two components which is powder transparent and cold-curing resin liquid had been weighted carefully with same weight. The two components are then mixed thoroughly and poured over the specimens. The specimens were placed inside the cold mounting machine for 20 minutes. Figure 3.9 shows the cold mounting process.









Figure 3.9: Cold mounting process: (a) copper wire was placed inside the specimens hole, (b) specimens was placed inside mounting cup, (c) specimens was placed inside mounting machine, (d) complete specimens after cold mounting.

Surface damaged of specimens after cutting process had been removed by grinding and polishing. The specimens were grinded using 180, 240, 320 and 400 SiC paper disc and lubricated using water as shown in Figure 3.10. Finally, the specimens had been polished using polish machines with three different type of lubricant which is 6  $\mu$ , 3  $\mu$  and 1  $\mu$  PC diamond. Figure 3.11 shows the polishing process with different lubricant.



Figure 3.10: Grinding process





**Figure 3.11:** Polishing process with: (a) 6 μ PC diamond lubricant, (b) 3 μ PC diamond lubricant, (c) 1 μ PC diamond lubricant



Figure 3.12: Etching Process

Microstructure of the stainless steel specimens had been revealed by etching process. Stainless steel had been etched by mixing of 30 ml hydrochloric acid (HCl), 30 ml glycerol and 10 ml nitric acid (HNO<sub>3</sub>) for 10 minutes as shown in Figure 3.12.



Figure 3.13: Inverted Microscope IM7000

### 3.6 ELECTROCHEMICAL TEST AND MEASUREMENTS

The electrochemical study was conducted using a potentiostat WPG1000, interfaced to a computer. The basic instrumentation needed for the electrochemical tests includes:

- (i) Test or working electrode (WE)
- (ii) Counter electrodes (CE)
- (iii) Reference electrode (RE)
- (iv) Test cell
- (v) Potentiostat
- (vi) Computer with software program

The tests was conducted in an aqueous solution (electrolyte) used for simulating human body fluid conditions which are; PBS and HBSS as shown in Figure 3.13. All of the solution had been stored at the required temperature to prevent from any condition which might affect the solution. PBS and HBSS had been stored at 4°C. Freshly prepared solution was used for each experiment.



Figure 3.14: Types of simulated body fluid solution

#### 3.6.1 Electrochemical Cell Set-Up



Figure 3.15: Electrochemical test cell

The working electrodes were connected to the specimens which is SS304 stainless steel, a graphite wire as the counter electrode, CE (red cable) and saturated calomel electrode as the reference electrode, RE (blue cable) as shown in Figure 3.15 and Figure 3.16. Saturated calomel electrode was used as reference electrode as it is provides a stable 'reference' 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 a graphite rod.

The luggin capillary connected as reference electrode that allows sensing of the solution potential close to the working electrode without the adverse effects that occur when the large reference electrode is placed near the working electrode. A constant electrolyte temperature of  $37 \pm 2^{\circ}$ C were maintained using hot plate. The pH of the solution was precisely maintained at 7.4 based on the nature of the human body solution. All the potential measurement had been analyzed by using Wonatech Potentiostat (WPG 100) as shown in Figure 3.17(a) and Figure 3.17(b).



Figure 3.16: Type of Electrode





**(b)** 

Figure 3.17 (a) and (b) : Result and data interpreting by using WPG Software connected to WPG100 Potentiostat.

#### 3.6.2 General Parameters

There are a number of parameters which must be specified and can be controlled by this IVMan software. The first is the initial and final potentials which define the 'path' 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 we use is 2 mV/S. Several particular parameters such as temperature, sampling time, voltage, density and area of sample will be set with desired value for use in the test. The density of SS304 stainless steel was set up to 7.94 g/cm<sup>3</sup>, sample area is 1.767 cm<sup>2</sup> and equivalent weight is 25.12.



Figure 3.18: Sample parameters of Potentiodynamic Polarization

#### 3.7 CORROSION RATE ANALYSIS

#### 3.7.1 Tafel plot

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

Figure 3.20 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 reactions was shown as straight lines. The curved line is the total current which is the sum of the anodic and cathodic currents. Sharp point in the curve as shown in Figure 3.19 is actually the point where the current changes signs as the reaction changes from anodic to cathodic, or vice versa. 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.

