

EFFECT OF HEAT TREATMENT ON THE CORROSION
RESISTANCE OF ALUMINIUM ALLOYS

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BORANG PENGESAHAN STATUS TESIS♦

JUDUL: **EFFECT OF HEAT TREATMENT ON THE CORROSION
RESISTANCE OF ALUMINIUM ALLOYS**

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EFFECT OF HEAT TREATMENT ON THE CORROSION RESISTANCE OF
ALUMINIUM ALLOYS

AHMAD ZUL HAFIZ BIN RAMLI

A report submitted in partial fulfilment of the requirements
For the award of the degree of
Bachelor of Mechanical Engineering Pure

Faculty of Mechanical Engineering
UNIVERSITI MALAYSIA PAHANG

DECEMBER 2010

UNIVERSITI MALAYSIA PAHANG
FACULTY OF MECHANICAL ENGINEERING

We certify that the project entitled “*Effect of Heat Treatment on the Corrosion Resistance of Aluminium Alloys*” is written by *Ahmad Zul Hafiz Bin Ramli*. 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 fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering.

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I hereby declare that the work in this thesis 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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Specially dedicated to
My beloved family, and those who have guided and inspired me
Throughout my journey of learning

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ABSTRACT

Aluminium alloy 5083 is commonly used in the ships and boats in the marine environment and susceptible to corrosion when they are run at high speed velocity and high flow rate condition. The purpose of this study is to investigate the effect of solution heat treatment on the corrosion resistance of aluminium alloys by using aluminium 5083 as a material for the experiments. The objective of this study is to investigate the corrosion resistance by differing the aging time during solution heat treatment process and to determine the corrosion rate by using immersion test method. The microstructures of heat treated specimens have been observed using metallurgical microscope and scanning electron microscope while the hardness test has been done using Vickers hardness tester. The corrosion rates of the heat treated specimens were determined using weight loss method. The corrosion resistance of heat treated specimens increased after solution heat treatment while the hardness of the specimens slightly decreased. The type of corrosion occurred on the surface of specimens were found to be uniform corrosion.

ABSTRAK

Aloi aluminium 5083 umumnya digunakan di kapal-kapal dan perahu di dalam persekitaran lautan dan terdedah kepada pengaratan apabila digunakan dalam kelajuan tinggi dan dalam keadaan aliran yang tinggi. Tujuan kajian ini adalah untuk mengetahui pengaruh rawatan haba larutan pada ketahanan pengaratan aloi aluminium dengan menggunakan aloi aluminium 5083 sebagai bahan untuk eksperimen. Tujuan kajian ini adalah untuk mengetahui ketahanan pengaratan dengan waktu penuaan yang berbeza semasa proses rawatan haba larutan dan untuk menentukan kadar pengaratan dengan menggunakan kaedah rendaman untuk pengaratan. Struktur mikro spesimen selepas rawatan haba larutan telah diperhatikan menggunakan mikroskop metalurgi dan mikroskop elektron pengimbas sementara ujian kekerasan telah dilakukan menggunakan alat pengujian kekerasan Vickers. Kadar pengaratan sampel rawatan haba ditentukan dengan menggunakan kaedah berat hilang selepas rendaman. Kadar ketahanan pengaratan spesimen yang mengalami rawatan haba meningkat selepas rawatan haba larutan tetapi nilai kekerasan spesimen sedikit menurun. Jenis pengaratan yang berlaku pada permukaan spesimen dikenali sebagai pengaratan seragam.

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LIST OF SYMBOLS

A	Area
Al	Aluminium
Al ³⁺	Aluminium dissolves to 3 electron
Al ₂ O ₃	Aluminium oxide
Al(OH) ₃	Hydroxide
AlOOH	Oxyhydroxide
C	Carbon
-COOH	Carboxyl
d	Diameter
e	electron
H	Hydrogen
H ₂	Hydrogen gas
HCL	hydrochloric acid
H ⁺	Hydrogen ion
H ₂ O	Water
H ₂ SO ₄	Sulphuric acid
K	Corrosion constant
Mg	Magnesium
-NH	Amine
-OH	Hydroxyl
O ₂	Oxygen
-PO ₃	Phosphate
S	Sulphur

Si	silicon
T	Time of exposure
W	Weight loss
ZnCl ₂	zinc chloride
Zn ²⁺	Zincs ion
Zn	Zinc
&	And
°C	Celcius

LIST OF ABBREVIATIONS

Al	Aluminium
Al Mn	Aluminium manganese
Al Mg	Aluminium magnesium
Al Si	Aluminium silicon
ASTM	American society for testing and material
ANSI	American national standard institute
ed	Edition
FYP	Final year project
ipm	Inches per month
ipy	Inches per year
ml	Milliliter
mm	Millimeter
mpy	Mile per year
SEM	Scanning electron microscope
UMP	Universiti Malaysia Pahang

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

With a huge consumption every year, aluminium alloy is one of the world's most commonly used metals. Its lightness coupled with its strength, conductivity, barrier properties and its excellent corrosion resistance is the most important advantages and the main reasons for the continued growth in the usage of aluminium alloy.

Aluminium alloy's lightweight performance delivers great benefit in transport applications such as aerospace, cars, ships, trains and buses. The metal's excellent characteristics help give automotive and other transport users improved driving performance as well as increasing fuel economy and reducing emissions. Another significant advantage of aluminium alloy is its corrosion resistance. This characteristic is valuable for products used in architecture, construction, civil engineering, transport, heat exchangers and many other applications.

From days to days, aluminium alloy is used in many areas of transportation, ship building and other harsh operating conditions. The alloys developed by these metallurgists as well as having excellent corrosion resistance are also weld able, which led to them being firmly established as a key construction material by ship and high speed ferry builders.

Improving corrosion performance has been a key focus of this metallurgical development. In the 1970's, alloys were developed with very good thermal conductivity, combined with excellent resistance to engine coolants. This combination

means that it was possible to manufacture heat exchangers that were cheaper and of course lighter than the traditional copper versions. More recently new alloy developments have included automotive, aerospace, desalination of sea water, renewable energy and maritime applications, as well as many civil engineering and industrial applications (Vargel, 2004).

Aluminium alloy 5000 series is ideal for the construction of small leisure boats, luxury vessels, workboats, fishing vessels, and the world's largest high speed passenger such as ferries. In the case of floating marine structures, it can improve stability, increase dead weight, reduce draft, minimize maintenance and increase speed. In the case of stationary structures it offers reduced maintenance cost and decreased erection costs. But, aluminium alloys also suffer a little corrosion in the marine environment such as in the sea water. Therefore, this study is going to investigate the effect of heat treatment on corrosion resistance of aluminium alloy. The type of aluminium choose in this study is 5083 series aluminium alloys which is the most commonly used in marine application such as boats and ships.

1.2 PROBLEM STATEMENT

The used of fiber reinforced polymer in the ships and small fishing due to high corrosion resistance have many environmental and recycling problems. Therefore, aluminium alloys are used as substitutes for fiber reinforced polymer (FRP) in ships and boats. However, ships constructed with 5000 series aluminium alloys suffer a little corrosion in the marine environment especially when they run in high speed and high flow rate. Thus, solution heat treatment was carried out to investigate the effect of heat treatment whether increase or decrease the corrosion resistance of aluminium alloys with different time of aging process. The difference time of aging process will affect the corrosion rate and mechanical properties of the material such as hardness and ductility (Kim & Jang, 2009).

1.3 OBJECTIVES OF STUDY

The objectives of this study are:

- (i) To investigate the effects of heat treatment on corrosion resistance of aluminium alloys with different time of aging process.
- (ii) To determine the corrosion rate of the aluminium alloys by using immersion test method.
- (iii) To analyze the corrosion type occurs to the aluminium alloys.
- (iv) To determine the hardness of the aluminium alloys after heat treatment process.

1.4 SCOPES OF PROJECT

The scopes of this study include:

- (i) Perform the solution heat treatment experiment on aluminium alloy 5083 using furnace in material laboratory in UMP pekan.
- (ii) Heat treated the aluminium alloy specimen at 420 °C, cooling in the water and aging at 180°C for 30, 60, 90, 120, 180 and 240 minutes.
- (iii) Evaluate the corrosion rate by using immersion corrosion test for 23 days based on weight lost method.
- (iv) Determine the corrosion type occur in the specimen after immersion test by using Scanning Electron Microscope (SEM).
- (v) Using Vickers hardness test to analyze the hardness of each specimen.

1.5 THESIS ORGANIZATION

This thesis will be divided into five chapters. First chapter is the introduction to the thesis and briefing about the project's ideas such as the information about the characteristics of aluminium alloys and its advantages. It also included the corrosion resistance of this material and application in various industries. There will also include the problem statement, objectives and the scopes of the project.

Second chapter is for the literature review. This chapter will discuss about the corrosion process including electrochemical reaction occur during corrosion process and types of corrosion. This chapter also includes the briefing about the aluminium alloys and solution heat treatment process. The details information about the project is also stated in this chapter.

The third chapter is methodology. This chapter is all about the method and procedures used in the experiment process. This chapter can be divided into three experiments which is the solution heat treatment process, immersion of corrosion test and hardness test by using Vickers hardness test. The surface analysis is investigated by using Scanning Electron Microscope and metallurgical microscope. The detail of the procedure was discussed in this chapter.

Next chapter is chapter four which is for the data collection and analysis the result of the experiments. The collected data will be analyzed and the results will be shown graphically or in tables. This will help readers to understand the result thoroughly and completely. This chapter also included the discussion of the result that obtains from the experiments and factors that affecting the result.

The last chapter is for the conclusion. All the recommendation and suggestion will be discussed in this for the further research and study. This chapter is very crucial for improving the future research.

1.6 PROJECT METHODOLOGY

Methodology is one of the most important things to be considered to make sure that the project will run smoothly without any problems and achieve the objective. Project methodology will describe the flow of the project progress and shows us how the project started, how data was collected, and how the next steps are done. Methodology process is related to the flow chart and Gantt chart. In order to complete this project, the scheduling by using Gantt chart is very important and to make sure all the planning is on time.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Corrosion can be defined as the destruction or deterioration of a material due to reaction with its environment. Material can be metals, polymers, ceramics, plastics, rubber and others nonmetallic materials. Corrosion can be fast or slow. For example, deterioration of paint and rubber by sunlight or chemicals, fluxing of the lining of a steelmaking furnace, and attack of a solid metal by another molten metal (liquid metal corrosion) are all considered to be corrosion (Fontana, 1910).

2.2 ELECTROCHEMICAL REACTION

The electrochemical nature of corrosion can be illustrated by attack on zinc by hydrochloric acid. When the zinc is placed in dilute hydrochloric acid, a reaction occurs and hydrogen gas is evolved. The zinc dissolves and forms a solution of zinc chloride (Fontana, 1910).



This equation can be written in the simplified form because the chloride ion is not involved in the reaction.



Therefore, zincs react with hydrogen ion of the acid solution to form zinc ion and hydrogen gas. From equation, we can see that zinc is oxidized to zinc ions and hydrogen ions are reduced to hydrogen (Fontana, 1910).

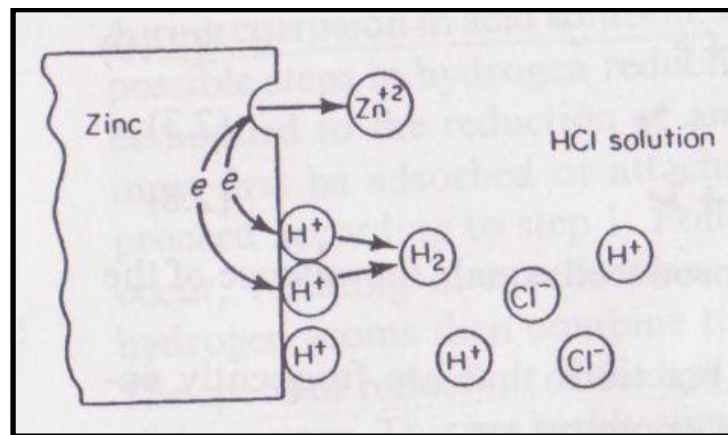
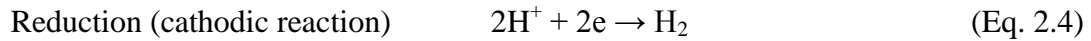
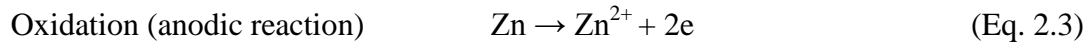


Figure 2.1: Electrochemical reaction between zinc and hydrochloric acid

Source: Fontana, 1910

2.3 FORMS OF CORROSION

The different forms of corrosion can be divided into the following eight categories based on the appearance of the corrosion damage or the mechanism of attack.

2.3.1 Uniform Attack

Uniform attack is the most common form of corrosion. It normally characterized by a chemical or electrical reaction that proceed uniformly over the entire exposed surface for over a large area. The metal become thinner and eventually become fail (Fontana, 1910).

The general attack results from local corrosion-cell action; that is, multiple anodes and cathodes are operating on the metal surface at any given time. The location of the anode and cathode areas continues to move about on the surface, resulting in uniform corrosion. Uniform corrosion often results from atmospheric exposure especially polluted industrial environments, exposure in fresh, brackish, and salt waters or exposure in soils and chemicals (Davis, 2001).

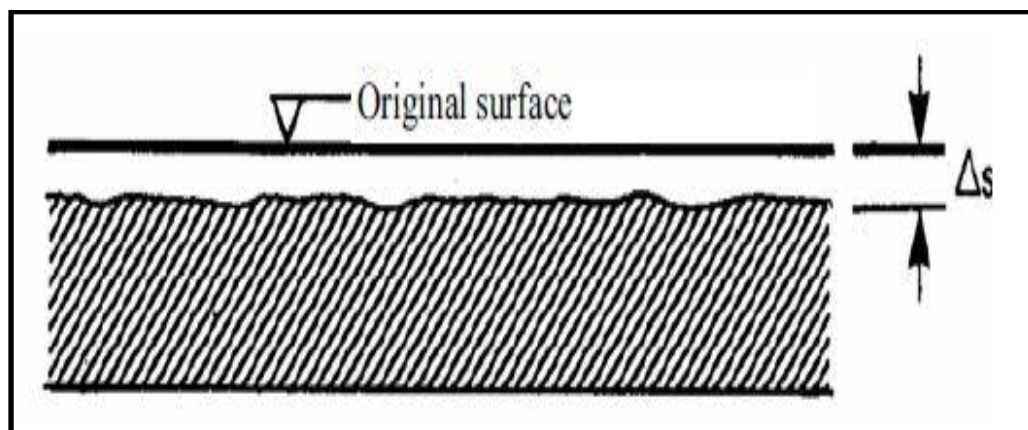


Figure 2.2: Example of uniform corrosion

Source: Bardal, 1933

2.3.2 Galvanic Corrosion

Galvanic corrosion is an electrochemical action of two dissimilar metals in the presence of an electrolyte and an electron conductive path. It occurs when dissimilar metals are in contact and the potential difference produces electron flow between them.

Corrosion of the less corrosion resistant metal is usually increased and attack of the more resistant material is decreased, as compared with behavior of these metals when they are in contact. The less resistant metal becomes anodic and the more resistant metal becomes cathodic. Usually the cathode or cathodic metal corrodes very little or not at all in this type of couple (Fontana, 1910).

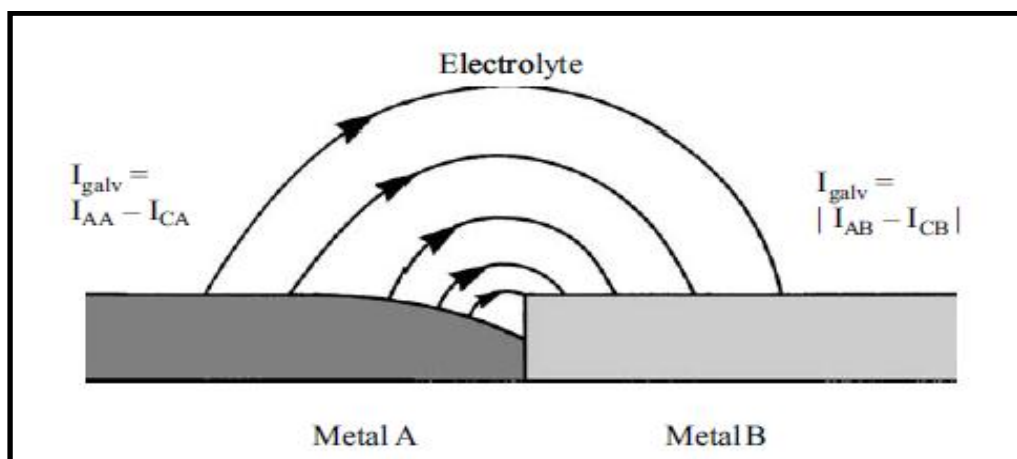


Figure 2.3: Example of galvanic corrosion

Source: Bardal, 1933

2.3.3 Crevice Corrosion

Crevice corrosion is a form of localized attack that occurs at narrow openings or spaces (gaps) between metal-to-metal or nonmetal-to-metal components. This type of attack results from a concentration cell formed between the electrolyte within the crevice, which is oxygen starved, and the electrolyte outside the crevice, where oxygen is more plentiful.

The material within the crevice acts as the anode, and the exterior material becomes the cathode. Crevices may be produced by design or accident. Crevices caused by design occur at gaskets, flanges, rubber O-rings, washers, bolt holes, rolled tube ends, threaded joints, riveted seams, overlapping screen wires lap joints, beneath coatings (filiform corrosion) or insulation (poultice corrosion), and anywhere close-fitting surfaces are present (Davis, 2001).

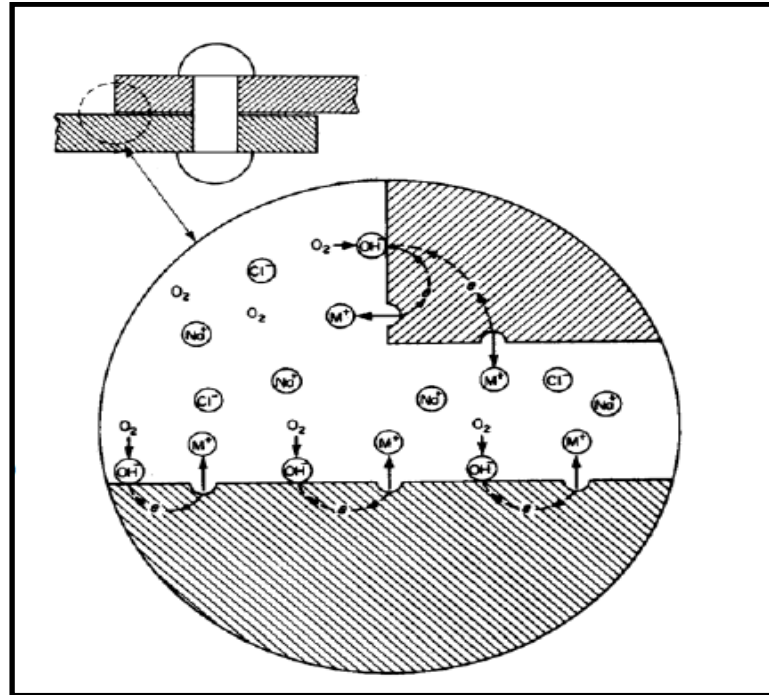


Figure 2.4: Crevice corrosion at initial stage

Source: Bardal, 1933

2.3.4 Pitting Corrosion

Pitting is a form of extremely localized attack that results in holes in the metal. These holes may be small or large in diameter, but in most cases they are relatively small. Pits are so close together that they look like rough surface. Generally a pit may be described as a cavity or hole with the surface diameter about the same or less than the depth. Pitting is one of the most destructive forms of corrosion. It is often difficult to detect pits because of their small size and because the pits are often covered with corrosion product (Fontana, 1910).

This form of corrosion can be found on aluminium and its alloys and automobile chromium-plated bumpers or body coated (painted) parts due to film or coating breakdown at isolated surface sites. Pits vary in shape, but are very small surface holes due to the extremely localized anodic reaction sites (Perez, 2004).

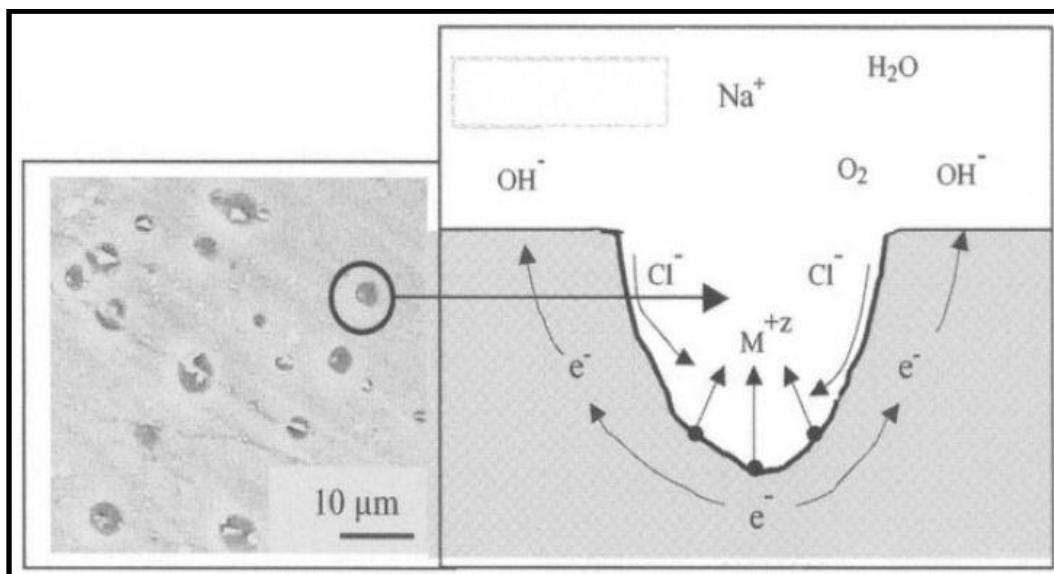


Figure 2.5: Localized corrosion on 2195 Al-Li alloy

Source: Perez, 2004

2.3.5 Intergranular Corrosion

Intergranular corrosion is defined as the selective dissolution of grain boundaries, or closely adjacent regions, without appreciable attack of the grains themselves. This dissolution is caused by potential differences between the grain-boundary region and any precipitates, intermetallic phases, or impurities that form at the grain boundaries (Davis, 2001).

The general cause of intergranular corrosion is the presence of galvanic elements due to differences in concentration of impurities or alloying elements between the material in or at the grain boundaries and interior of the grains (Bardal, 1933).

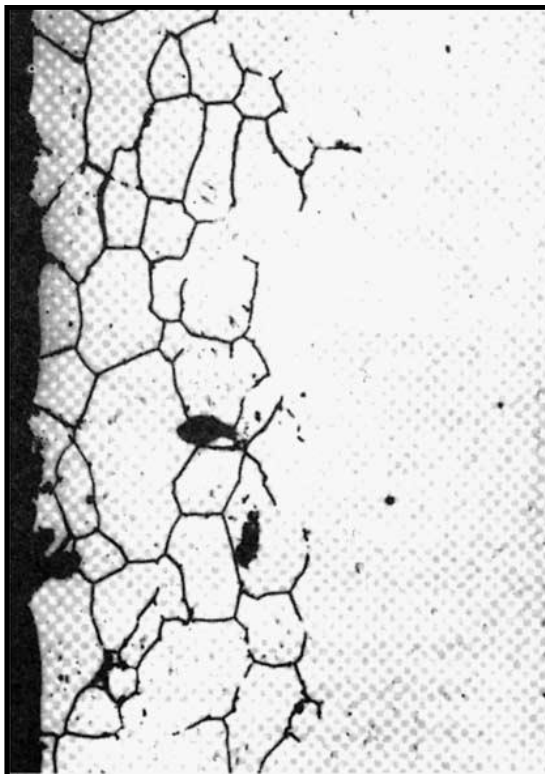


Figure 2.6: Intergranular corrosion

Source: Vargel, 2004

2.3.6 Erosion Corrosion

Erosion corrosion is the acceleration or increase in the rate of deterioration or attack on a metal because of relative movement between corrosive fluid and the metal surface. Generally this movement is quite rapid, and mechanical wear effects or abrasion are involved. Metal is removed from the surface as dissolved ions, or it forms solid corrosion products that are mechanically swept from the metal surface (Fontana, 1910).

The results of erosion corrosion are grooves or pits with a pattern determined by the flow direction and the local flow condition. The corrosion form is typical at relatively high velocities between the material surface and the fluid. Components often having this corrosion are propellers, pumps, turbine parts, valves, heat exchanger tubes, bends, and equipment exposed to liquid sputter or jets (Bardal, 1933).

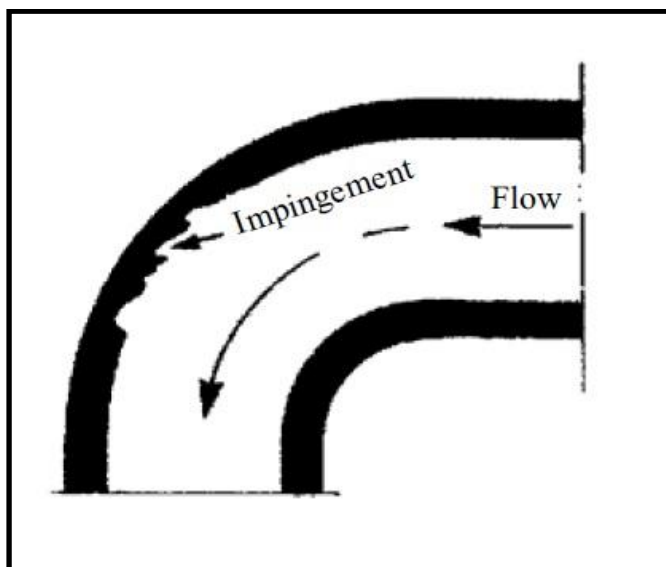


Figure 2.7: Erosion corrosion at pipe elbow

Source: Bardal, 1933

2.3.7 Fretting Corrosion

Fretting wear occurs at the interface between two closely fitting components when they are subject to repeated slight relative motion (slip). The relative motion may vary from less than a nanometer to several micrometers in amplitude. Vulnerable objects are shrink fits, press fits, bolted joints, and other assemblies where the interface is under load (Bardal, 1933).

Oxidation is the most common element in the fretting process. In oxidizing systems, fine metal particles removed by adhesive wear are oxidized and trapped between the fretting surfaces (Figure 2.7). The oxides act like an abrasive (such as lapping rouge) and increase the rate of material removal.

This type of fretting in ferrous alloys is easily recognized by the red material oozing from between the contacting surfaces. These occur in regions where slight relative movements have occurred between mating, highly loaded surfaces (Davis, 2001).

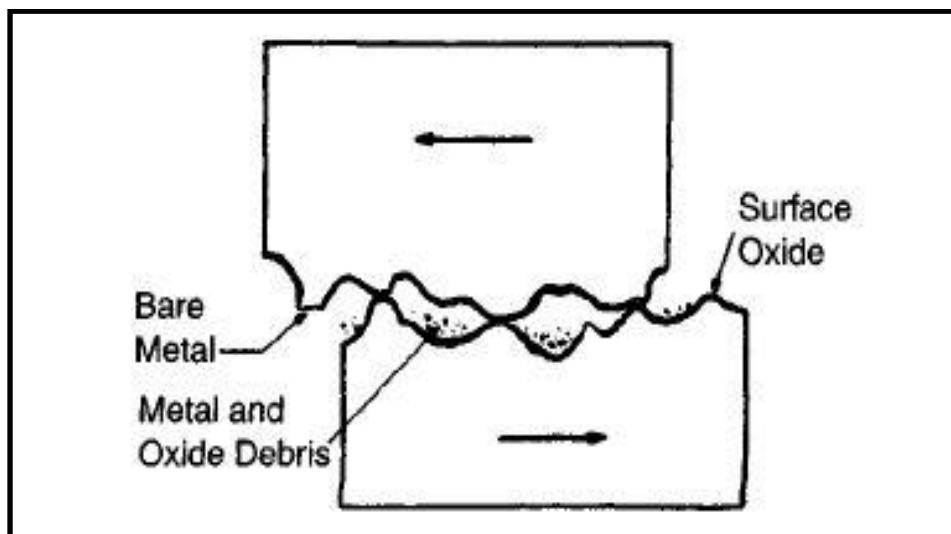


Figure 2.8: Fretting corrosion.

Source: Davis, 2001

2.3.8 Stress Corrosion

Stress corrosion cracking can be defined as crack formation due to simultaneous effects of static tensile stresses and corrosion. The tensile stresses may originate from external load, centrifugal forces or temperature changes, or they may be internal stresses induced by cold working, welding or heat treatment.

The cracks are mainly formed in planes normal to the tensile stresses, and propagate intergranularly or transgranularly, more or less branched. If they are not detected in time, they will cause fast, unstable fracture (Bardal, 1933).

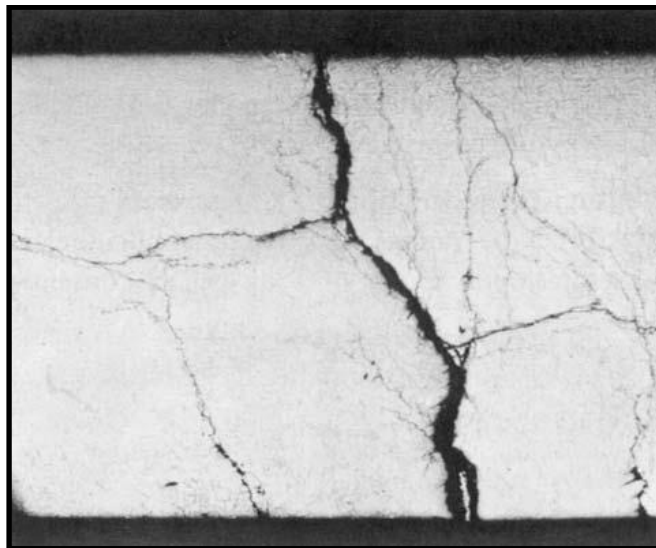


Figure 2.9: Stress corrosion cracks in tube of AISI 304 steel.