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. Suppose the anodic reaction releases too many electrons into the metal. Excess electrons shift the potential of the metal more negative, which slows the anodic reaction and speeds up the cathodic reaction. The equilibrium potential assumed by the metal in the absence of electrical connections to the metal is called the Open Circuit Potential,  $E_{oc}$ . In electrochemical corrosion experiments, the first step is the measurement 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 specimens using IVMAN software. Figure 3.21 shows the Tafel analysis using IVMAN software to get the specimens corrosion rate value



Figure 3.19: Tafel Plot

## Source: Hench (1985)



Figure 3.20: Tafel analysis using IVMAN software

# **CHAPTER 4**

# EXPERIMENTAL RESULTS AND DISCUSSION

## 4.1 Sample Characterization

Microstructure of the specimen had been revealed under Inverted Microscope IM7000 series before electrochemical tests. Stainless steel had been etched by mixing of 30 ml hydrochloric acid (HCl), 30 ml glycerol and 10 ml nitric acid (HNO<sub>3</sub>) for 10 minutes.

The following figure shows the microstructure of SS304 stainless steel after the etching process using Inverted Microscope at magnifications 100x and 200x. Five samples had been selected, first the control specimen, which is non heat treated SS304 stainless steel and the others four specimens which have been heat treated SS304 stainless steel (carburizing at different time and temperature).





Figure 4.1: Microstructure of SS304 stainless steel at; (a) Magnification 100x (b) Magnification 200x





**Figure 4.2:** Microstructure of SS304 stainless steel after carburizing for 6 hours at 900°C at; (a) Magnification 100x (b) Magnification 200x





**Figure 4.3:** Microstructure of SS304 stainless steel after carburizing for 6 hours at 950°C at; (a) Magnification 100x (b) Magnification 200x





**Figure 4.4:** Microstructure of SS304 stainless steel after carburizing for 8 hours at 900°C at; (a) Magnification 100x (b) Magnification 200x





**Figure 4.5:** Microstructure of SS304 stainless steel after carburizing for 8 hours at 950°C at; (a) Magnification 100x (b) Magnification 200x

The carburization process is the most widely used method for the case hardening of steels. The surface hardness depends on the degree of surface carbon enrichment which is determined by the heating conditions and the composition of the atmosphere. The carburization process diffuses carbon into the surface of stainless steel and results in significant hardening by achieving a super saturation of carbon within the matrix of austenitic stainless steels.

In this process, the part that is to be carburized is packed in a steel container so that it is completely surrounded by granules of charcoal. The charcoal is treated with an activating chemical such as barium carbonate (BaCO<sub>3</sub>) that promotes the formation of carbon dioxide (CO<sub>2</sub>). This gas in turn reacts with the excess carbon in the charcoal to produce carbon monoxide; CO. Carbon Monoxide reacts with the low-carbon steel surface to form atomic carbon which diffuses into the steel. Carbon monoxide supplies the carbon gradient that is necessary for diffusion.

From figure 4.1 to 4.5, we can see the carbon content at the surface of the specimens increase gradually, when the time and temperature of the carburizing process increased. If the total carbon content in surface the layer is compared with the carbon in solid solution it follows that the carbon supplied to the metal combines with the iron and chromium largely into carbides. The carbides in the austenitic matrix are extended in the direction of carburization. The presence of chromium and nickel in SS304 stainless steel may result in a non-uniform distribution of carbon in the austenite. Diffusion changes in the composition of multi component alloys may lead to solid transformations which involve the formation of more phases. The structure of the carburized layer shows carbide precipitation within the grain boundaries which produces a continuous superficial carbide, the proportion of carbides in the structure decreased gradually from the surface of the specimen to the interior (Ismail and Iskander, 1980).

Case hardening of stainless steels by traditional carburization techniques has seldom been employed in applications requiring corrosion resistance, due to the resultant formation of chromium carbides, which depletes chrome and decreases the resistance to corrosion. (Martin and Natishan, 2009) These carbides also compromise fatigue resistance. When austenitic steels have been exposed for a period of time in the range of approximately 425°C to 950°C, or when the steel has been heated to higher temperatures and allowed to cool through that temperature range at a relatively slow rate (such as occurs after welding or air cooling after annealing), the chromium and carbon in the steel combine to form chromium carbide particles along the grain boundaries throughout the steel (Kadry, 2008).