Source: Bardal, 1933

2.4 CORROSIVE CONDITION

If oxygen and water are both present, corrosion will normally occur on iron and steel. Rapid corrosion may take place in water and the rate of corrosion being accelerated by several factors such as:

- (i) The velocity or the acidity of the water,
- (ii) The motion of the metal,
- (iii) An increase in temperature.
- (iv) The presence of certain bacteria.

2.4.1 Water

Water can readily dissolve a small amount of oxygen from the atmosphere and become highly corrosive. When the free oxygen dissolved in water is removed, the water becomes practically noncorrosive unless it becomes acidic or anaerobic bacteria incite corrosion. If oxygen-free water is maintained at a neutral pH or at slight alkalinity, it is practically noncorrosive to structural steel (Davis, 2001).

2.4.2 Soils

Dispersed metallic particles or bacteria pockets can provide a natural electrical pathway for buried metal. If an electrolyte is present and the soil has a negative charge in relation to the metal, an electrical path from the metal to the soil will occur, resulting in corrosion.

Differences in soil conditions, such as moisture content and resistivity, are commonly responsible for creating anodic and cathodic areas. Where a difference exists in the concentration of oxygen in the water or in moist soils in contact with metal at different areas, cathodes develop at points of relatively high oxygen concentrations and anodes at points of low concentration (Davis, 2001).

2.4.3 Chemical

In an acid condition, the metal at the anode is attacked at rapid rate even without the presence of oxygen. At the cathode, atomic hydrogen is released continuously to become hydrogen gas. Corrosion by acid can produce the formation of salt which slows the reaction due to the formation on the surface is attacked. Corrosion by direct chemical attack is the single most destructive force against steel surfaces. Substances having chlorine or other halogens in their composition are particularly aggressive (Davis, 2001).

2.4.4 Atmospheric Corrosion

Corrosion actions that occur in the atmosphere are different than in water or underground because sufficient oxygen is always present. In atmospheric corrosion, the presence of moisture and deposits from the atmosphere control the rate of corrosion. Contaminants such as sulfur compounds and salt particles can accelerate the corrosion rate.

Atmospheric corrosion occurs primarily through electrochemical means and is not directly caused by chemical attack. The anodic and cathodic areas are usually quite

small and close together so that corrosion appears uniform, rather than in the form of severe pitting which can occur in water or soil (Davis, 2001).

2.5 CORROSION PREVENTION

Corrosion prevention aims at removing or reducing the effect of one or more of the conditions leading to corrosion using the following measures (Bardal, 1933):

- (i) Selecting a material that does not corrode in the actual environment.
- (ii) Changing the environment such removing the oxygen or adding anticorrosion chemicals (inhibitors).
- (iii) Using a design that will avoid corrosion, such preventing the collection of water so that the metal surface can be kept dry.
- (iv) Changing the potential, most often by making the metal more negative and thus counteracting the natural tendency of the positive metal ions to be transferred from the metal to the environment.
- (v) Applying coatings on the metal surface, usually in order to make a barrier between the metal and the corrosive environment.

2.6 ALUMINIUM ALLOYS

Aluminium alloys are material group whose application has increased strongly due to a number of beneficial properties such as low weight, excellent formability, and high corrosion resistance in many environments, colorless and nonpoisonous corrosion products, and high electrical and thermal conductivity (Bardal, 1933).

2.6.1 Material Properties

Aluminium alloys are a commercially important metal and abundant. It has relatively low strength and the density of alloys is about 2.7 g/ml. The recyclability and available new smelting processes, low cost and high strength to weight ratio are attractive properties. The metal and its alloy are nonmagnetic and have high electrical and thermal conductivity and high reflectivity (Sastri, Ghali, & Elboujdaini, 2007).

2.6.2 Aluminium Alloys Series

Some features of the aluminium alloys are given below (Sastri, Ghali, & Elboujdaini, 2007):

(i) 1000 series

99% contain of Al, excellent resistance to corrosion; high electrical, thermal conductivity and poor mechanical properties.

(ii) 2000 series

Contains copper; high strength and heat-treatable; low corrosion resistance; subject to intergranular attack and difficult to inhibit.

(iii) 3000 series

Contains manganese; cannot be heat-treated; most widely used alloys; 3003 has moderate strength; good workability can be inhibited in some media.

(iv) 4000 series

Contains silicon; used mainly for welding because of lower melting points; used in architectures because of color effects due to applied anodic coatings; have good corrosion resistance and can be inhibited.

(v) 5000 series

Contains magnesium; corrosion-resistant, can be inhibited; widely used in marine atmospheres with good performance.

(vi) 6000 series

Contains Si and Mg in a ratio required to form magnesium silicide; have good corrosion resistance and the alloys can be inhibited effectively

(vii) 7000 series

Contains zinc, small amounts of Mg, Cu and Cr; heat-treatable, have high strength (e.g., 7075); inhibitors can be used.

2.6.3 Composition of Aluminium Alloys

The nominal chemical composition of some wrought alloys in tempers typical of their most common usage is given in Tables 1.

Table 2.1: Composition of alloys

Alloys	Percentage of alloying elements					
	Si	Cu	Mn	Mg	Cr	Zn
Non-heat-treatable alloys						
1060	99.60%	min Al				
1100	99.60%	min Al				
1350	99.50%	min Al				
3003		0.12	1.20			
3004			1.20	1.0		
5052				2.5	0.25	
5054			0.80	2.7	0.12	
5456			0.80	5.1	0.12	
5083			0.70	4.4	0.15	
5086			0.45	4.0	0.15	
7072						1.0

Source: Sastri, Ghali, & Elboujdaini, 2007.

2.6.4 Corrosion Behavior of Aluminium Alloys

Aluminium alloys are an active metal and its resistance to corrosion depends on the formation of the protective oxide film. Protective film is stable in aqueous solutions of pH range 4-9. The protective oxide film formed in water and atmospheres at ambient temperatures is amorphous and a few nanometers in thickness. The stability of the oxide film and its disruption results in corrosion.

The dissolved oxygen in acid solution causes corrosion of aluminium while the hydrogen, nitrogen, carbon dioxide and hydrogen sulfide have no effect. Hydrogen chloride is strongly corrosive while sulfur dioxide in solutions has an etching effect. Most of the alloys are inert to concentrated nitric and acetic acids, but are attacked by dilute nitric, sulphuric and hydrochloric acids. The sensitivity to acid attack also depends upon the alloy (Sastri, Ghali, & Elboujdaini, 2007).

2.6.5 The Electrochemical Reaction of Aluminium

The fundamental reactions of the corrosion of aluminium in aqueous medium have been the subject of many studies. In simplified terms, the oxidation of aluminium in water proceeds according to the equation:



Metallic aluminium, in oxidation state 0, goes in solution as trivalent cation Al^{3+} when losing three electrons.

This reaction is balanced by a simultaneous reduction in ions present in the solution, which capture the released electrons. In common aqueous media with a pH close to neutral such as fresh water, seawater, and moisture it can be shown by thermodynamic considerations that only two reduction reactions can occur:

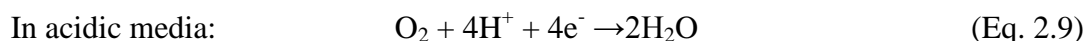
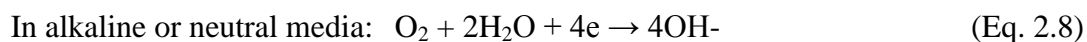
Reduction of H^+ proton:



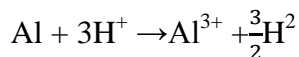
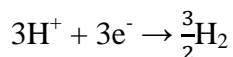
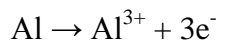
H^+ protons result from the dissociation of water molecules:



Reduction of oxygen dissolved in water:



Globally, the corrosion of aluminium in aqueous media is the sum of two electrochemical reactions, oxidation and reduction:



Or



Aluminium corrosion results in the formation of alumina $\text{Al}(\text{OH})_3$, which is insoluble in water and precipitates as a white gel, which is found in corrosion pits as white gelatinous flakes (Vargel, 2004).

2.6.6 Reaction of Aluminium with Sulphuric Acid

The corrosion rate of aluminium increases with the acid concentration and very sharply increases with temperature. For a given concentration, it is much smaller than what is observed with hydrochloric acid. Table 2 shows the corrosion in sulphuric acid per year with difference percent of concentration. Therefore, there are a few applications of aluminium in contact with dilute sulphuric acid such as cathodes of electrolysis cells for the production of zinc sulphate.

Battery fluid is very aggressive towards aluminium. That is why components in aluminium alloys must be protected against the spillage of battery fluid (Vargel, 2004).

Table 2.2: Corrosion rate of 1050 in sulphuric acid influence of temperature

Concentration (%)	Corrosion rate (mm/year)		
	20 °C	50 °C	98 °C
1.0	0.15	1.60	3.60
10.0	0.22	3.20	17.50
25.0	0.27	6.00	Dissolved
62.5	3.34	Dissolved	Dissolved
78.0	11.60	Dissolved	Dissolved
96.0	3.60	1.10	2.94

Source: Vargel, 2004

2.6.7 Application Of Aluminium Alloys

Aluminium and its alloy represent an important category of materials. This material is widely used in many applications such as in aerospace, automotive, marine technology and food industries others due to its excellent characteristic such as light weight, high corrosion resistance and high conductivity.



Figure 2.10: Marine industries

Source: <http://www.hot.ee/kloostri/ship.htm>

2.7 HEAT TREATMENT OF ALUMINIUM ALLOYS

Heat treatment of aluminium alloys is done for the purpose of making them more like pure aluminium, which is light, lustrous, and corrosion-resistant. The heat treatment of aluminium alloys strengthens their hardness, tensile strength, and electrical conductivity, thus making them even more suitable for the manufacture of aircraft components ("Heat Treatment of Aluminium," 2008).

2.7.1 Solution Heat Treatment of Aluminium Alloy

Solution Treatment, or solutionizing, is the first step in the precipitation-hardening process where the alloy is heated above the solvus temperature and soaked

there until a uniform solid solution is produced. The precipitates are dissolved in this step and any segregation present in the original alloy is reduced.

This heat treatment of aluminium method is usually done to increase the strength of an alloy. It involves heating an alloy at a high temperature for a specific period of time, and then rapidly cooling or quenching the material by immersing it in water or a water-glycol solution.

2.7.2 Quenching

Quenching is the second step where the sample is rapidly cooled to lower temperature, usually room temperature, and the cooling medium is usually water at room temperature. The structure of alloy sample after quenching consists of supersaturated solid solution of α phase (Smith & Hashemi, 1990).

2.7.3 Artificial Aging

Aging is the third step where the solution heat-treated and quenched alloy sample is necessary so that a finely dispersed precipitate forms. The formation of a finely dispersed precipitate in the alloy is the objective of the precipitation-hardening process. The fine precipitates in the alloy impede dislocation movement by forcing the dislocations to either cut through the precipitated particles or go around them. By restricting dislocation movement during deformation, the alloy is strengthened (Smith & Hashemi, 1990).

2.7.4 Natural Aging

Aging at room temperature is called natural aging, whereas aging at elevated temperature is called artificial aging. Most alloys require artificial aging, and the aging temperature is usually between about 15 and 25 percent of the temperature difference between temperature and the solution heat treatment temperature (Smith & Hashemi, 1990).

2.7.5 Solution Heat Treatment Diagram

The process of solution heat treatment process can be illustrated as Figure 2.10. The process start with solutionising process, followed by quenching and aging process at much lower temperature than solutionising process.

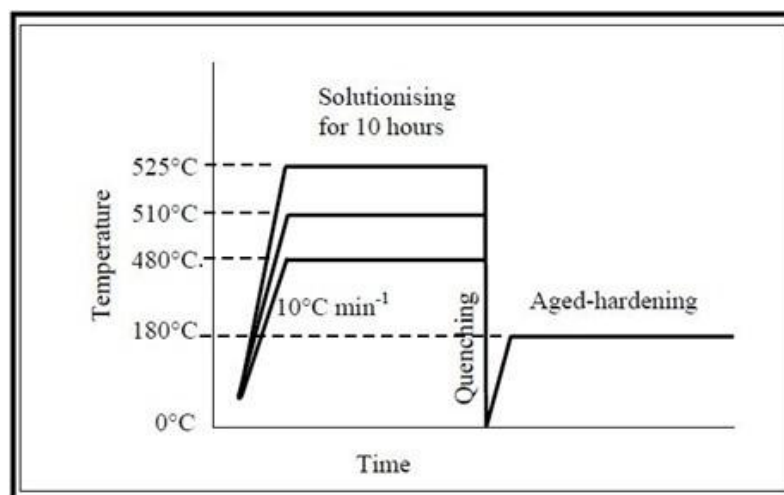


Figure 2.11: The schematic diagram of solution heat treatment process

Source: Muzium & Shamsul, 2007

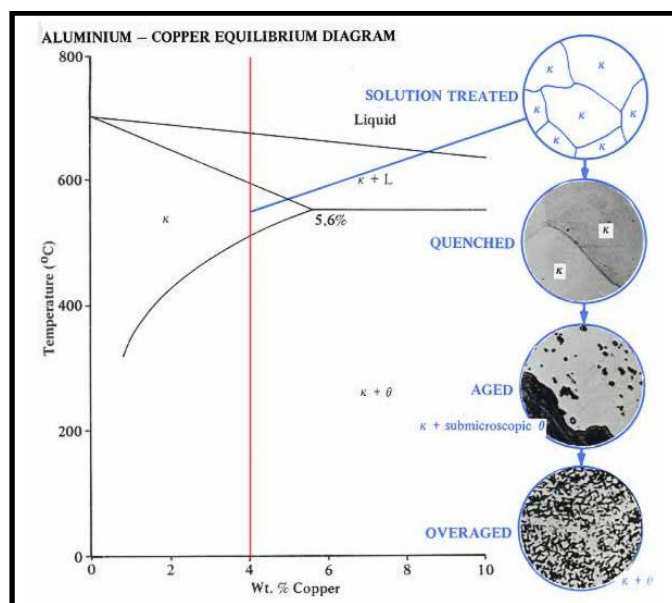


Figure 2.12: Age hardening 2xxx series aluminium alloy

Source: Gibson, 2000

2.7.6 Laboratory Furnace

For continuous heating to process samples and materials laboratory furnaces are essential. High temperature refractory materials are used for making laboratory furnace so that so that without breaking down high temperature can be maintained.



Figure 2.13: Laboratory furnace in UMP

2.7.6.1 Configuration

Laboratory furnace can be configured in ashing, box or muffle, bottom loading, top loading, and tube. Ashing is designed for removing matrix constituents that might interfere with the measurement of the analyte. Box furnaces have a box shape and a box-shaped interior. Product is loaded in the bottom loading furnace which is treated through the bottom of the chamber via a platform elevator. In top loading laboratory furnaces the products are placed from the top. Laboratory furnace can also be configured into tube furnaces that are designed to heat a tube that is usually 50 to 100 cm in length and from 25 to 100 mm in diameter.

2.7.6.2 Operating Temperature

They can be operated at high temperatures and are very well insulated which tap the heat. The heat distribution is uniform. There is a proper mechanism that keeps the temperature stable. Heat is generally generated through various sources like combustion, electrical or resistance, indirect or contact or conduction, induction, infrared or radiant, natural gas etc.

2.8 SCANNING ELECTRON MICROSCOPE (SEM)

Scanning electron microscope is an important tool in material sciences and engineering. It is used for microscopic features measurement, fracture characterization, microstructure studies, thin coating evaluations, surface contamination examination and failure analysis of material.

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. Basically, an electron gun produces an electron beam in an evacuated column that is focused and directed so that it impinges on a small spot on the target. The resolution of many SEM instruments is about 5nm, with a wide range of magnification about 15 to 100,000X (Smith & Hashemi, 2006).



Figure 2.14: Scanning electron microscope in UMP

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, the methodology of this study was carefully discussed in details. The specimen type used for this study is aluminium alloys 5083. Methodology included in this study is divided into four experiments which are solution heat treatment, microstructures observation, hardness test and immersion test for corrosion.

For the solution heat treatment process, the specimen is artificially aged at 180⁰C with difference time after being heat treated at 420⁰C for 60 minutes and then cooled in water. The microstructures produce after heat treated were analyzed using metallurgical microscope and the details analysis was carried out using Scanning Electron Microscope (SEM).

For the hardness test, Vickers harness test is used to determine the hardness for each specimen. Next experiment is immersion test including the preparing and cleaning specimen for corrosion test, weighing the specimen before and after test, corrosion rate analysis and finally surface morphology to determine the type of corrosion occurred in the surfaces of the specimens.

The most methods and procedures used in this study was referred from the American National Standard (ASTM) depends on the suitability and applications.

3.1.1 General Experiment Procedures

Figure 3.1 shows the general experiment procedures for solution heat treatment of aluminium alloys and immersion test method. The specimens were cut before heating in the furnace and then prepared for immersion test.

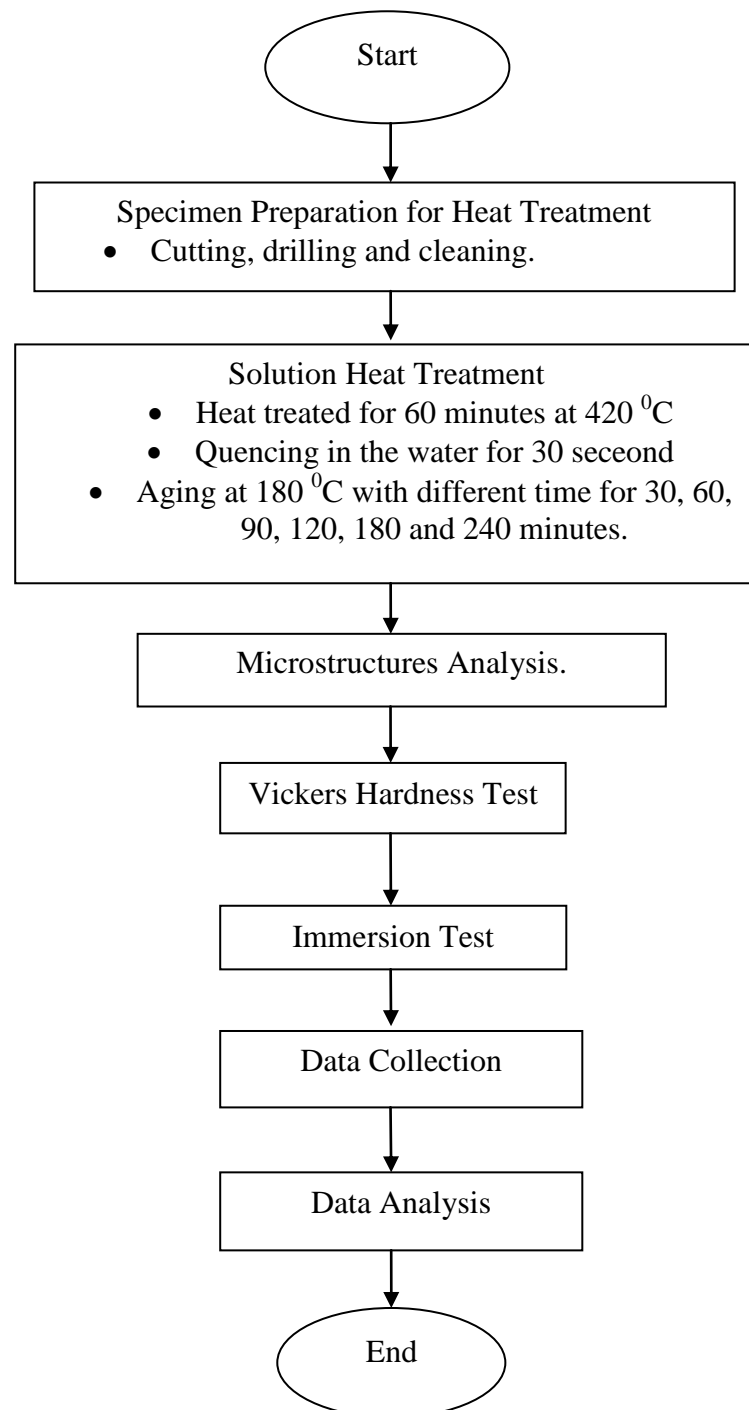


Figure 3.1 General experimental procedures.

3.1.2 Immersion Test Procedure

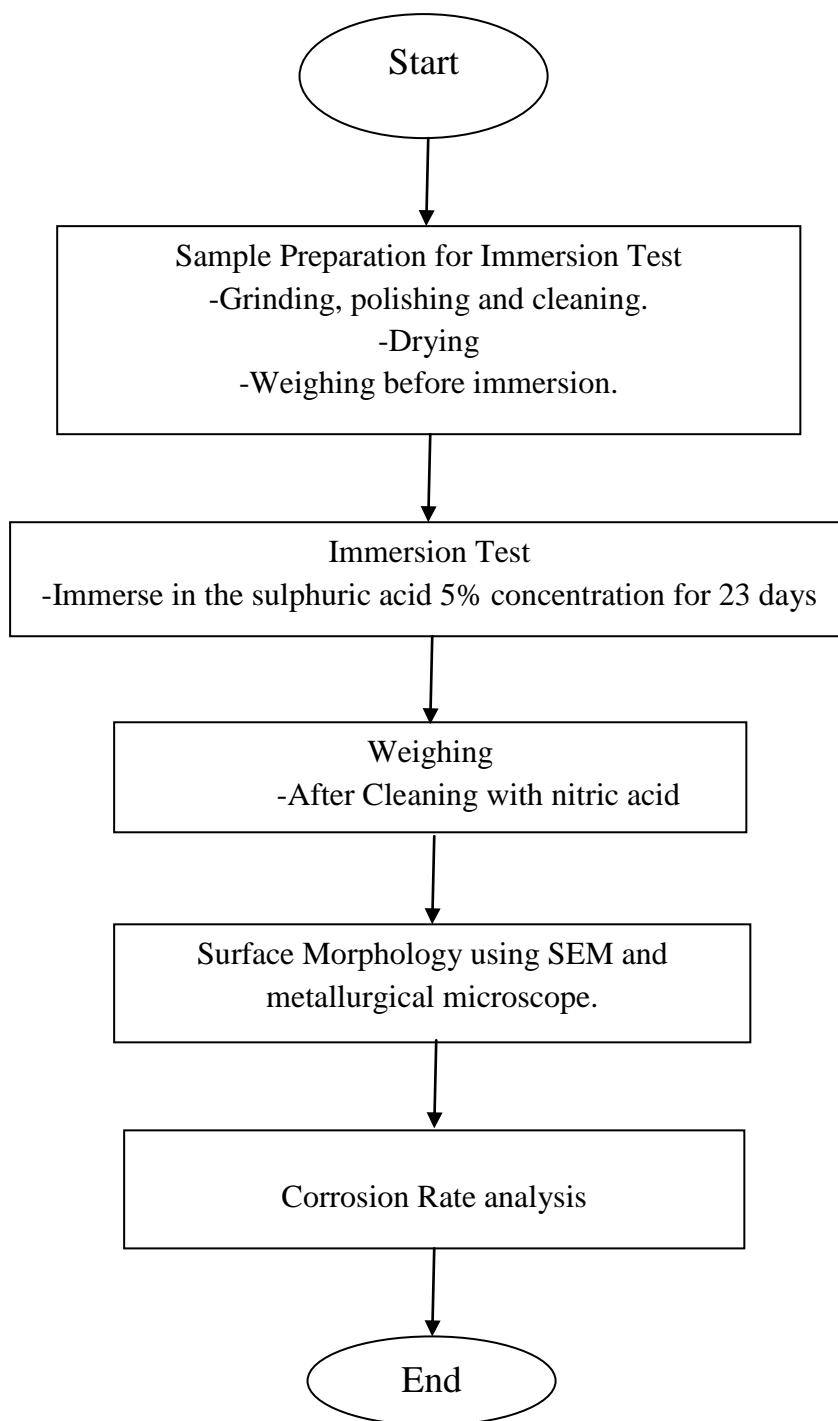


Figure 3.2 General immersion test procedures.

3.2 SPECIMEN PREPARATION FOR SOLUTION HEAT TREATMENT

The type of material used in this study is aluminium alloys 5083 sheet. Figure 3.3 shows the dimension of specimen. The specimens were cut by using hydraulic shear machine with the same dimension. The length of the specimen was set to be 40mm, width equal to 30mm and thickness of the sheet used was 3mm. A small hole, 2.5mm was drilling in the center of the specimen

The surfaces of specimens were cleaned from oil, grease and dirt by degreasing with a solvent cleaner and scrubbing with emery paper. The total surface area of the specimen is calculated by using formula (3.0).

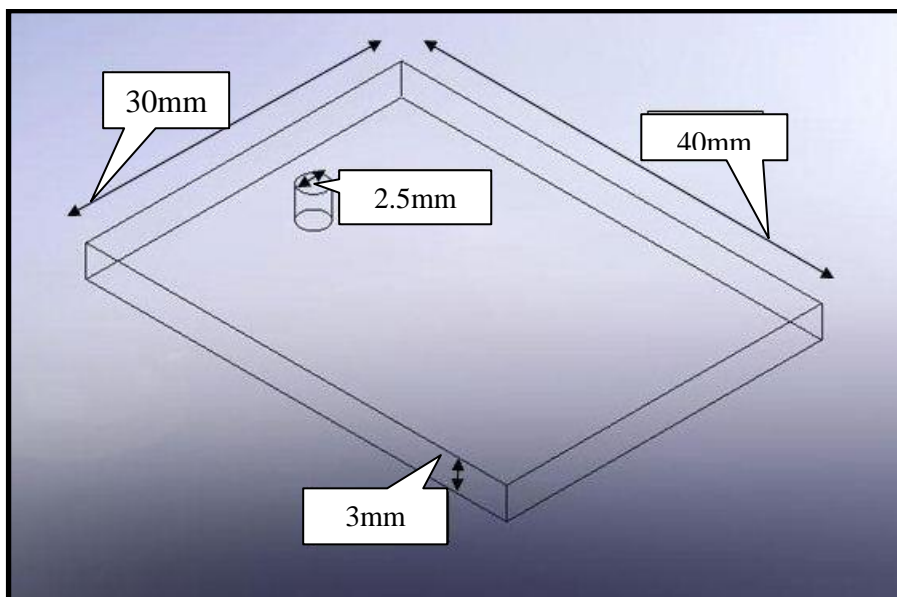


Figure 3.3: Dimension of specimen.

The total surface area of the specimen is given by the following equation:

$$\begin{aligned} \text{Total Area} = & 2(\text{Width} \times \text{Length}) + 2(\text{Width} \times \text{Thickness}) + 2(\text{Length} \times \text{Thickness}) \\ & - (\pi \times \text{Diameter}^2 / 2) + (\pi \times \text{Diameter} \times \text{Thickness}) \end{aligned} \quad (\text{Eq. 3.1})$$

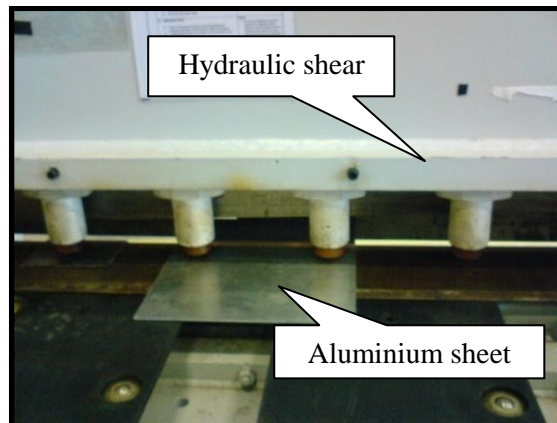


Figure 3.4: Cutting the specimen by using hydraulic shear machine.



Figure 3.5: Specimen after cutting process.

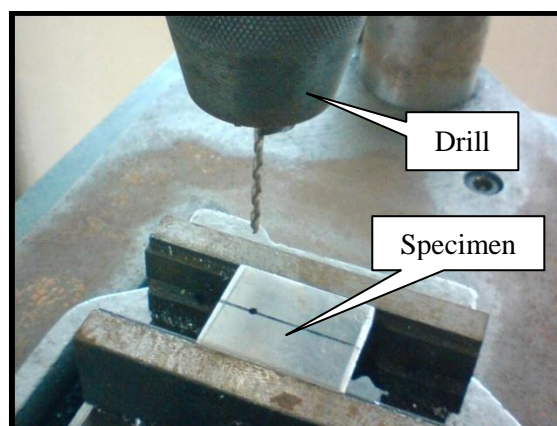


Figure 3.6: Drilling the specimen.

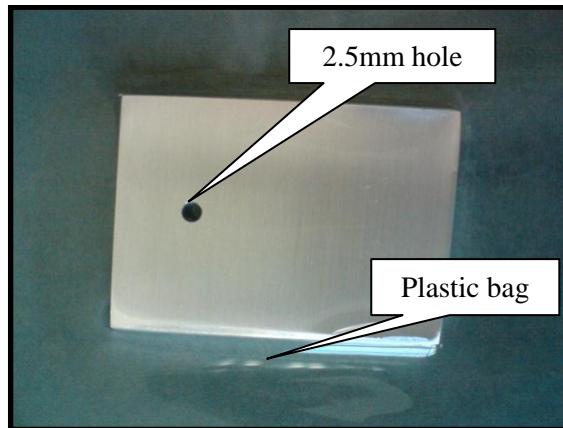


Figure 3.7: Specimen after grinding process.

Figure 3.4 shows the aluminium sheet was cut using hydraulic shear according to the dimension shown in Figure 3.3. Hydraulic shear was commonly used to cut sheet metal and other material that has low thickness. The specimens after cutting process were showed in the Figure 3.5. After cutting process, the specimens were drilled in the center to make a 2.5 mm hole as shown in Figure 3.6. After that, the specimens were cleaned and grinded to make sure the smooth surface was produced. Figure 3.7 shows the specimen after grinding and cleaning process. The specimens were kept in the plastic bag to keep clean from any dirty.