Formation of these carbide particles in the grain boundaries depletes the surrounding metal of chromium and reduces its corrosion resistance, allowing the steel to corrode preferentially along the grain boundaries. Steel in this condition is said to be "sensitized" (Kadry, 2008). It should be noted that carbide precipitation depends upon carbon content, temperature and time at temperature. Carbide precipitation is a form of relatively rapid and localised corrosion associated with a defective microstructure known as Intergranular corrosion. A mismatch in galvanic potential between the base metal and the grain boundary results galvanic corrosion begins. As the grain boundaries corrode, the grain and the chromium carbides drop out like particles of rusty sand.

# 4.2 Potentiodynamic Tests

During corrosion, at least two electrochemical reactions, an oxidation and a reduction reaction, occur at a metal-electrolyte interface. Because corrosion is due to an electrochemical mechanism, it is clear that electrochemical techniques can be used to study corrosion reactions and mechanisms. Figure 4.6(a), illustrates a potentiodynamic polarization curve of specimen 1 obtained during corrosion test in PBS solution with pH 7.4 with temperature, 37°C, which corresponds to the human body temperature. Figure 4.6(b) shows the tafel extrapolations for the potentiodynamic polarization curve of the samples, which can determine the corrosion rate of the sample during the test.

Corrosion normally occurs at a rate determined by equilibrium between opposing electrochemical reactions. The first is the 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 (electrical current) occurs.

From figure 4.7(b), the vertical axis is potential and the horizontal axis is the logarithm of absolute current. The theoretical current for the anodic and cathodic reactions are shown as straight lines. 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 your potentiostat. The sharp point in the curve is actually the point where the current changes signs as the reaction changes from anodic to cathodic, or vice versa and the sharp point is due to the use of a logarithmic axis. The used of a log axis is necessary because of the wide range of current values that must be displayed during a corrosion experiment. The experimental Tafel extrapolation plots were analyzed. The cathodic ( $\beta_c$ ) and anodic ( $\beta_a$ ) Tafel slopes, the estimated corrosion current density (*i*<sub>corr</sub>) and estimated corrosion potential (*e*<sub>corr</sub>), corrosion resistance (RP) and corrosion rate are tabulated in Table 4.3 for HBSS and Table 4.2 for PBS. The corrosion rates were fairly reproducible and the corrosion rates of the stainless steel were comparable.

From the Table 4.2 and Table 4.3, stainless steel shows higher corrosion rate in HBBS compared to PBS due to high concentration of chloride ion in HBSS. While it is well known that chloride solutions are among the most aggressive and corrosive to metals. However, it is unavoidable that all metals experience electrochemical dissolution in the implant environment, which consists of an ample amount of fluids, minerals such as amino acids and proteins. Corrosion not merely threatens the integrity of the implants, but also leads to concern about leaching of harmful metallic ions into the body.







**Figure 4.6:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in PBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 950°C) in contact.







**Figure 4.7:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in PBS solution at pH 7.4 and  $37\pm2^{\circ}C$  for SS304 stainless steel (carburizing for 8 hours at 900°C) in contact







**Figure 4.8:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in PBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 950°C) in contact







**Figure 4.9:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in PBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 900°C) in contact







**Figure 4.10:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in PBS solution at pH 7.4 and 37±2°C for SS304 stainless steel in contact



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**Figure 4.11:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 950°C) in contact



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**Figure 4.12:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in HBSS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 8 hours at 900°C) in contact







**Figure 4.13:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 950°C) in contact







**Figure 4.14:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel (carburizing for 6 hours at 900°C) in contact







**Figure 4.15:** (a) Potentiodynamic polarization curves and (b) Tafel extrapolation plot obtained in HBBS solution at pH 7.4 and 37±2°C for SS304 stainless steel in contact
Specimens	Process	
1	Control Specimens	
2	Carburizing for 6 hours at 900°C	
3	Carburizing for 6 hours at 950°C	
4	Carburizing for 8 hours at 900°C	
5	Carburizing for 8 hours at 950°C	

Table 4.1: Type of specimens and process

Table 4.2: Corrosion rates determined by Tafel extrapolation method in PBS

Specimens	$E_{corr}$ (V)	I <sub>corr</sub> (A)	Ba (V/div)	$\beta_c$ (V/div)	R <sub>p</sub> (ohm)	Corr Rate
						(mm/year)
1	-0.448623	8.805µ	0.121	0.190	9.647k	0.051580
2	-0.431732	8.972µ	0.442	0.231	7.340k	0.052562
3	-0.413812	7.976µ	0.080	0.079	8.714k	0.054073
4	-0.443881	9.669µ	0.105	0.144	6.725k	0.056642
5	-0.432373	7.451µ	0.255	0.169	5.921k	0.059632