3.3 SOLUTION HEAT TREATMENT PROCESS

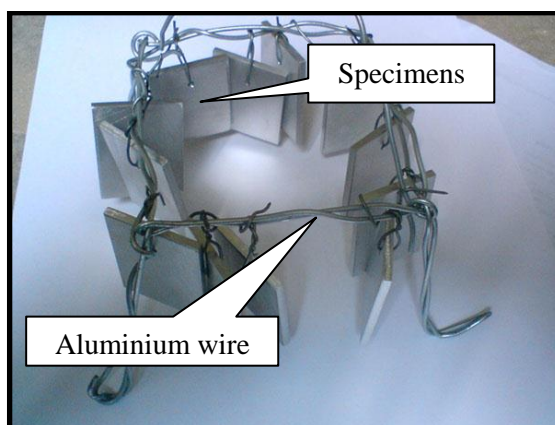
Before solution heat treatment process was carried out, the specimens were hung by using aluminium wire according to the Figure 3.8 to make sure the specimens were heat uniformly around the specimens and to prevent the overheated in the furnace. The specimens were typically heated for 60 minute at 420 °C in the furnace as shown in Figure 3.9.

After one hour, the specimens were quenched in the water at room temperature for 30 seconds to cool the specimens. Figure 3.10 shows the water used for quenching process. After quenching process, the specimens were aged at 180 °C with difference time of aging process. The experiments were repeated for 6 different times of aging process as shown in the Table 3.1.

Table 3.1: Time of aging process

Specimens	Time of aging process (minutes)
1	Control
2	30
3	60
4	90
5	120
6	180
7	240

Table 3.1 shows the specimens after solution heat treatment process. Specimen 1 is un-heated specimen and used as a control. Each specimen was aged at difference time of aging process. Specimen 2 was aged for 30 minutes, specimen 3 for 60 minutes, specimen 4 for 90 minutes, specimen 5 for 120 minutes, specimens 6 for 180 minutes and specimen 7 for 240 minutes. After aging process, specimens were cooled at room temperature.

**Figure 3.8:** Specimens were hung using aluminium wire.

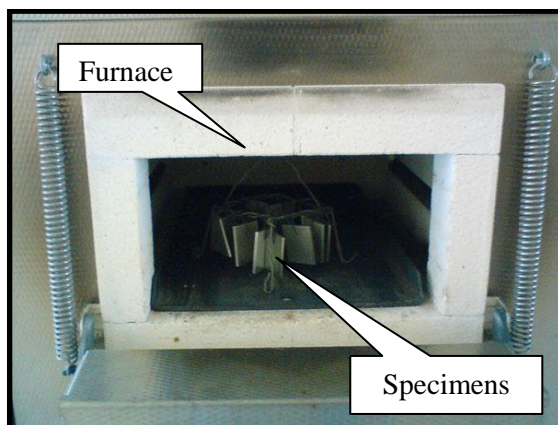


Figure 3.9: Specimens were heated in the furnace



Figure 3.10: Specimens were quenched in the water

3.4 MICROSTRUCTURES OBSERVATION

After solution heat treatment process, the surfaces of the specimens were observed by using Scanning Electron Microscope (SEM) and metallurgical microscope. The surface of the specimens were observed for determines the grain growth on the surface after being treated with difference time of aging process.

3.4.1 Specimen Preparation

Before the surface was analyzed, the specimens were cut into small size, mounted, grinded and polished to make sure the surface is very smooth and mirror

images were formed in the surface. The specimens were mounted according to Figure 3.11 for polishing process. The polishing process involves several stages.

First, the specimens were grinded using emery paper to remove any scratch on the surface of the specimens as shown in Figure 3.12. Then, the specimens were polished by using 6 μm DIAMAT Polycrystalline Diamond followed by 1 μm DIAMAT Polycrystalline Diamond as shown in Figure 3.13. The last stage, the specimens were polished by using 0.05 μm NANOPOLISH Alumina to form a mirror polished. Figure 3.14 shows the last process of polishing stages.

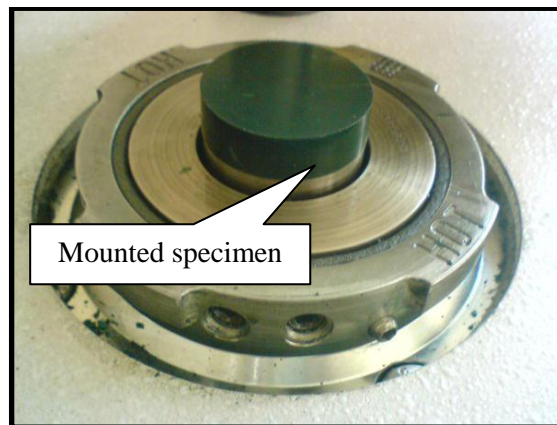


Figure 3.11: Specimens were mounting

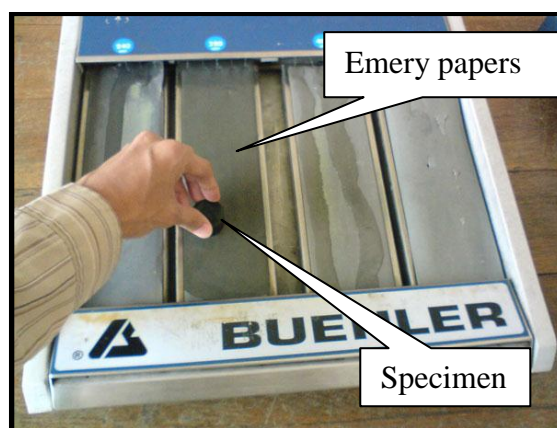


Figure 3.12: Specimens were grinded using emery paper



Figure 3.13: Specimens were polish using 6 µm DIAMAT Polycrystalline Diamond followed by 1 µm DIAMAT Polycrystalline Diamond



Figure 3.14: Specimens were polish using 0.05 µm NANOPOLISH Alumina.

For microstructure analysis, the surface of specimens were etched using etching solution for 1 minutes then cleaned using distilled water to make sure the grains were visible. The solution of etching was prepared for the specimens.

Table 3.2: Etchant for aluminium

Chemical Reagent	Amount (ml)
Distilled water	95.0
Hydrochloric Acid	1.5
Hydrofluoric Acid	1.5
Nitric Acid	2.5

The content and amount of chemical reagent used for etching process was shown in the Table 3.2. The procedures for specimen's preparation were followed based on the American National Standard (ASTM) depends on the suitability.

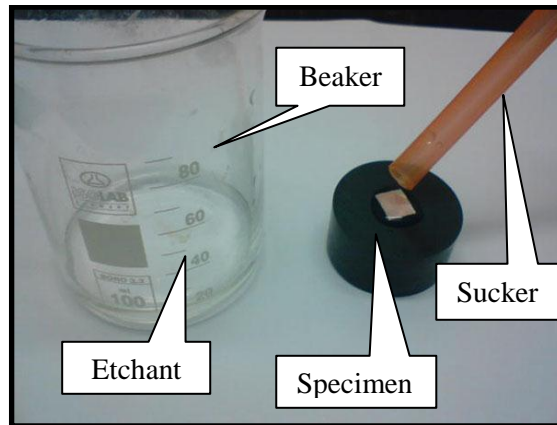


Figure 3.15: Specimens were etched using etching solution for 1 minute.

Figure 3.15 shows the specimens were etched using etching solution. The etching solution was drop on the surface of the specimens for approximately 1 minute. After 1 minute, the specimens were cleaned using distilled water and dried by using high air pressure blowing. After that, specimens were ready for microstructure analysis.

3.4.2 Metallurgical Microscope

After the specimens were etched for 1 minute, and the surface become fade, they were cleaning using distilled water and blowing with high air pressure to remove any liquid on the surfaces. The surfaces of specimen were analyzed using metallurgical microscope and the images of microstructure were taken. Figure 3.16 shows the metallurgical microscope used for microstructure analysis.

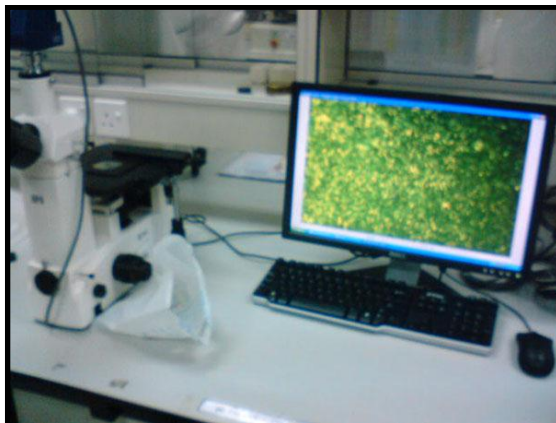


Figure 3.16: Metallurgical microscope

3.4.3 Scanning Electron Microscope

There are many advantages to using the SEM instead of a light microscope. The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification.

Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today. Figure 3.17 shows the type of SEM has used in the experiment.

3.4.3.1 Procedure for Using Scanning Electron Microscope

The procedures required for using SEM are:

- (i) Sample preparation
- (ii) Magnification range expected to be the most effective
- (iii) Depth of focus needed
- (iv) Choose working distance, aperture size, lens current, beam voltage

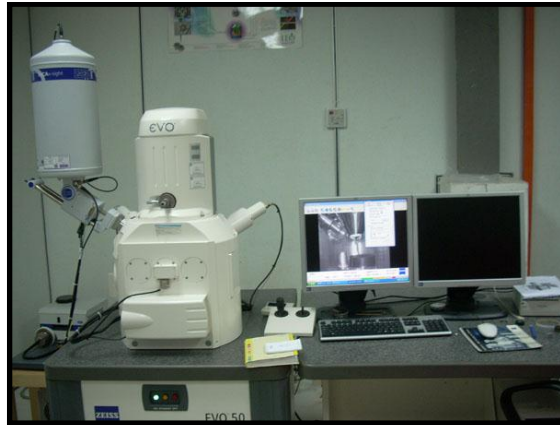


Figure 3.17: Scanning electron microscopy (SEM)

3.5 HARDNESS TEST

After heat treatment process, the specimens were test using Vickers hardness test to measure the hardness for different time of aging. Vickers hardness test is a general instrument used for measuring the hardness of the materials. It consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf.

The full load normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load were measured using a microscope and their average calculated. The area of the sloping surface of the indentation was calculated.

Before used Vickers hardness test, the specimens were grinded and polished to make sure the surfaces were smooth and obtained an accurate result during the test. Ten values for each specimen were taken around the specimens and the average values were calculated for accurate result. Figure 3.18 shows the Vickers Hardness Test used in the experiments.

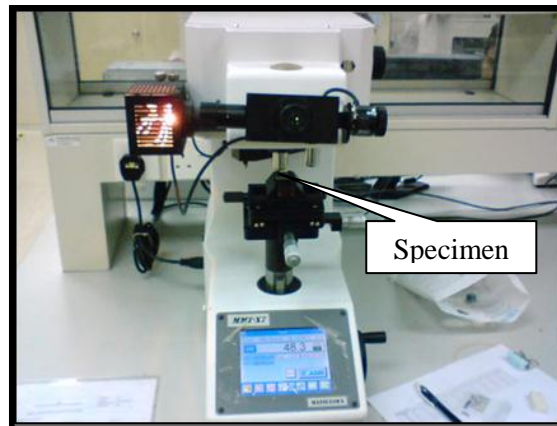


Figure 3.18: Vickers hardness test

3.6 IMMERSION TEST

The immersion test was carried out by immerse the specimens in the sulphuric acid 5% for 23 days. The immersion test was carried out at room temperature around 20 °C to 25 °C. The procedures used for this experiment based on the Standard Practice for Laboratory Immersion Corrosion Testing of Metal (ASTM G 31-72). This practice cover all specimen preparation, time of exposure in chemical and procedure used along immersion process.

3.6.1 Specimens Preparation

After heat treatment process, the specimens were cleaned from material remaining at the surface of specimens. This cleaning procedure was followed according to American National Standard, ASTM G 1 -03. This practice covers suggested procedures for preparing bare, solid metal specimens for test, for removing corrosion products after the test has been completed, and for evaluating the corrosion damage that occurred. Before immersion test, the specimens were prepared by polishing process until mirror images were formed in the surfaces of the specimens as shown in Figure 3.19



Figure 3.19: Specimen for immersion Test

3.6.2 Weighing Before and After Immersion Test

After cleaning and drying process, the specimens were weighed using micro weighing scale as shown in Figure 3.20 to take the initial weight of the specimens. After the specimen was immerse in the solution of sulphuric acid, the specimen was weighed again to take the final weight of the specimens by using same digital micro weighing scale. The value of weight loss is calculated. The weight different between before and after exposure are representing the weight loss of the specimen.

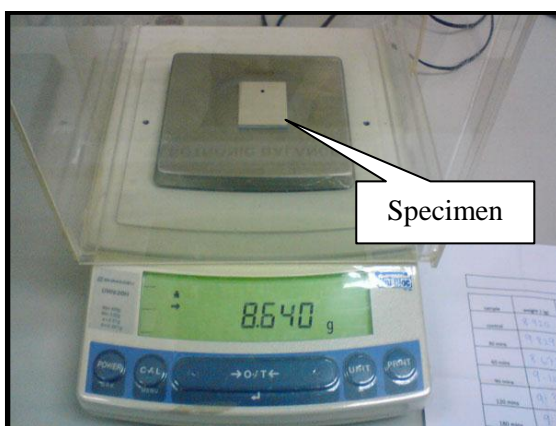


Figure 3.20: Micro weighing scale

3.6.3 Immersion Test Setup

The specimens were hung in the solution of sulphuric acid 5% concentration by using a insulator wire which attached to the wood at the top of beaker. Insulated wire is used because it does not involve in any corrosion process and will not affect the solution used.

The solution used in this test is sulphuric acid because aluminium alloys are not resistance in sulphuric acid. Volume of the sulphuric acid used is 1000ml. The volume of the acid should be large enough to avoid any appreciable change in its corrosivity during the test, or accumulation of corrosion products that might affect further corrosion. The Figure 3.21 and 3.22 show the setup of the experiments.

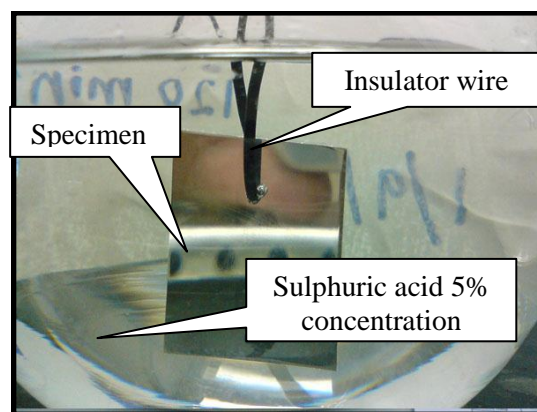


Figure 3.21: Specimen was immersed in sulphuric acid



Figure 3.22: Setup of the Experiment

3.6.4 Cleaning After Immersion Test

Corrosion removal product can be divided into three general categories which are mechanical, chemical and electrolytic. But, in this practice only mechanical and chemical procedures were used. Mechanical procedures are the methods that often utilized to remove heavily encrusted corrosion products which include scraping, scrubbing, brushing, ultrasonic cleaning, mechanical shocking, and impact blasting.

Chemical procedures involve immersion of the corrosion specimens in a specific solution that is designed to remove the corrosion products with minimal dissolution of any base metal.

After immersion test, the specimens were cleaned from any corrosion products that deposited at the surface of material before weighing process. Nitric acid, HNO_3 30% concentration was used to remove extraneous deposits and bulky corrosion products to avoid reactions that may result in excessive removal of base metal. The specimens were immersed in the nitric acid for 1 to 5 minutes depending on the type of corrosion on the surfaces of the specimens. Figure 3.23 shows the cleaning process.

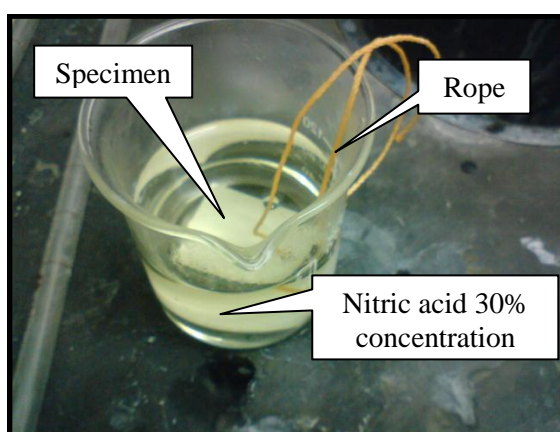


Figure 3.23: Cleaning process

After cleaning using nitric acid, specimen were cleaned using distill water and dried using high pressure of air. The specimens were put in the plastic bag and labeled.

Figure 3.24 shows the finish process of the specimens and weighed again using weighing scale.

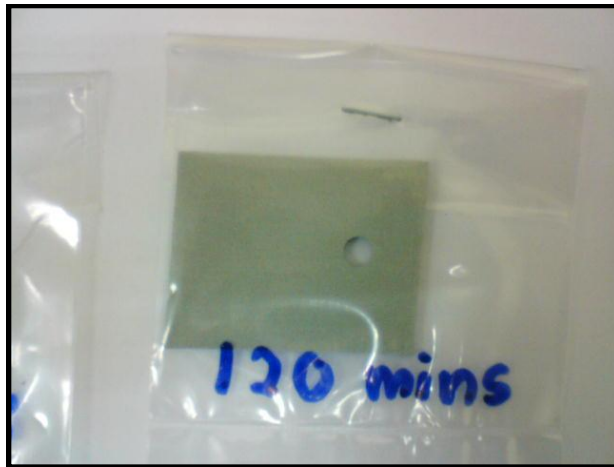


Figure 3.24: Labeled specimen

3.7 CORROSION RATE ANALYSIS

The initial total surface area of the specimen (making correction for the areas associated with mounting holes) and the mass lost during the test are determined. The average corrosion rate is calculated by using equation below:

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D} \quad (\text{Eq. 3.2})$$

Where,

K= a constant (3.45×10^6) for corrosion rate unit in mils per year (mpy)

A = area (cm^2)

W = mass loss in grams

D = density (g/cm^3)

T = Time of exposure in hours

Corrosion rates are not necessary constant with time of exposure. Many different units are used to express corrosion rate. The corrosion rate may also be expressed such as mil per year (mpy), inches per year (ipy), inches per month (ipm), and others.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

This chapter discussed about the results obtained from the solution heat treatment, immersion test for corrosion and hardness test by using Vickers Hardness Test. The microstructures of specimens were examined by using Metallurgical Microscope and Scanning Electron Microscope (SEM). The corrosion rate was calculated using weight loss method.

4.2 MICROSTRUCTURAL OBSERVATION

After solution heat treatment process, the microstructures of the specimens were analyzed using SEM and metallurgical microscope. Different time of aging produced difference type of grains size and grains boundaries on the surface of the specimens. By using metallurgical microscope, the different pattern of microstructures produced can be observed on the surface of specimens.

Figure 4.1 shows the microstructure of specimen 1 which is an un-heated specimen used for control and to compare with aging specimens. The grains boundaries can be distinguished clearly after etching process. The size of grain is estimated to be around 20 μ m to 60 μ m according to the Figure 4.1.

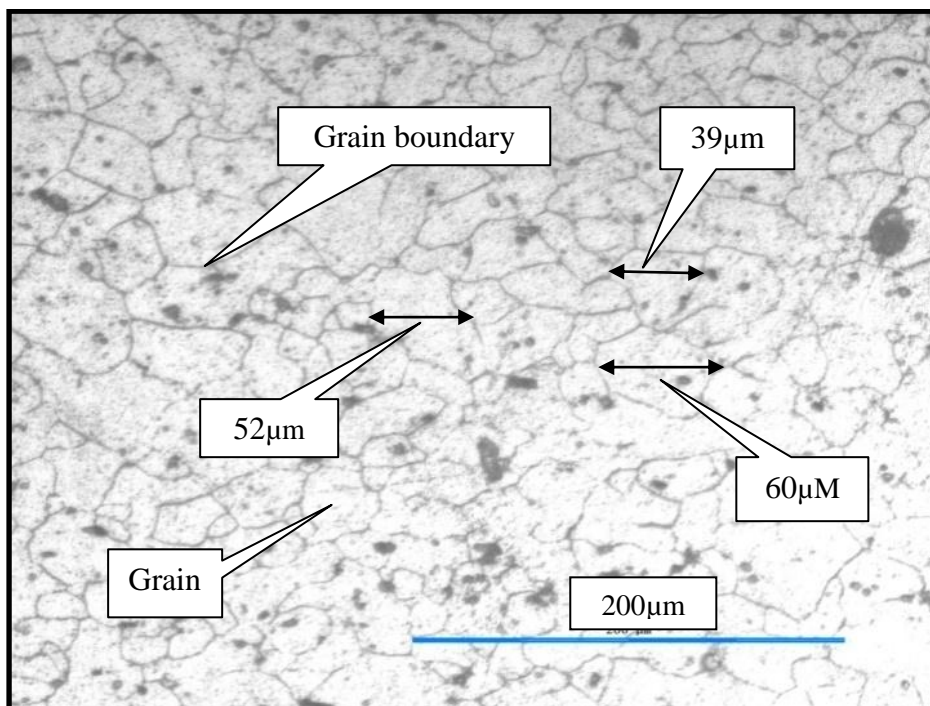


Figure 4.1: Microstructure of specimen 1 using metallurgical microscope (200X)

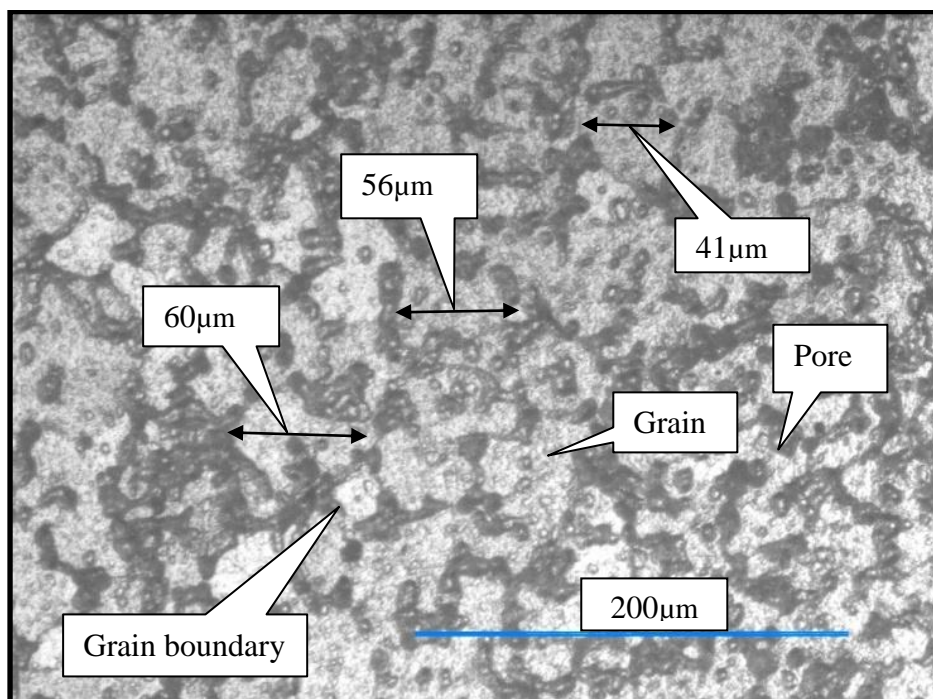


Figure 4.2: Microstructure of specimen 2 using metallurgical microscope (200X)

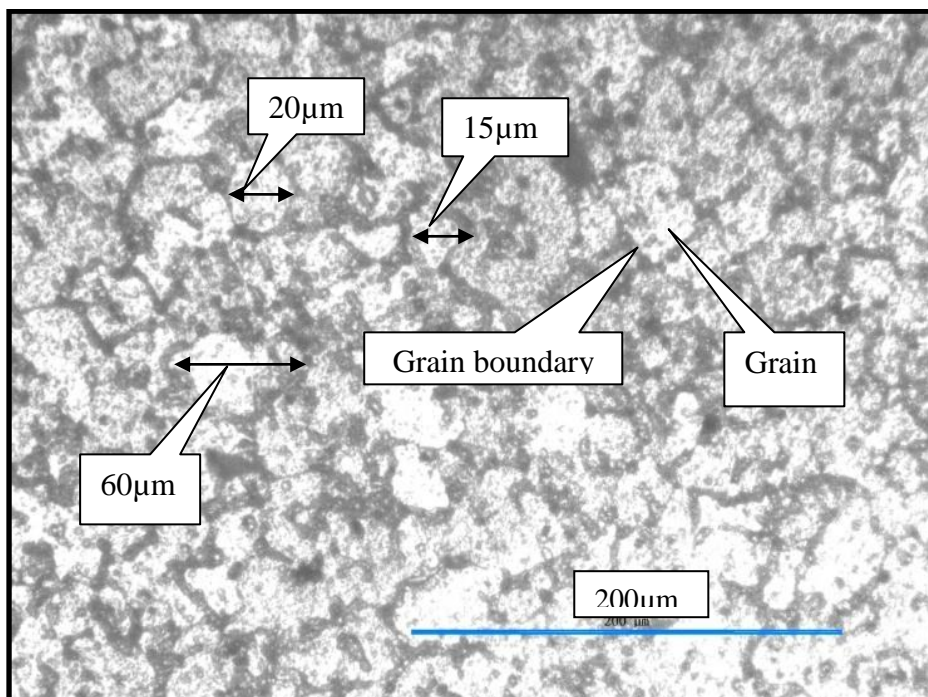


Figure 4.3: Microstructure of specimen 3 using metallurgical microscope (200X)

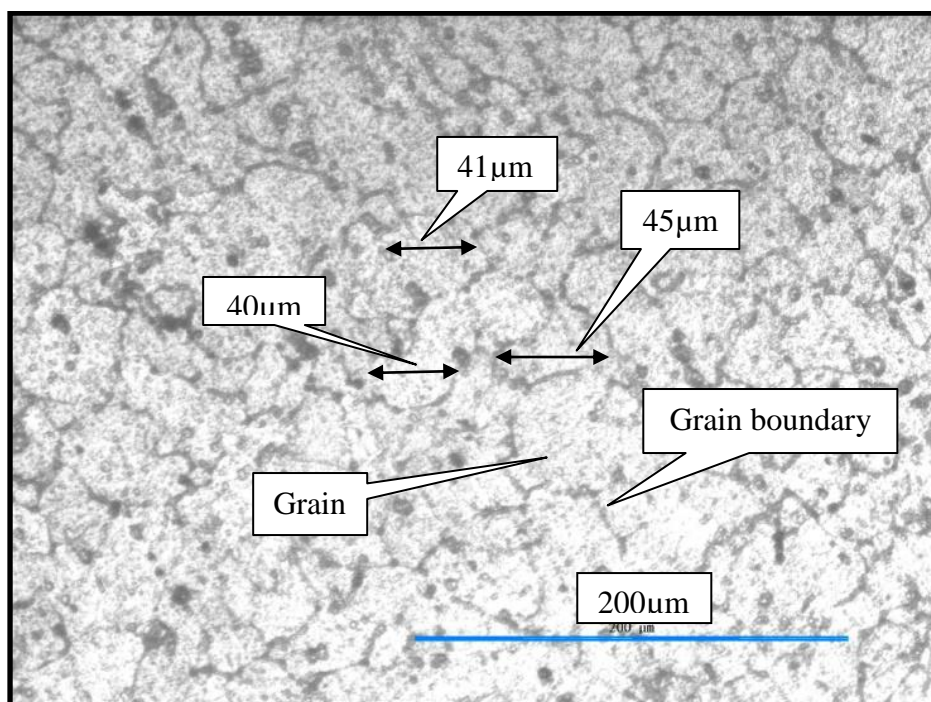


Figure 4.4: Microstructure of specimen 4 using metallurgical microscope (200X)

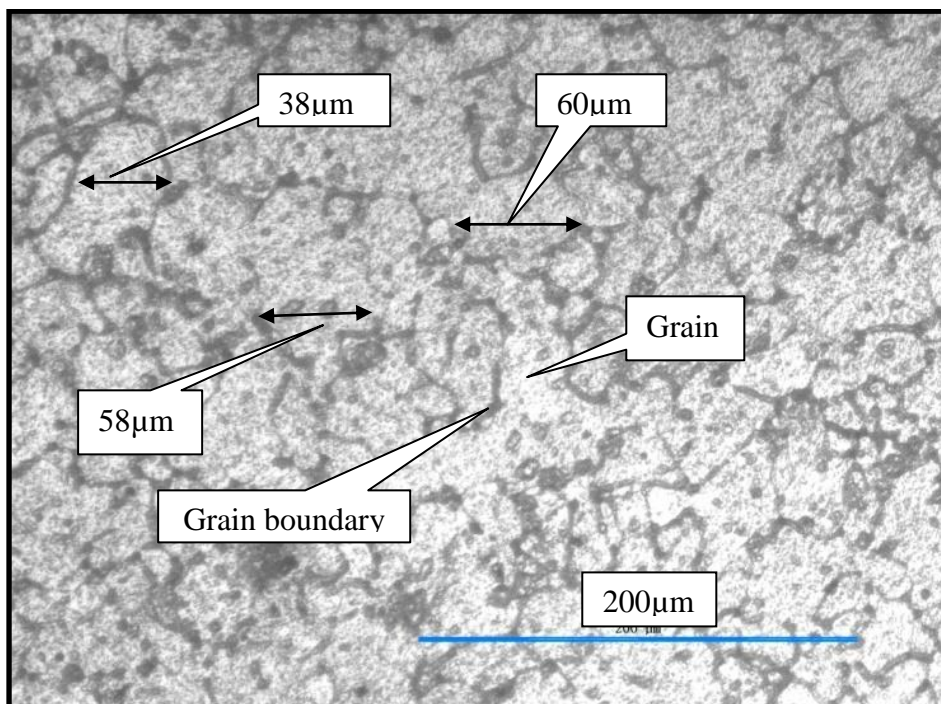


Figure 4.5: Microstructure of specimen 5 using metallurgical microscope (200X)