Table 4.3: Corrosion rates determined by Tafel extrapolation method in HBSS

Specimens	$E_{corr}$ (V)	I <sub>corr</sub> (A)	Ba (V/div)	$\beta_c$ (V/div)	<b>R</b> <sub>p</sub> (ohm)	Corr Rate
						(mm/year)
1	-0.420803	9.451µ	0.084	0.071	3.051k	0.064720
2	-0.424477	8.306µ	0.140	0.163	2.897k	0.066234
3	-0.470120	6.305µ	0.129	0.126	4.394k	0.068201
4	-0.489726	3.020µ	0.077	0.091	6.001k	0.070600
5	-0.440707	3.521µ	0.047	0.080	3.647k	0.073118







**(b**)

Figure 4.16: Graph of SS304 stainless steel corrosion rate in: (a) PBS and (b) HBSS

#### **CHAPTER 5**

#### CONCLUSIONS

#### 5.1 Introduction

Stainless steels are widely employed in medical implants such as stents and orthopedic replacements because of their relatively low cost, ease of fabrication, and reasonable chemical stability. However, it is unavoidable that all metals experience electrochemical dissolution in the implant environment, which consists of an ample amount of fluids, minerals such as chlorides, amino acids and proteins. This interaction can lead to either the failure of the implant to function as it was intended, or have an adverse effect on the patient.

Corrosion analysis and surface analysis had been carried out in order to reveal the corrosion rate of these materials during electrochemical test in two simulated body fluid, namely; PBS and HBSS.

### 5.2 Conclusion

From the test results, the following conclusions can be obtained.

(i) SS304 stainless steel shows increased in corrosion rate after the carburizing process in simulated body fluid due to the formation of chromium carbides. Formation of these carbide particles in the grain boundaries depletes the surrounding metal of chromium and reduces its corrosion resistance, allowing the stainless steel to corrode preferentially along the grain boundaries.

(ii) Stainless steel shows higher corrosion rate in HBSS compared to PBS due to high concentration of chloride ion in HBSS. Chloride ion reacts electrochemically with the surface of SS304 stainless steel to cause corrosion and chloride ion is among the most aggressive and corrosive to metals.

#### 5.3 **Recommendations**

- (i) Longer duration for electrochemical corrosion tests to show passivation and repassivation process during the test in the two solutions.
- (ii) Using the other heat treatment process for introducing carbon into stainless steels without formation of carbides. It is possible to reclaim steel which suffers from carbide precipitation by heating it above 1000°C, followed by water quenching to retain the carbon and chromium in solution and so prevent the formation of carbides.
- (iii)Another method used to overcome intergranular corrosion is to use the extra low carbon grades such as Grades 316L and 304L; these have extremely low carbon levels (generally less than 0.03%) and are therefore considerably more resistant to the precipitation of carbide.

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## **APPENDIX A**

# PROJECT PLANNING (GANTT CHART): FINAL YEAR PROJECT 1

Work Progress	Week														
_	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Get the project title and															
arrange discussion time															
with supervisor.															
Find the problem statement															
and project objectives.															
Find scope of the project,															
hypothesis. Verify problem															
statement, project															
objectives, scope and															
hypothesis.															
Do research and collect the															
information															
Study and Learning the															
theory															
Do the design of the															
experiment and state the															
experimental procedure															
Report Writing (Chapter 1,															
2, 3)															
(Introduction, Literature															
review, Methodology)															
Submit draft thesis and															
prepare slide presentation															
Final year project 1															
presentation															

Remark:



Planning progress Actual progress

Work Progress	Week																
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15- 18	19	20
Material selection																	
and specimens																	
preparation																	
Experiment setup																	
(carburizing)																	
Grinding,																	
Polishing and																	
Mounting																	
Surface Analysis																	
Experiment setup																	
(electrochemical																	
method)																	
Report writing																	
(chapter 4 and 5)																	
Submit draft																	
thesis and prepare																	
slide presentation																	
Submission draft																	
2,3,4 and logbook																	
Final year project	<u> </u>																
2 presentation																	
Submit thesis	<u> </u>																
report																	

## PROJECT PLANNING (GANTT CHART): FINAL YEAR PROJECT 2

Remark:



Planning progress Actual progress