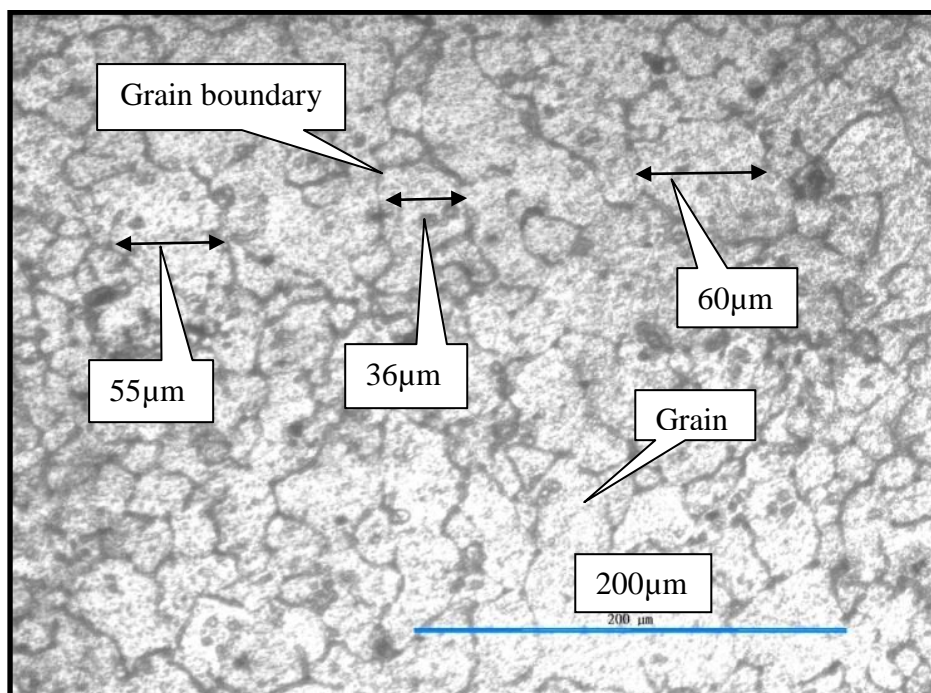


Figure 4.6: Microstructure of specimen 6 using metallurgical microscope (200X)

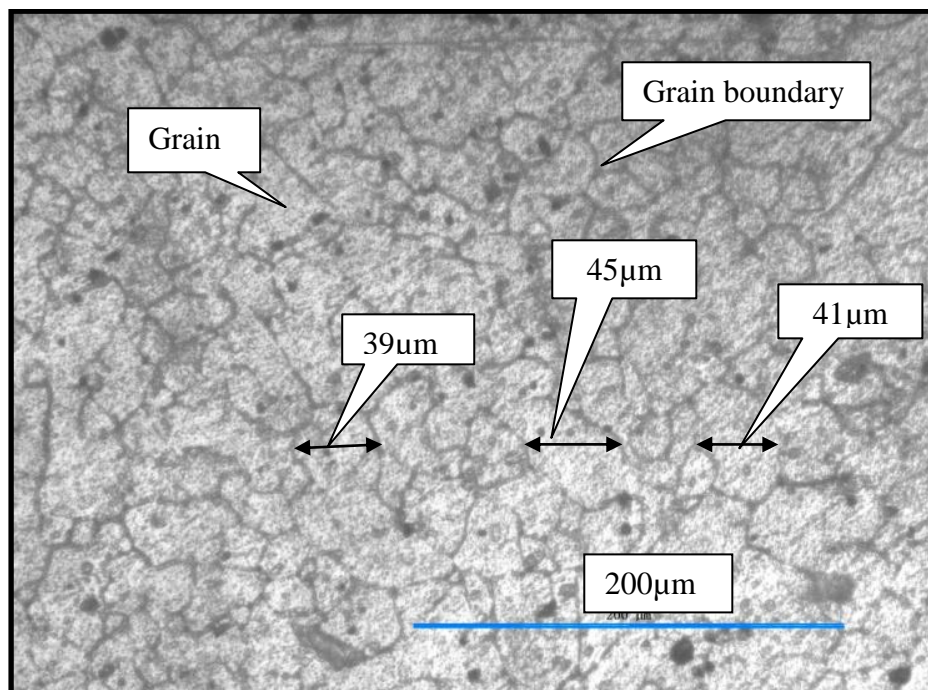


Figure 4.7: Microstructure of specimen 7 using metallurgical microscope (200X)

Figure 4.2 shows the microstructures of specimen 2. From the diagram, the grain boundaries are not clearly visible and it appears to expand and distribute around the surface. The grains produced were uniform distribution and almost similar in size. The precipitates were evident both in the grains and along grain boundaries. The number of pores appears between grain boundaries.

Figure 4.3 shows the microstructures of specimen 3. The grains are not really visible and the pores appear along the surface. With increasing time of aging process, the grain boundaries will be visible due to migration of precipitates. The sizes of grains were estimated around 60µm to 15µm.

From Figure 4.4 to 4.7, the grain sizes of specimen 4-7 were found to be around 30µm to 60µm. The grains were estimated to become smaller compared to specimen 2 and 3. The grain boundaries become more clearly compared to specimen 2 and 3.

The decreasing of grains size in specimen 4-7 happened due to the precipitate changes. The precipitate was grown at increase time during artificial aging process. With the time, these precipitates dissolved into the material, diffusing throughout the whole structure. When the time of aged increase, the grains also appears and become smaller in size. According to Youdelis, this happened due to the formation of partial subgrain and the rearrangement of some dislocation. After aged at maximum time, for 240 minutes, the grains boundaries can be seen clearly. Meanwhile, the width of subgrain increases slightly. A small quantity of nearly equiaxed fine grains was observed at increasing of time. Therefore, the grain boundaries appear with increasing time of aging process.

4.2.1 Evaluation of Oxide Film

Aluminium alloys usually forms a thin, compact, and adherent oxide film on the surface that limits further corrosion. When formed in air at atmospheric temperature, it is approximately 5 nm thick. If formed at elevated temperatures or in the presence of water or water vapor, it will be thicker. According to Schweitzer, this oxide film is stable in the pH range of 4–9. The oxide film is not homogeneous and contains weak points. Breakdown of the oxide film at weak points leads to localized corrosion.

Figure 4.8 shows the thickness of the un-heated aluminium oxide film by using SEM with magnification of 1500X. The thickness of film appears to be darker compare to the surface of aluminium alloys. The thickness produce in this figure is 72.88 μ m.

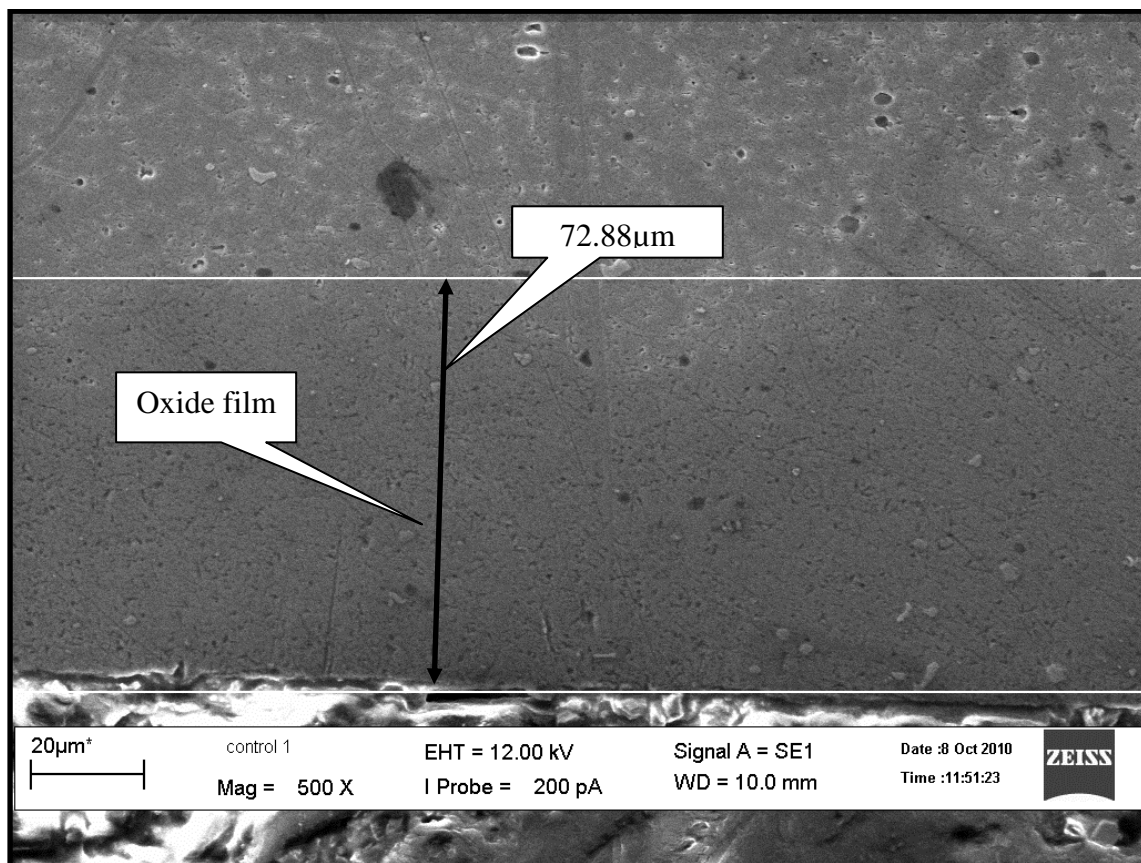


Figure 4.8: Oxide film on the surface of un-heated aluminium alloy using SEM (1500X)

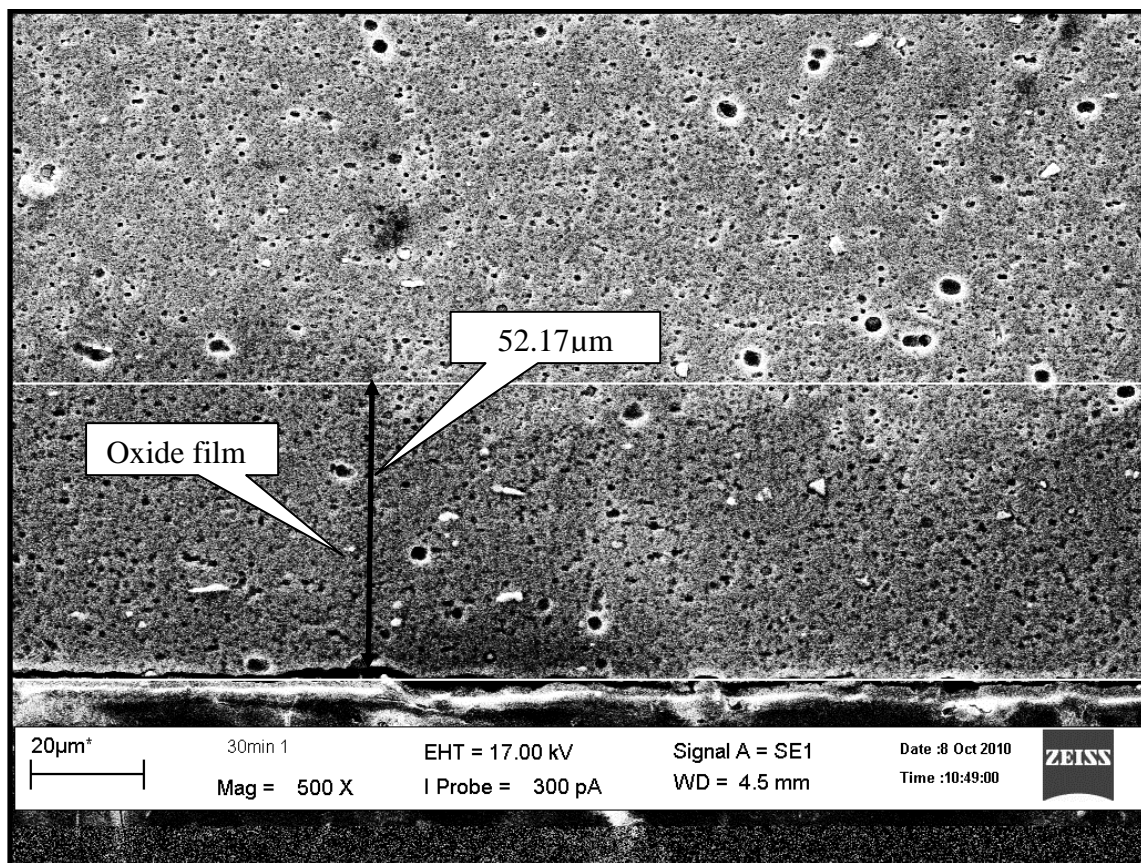


Figure 4.9: Oxide film on the surface of aluminium alloy after heat treatment and aged for 30 minutes at 180°C (1500X)

Figure 4.9 shows the thickness of the aluminium oxide film after heat treatment and aged for 30 minutes by using SEM with magnification of 1500X. The thickness of film appears to be thinner compare to the un-heated aluminium alloys. The thickness observed in this figure is 52.17 μm. The thickness was decreased due to the heat treatment.

4.3 HARDNESS TEST ANALYSIS

Vickers Test was used to measure the hardness of specimens after solution heat treatment process. Table 4.1 shows the hardness of the specimens artificially aged at 180°C after being heat treated for 60 minutes at 420°C and quenched in the water. Each hardness measurement was repeated 10 times in the whole surface of the specimen to get the accurate results and the average of the data was represented in the Table 4.1.

Table 4.1: Vickers Hardness (HV)

Specimens	Average (HV)
1	46.5
2	34.9
3	37.6
4	35.7
5	38.4
6	38.2
7	40.2

From the result in the Table 4.1, the specimen 1 shows the highest hardness. After solution heat treatment, the hardness of the specimens slightly decreases as exhibited by sample 2-7.

Figure 4.10 was plotted to get the comparison between each specimen. The variation of hardness exists between specimens and the difference of hardness values between un-heated with heat treated specimens can be clearly seen. From Figure 4.10, the hardness value of specimen 1 which was un-heat-treated specimen used for control is 46.5HV which is the highest compared to the heat treatment specimen which around 34.9HV to 40.2HV. After heat treatment, the maximum hardness was shown by specimen 7 by aging for 240 minutes. The minimum hardness was shown by specimen 2 by aging for 30 minutes.

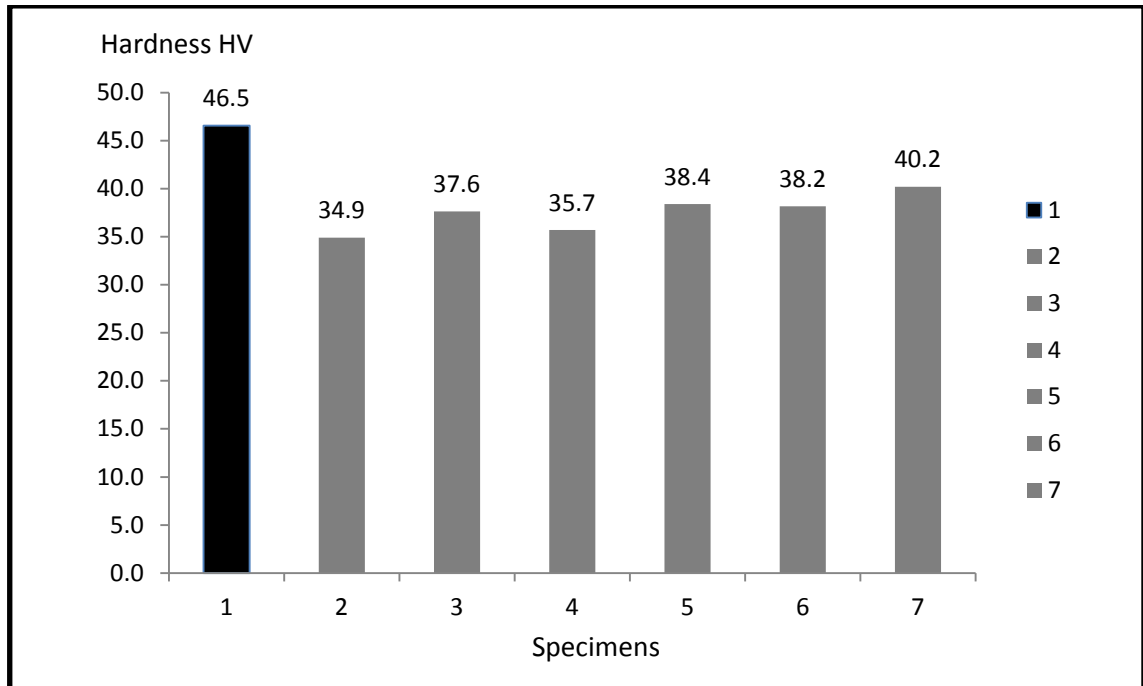


Figure 4.10: Graph of Vickers hardness test

Solution heat treatment alone decreased the hardness significantly, whereas after subsequent aging the hardness increased more or less to the original hardness of the as-received condition. This decrease can be attributed to the dissolution of residual S' phase (a matrix hardening phase) that was present in the as-received material. This result is in agreement with the work of Youdelis and Fang who reported that maximum hardness is achieved by the presence of the S' phase whereas the transformation of S' phase to the S phase by solution treatment reduces the hardness considerably.

According to Hummel, the highest strength was generally achieved when a large amount of closely spaced, small and round precipitates are coherently dispersed throughout an alloy. The variation of hardness produces when the specimens were subjected to the difference time of aging process. Extended aging time to 240 minutes has brought aluminium alloy 5083 to reach the optimum hardness, which was 40.2 HV.

Thus, if longer aging time is given, the hardness might still be able to increase a little to reach its highest strength.

4.4 IMMERSION TEST ANALYSIS

To evaluate the corrosion resistance of Al alloy, immersion tests were done for 23 days. The specimens were analyzed in terms of surface analysis, corrosion types occurred on the specimens and corrosion rate analysis.

4.4.1 Visual Inspection of Specimen

Figure 4.11 shows the specimen before immersion test. The specimens were shiny and a mirror image was observed on the surfaces. The specimens were mirror polished to make sure the corrosion products were formed uniformly around the surfaces.



Figure 4.11 Specimen before immersion test

From the visual inspection after immersion test in sulphuric acid for 23 days as shown in Figure 4.12, the surface of the specimen was observed to be in a whitish color, which represents the corrosion product of aluminium hydroxide. The whole surface of the specimen was affected by corrosion due to the reaction with sulphuric acid. The thickness slightly decreased and the diameter of the hole was expanded. The weight also decreased, and this represents the corrosion rate of the specimen. This shows that the reaction occurred on the entire surface of the aluminium, which was exposed to the acid.

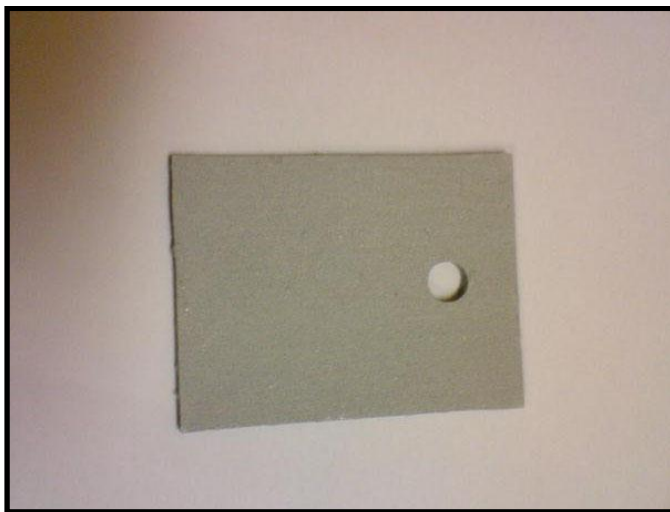


Figure 4.12 Specimen after immersion test

4.4.2 Corrosion Types

The corrosion type occur on the surfaces of specimens was determined to be uniform corrosion. These types of corrosion occur as the result in a fairly uniform penetration over the entire exposed specimen surface.

According to Davis, the uniform corrosion result from local corrosion cell action, that is multiple anodes and cathodes are operating on the metal surface at any given time. The location of the anodic and cathodic areas continues to move about on the surface, resulting in uniform corrosion. Uniform corrosion often results from atmospheric exposure, exposure in fresh, brackish, and salt waters, or exposure in soils and chemicals.

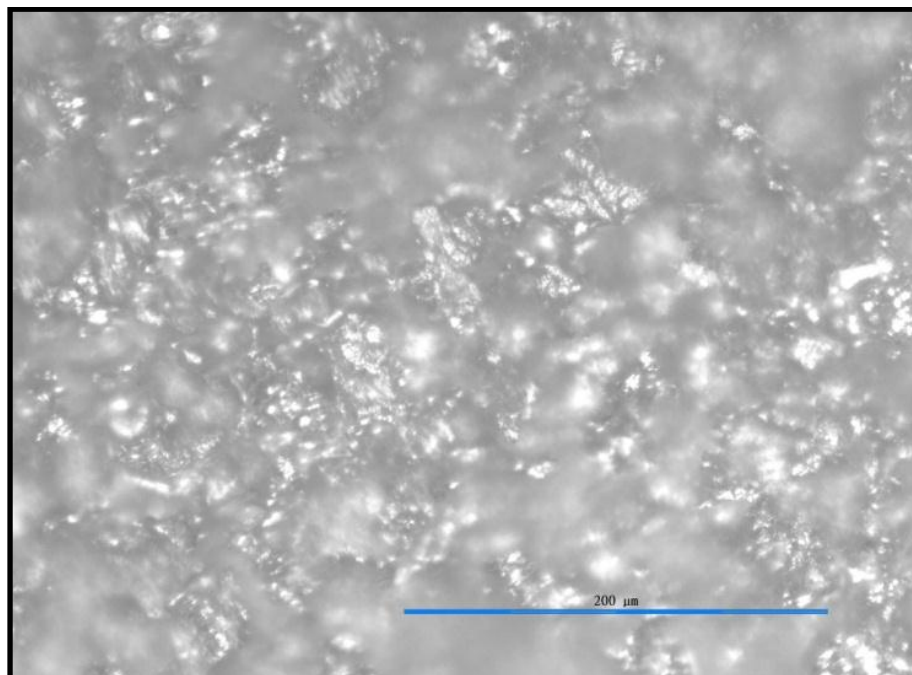


Figure 4.13: Surface of specimen after immersion test for 23 days using metallurgical microscope (200X)

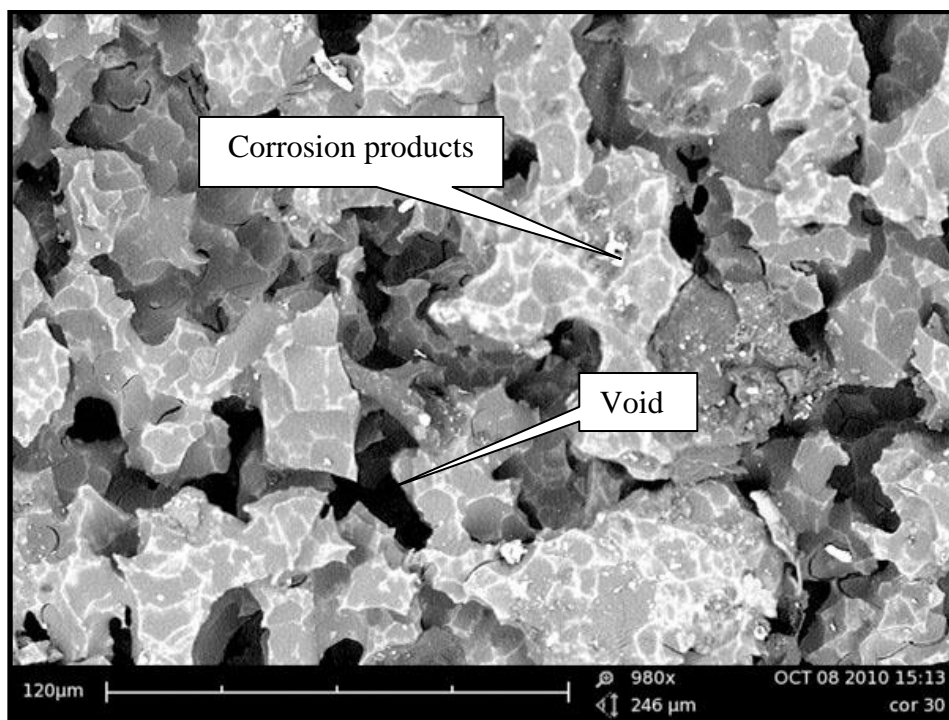


Figure 4.14: Surface of specimen after immersion test for 23 days using SEM (980X)

Figure 4.13 shows the surface of the specimen after immersed in the solution of sulphuric acid for 23 days by using metallurgical microscope. The corrosion product on the surface appears in the uniform distribution and this kind of corrosion is called uniform corrosion. By using Scanning Electron microscope with magnification of 980X, the corrosion product clearly seen in Figure 4.14 and it acts as barrier for the corrosion on the surface of specimens. Figure 4.15 show the thickness of the corrosion product. The thickness of uniform corrosion is around $11\mu\text{m}$

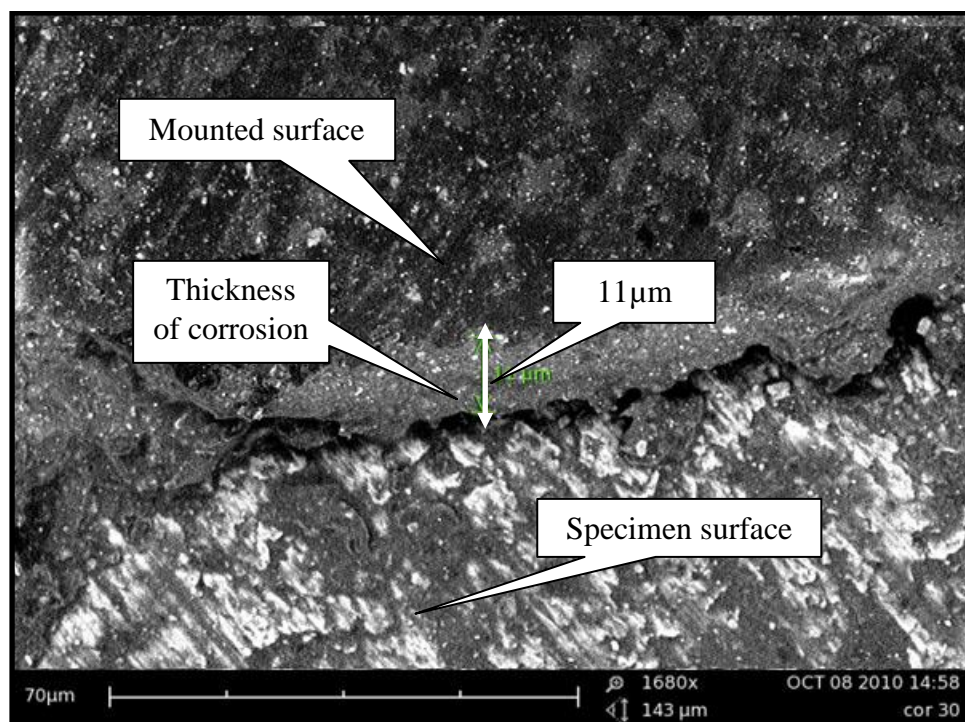


Figure 4.15: Thickness of corrosion using SEM (1680X)

4.4.3 Corrosion Rate Determined Based On Weight Loss Method

Corrosion rate of each sample was calculated based on weight lost method. The weight lost per area after immersion test represent corrosion rate of the specimens as discussed in Chapter 3. Table 4.2 shows the initial weight of the specimen while Table 4.3 shows the weight of the specimen after immersion. Three reading were taken and average values were calculated to get the accurate result.

Total weight loss of each specimen after the immersion test was determined by calculating the differences between the weights before immersion with the weight after immersion. The weight from calculation represents the weight loss during immersion test.

Table 4.2 Weight before immersion test

Specimens	Weight before (g)
1	8.920
2	9.830
3	8.671
4	9.142
5	9.314
6	9.128
7	8.640

Table 4.3 Weight after immersion test

Specimens	Weight after (g)
1	0.200
2	5.375
3	4.344
4	5.734
5	3.890
6	4.084
7	3.539

Table 4.4 showed the result of weight loss. Specimens with non-heated show the highest weight loss compared to the heat treated specimens.

Table 4.4 Weight loss

Specimens	Weight loss (g)
1	8.720
2	4.455
3	4.327
4	3.408
5	5.424
6	5.044
7	5.101

From the Table 4.4, the highest weight loss showed by specimen 1 which was 8.720g. Specimen 1 was used as a control and to compare the result with the heat treated specimens. The lowest weight loss was showed by specimen 4 with aging for 90 minutes.

4.4.4 Total Area of Specimens

Each specimen has different total areas due to the specimen preparation process such grinding and polishing process. The polishing process is important to make sure the material is mirror polished and the corrosion products after immersion test were clearly visible. After polishing process, the dimension of the length, width and thickness were taken and the total areas were calculated. Therefore, the total area of specimens were calculated were showed in the Table 4.5.

Table 4.5: Total area of specimens

Specimens	Total area (mm)
1	2888.053
2	2996.619
3	2804.079
4	2824.196
5	2866.361
6	2866.361
7	2879.613

4.4.5 Corrosion Rate Calculation

The corrosion rate was calculated for each specimen using the initial and final weights, surface area, and density of the alloy and exposure time by using the formula. The use of corrosion rates implies that all mass loss has been due to general corrosion and not to localized corrosion, such as pitting or intergranular corrosion of sensitized areas on welded specimens. The sample of corrosion rate was calculated below and the results were showed in the Table 4.6.

Specimen 1 (control)**Constant for mpy**

$$\begin{aligned} K &= \text{constant} \\ &= 3.45 \times 10^6 \end{aligned}$$

Total Area

$$\begin{aligned} A &= 2(\text{Width} \times \text{Length}) + 2(\text{Width} \times \text{Thickness}) + 2(\text{Length} \times \text{Thickness}) \\ &\quad - (\pi \times \text{Diameter}^2/2) + (\pi \times \text{Diameter} \times \text{Thickness}) \\ &= 2(29.40 \times 42.10) + 2(29.40 \times 2.8) + 2(42.10 \times 2.8) - (\pi \times 2.5^2/2) + \\ &\quad (\pi \times 2.5 \times 2.8) \\ &= 2888.053 \end{aligned}$$

Weight Loss

$$\begin{aligned} W &= \text{weight before} - \text{weight after} \\ &= 8.920 - 0.200 \\ &= 8.720 \end{aligned}$$

Density of Aluminium

$$D = 2.65 \text{g/cm}^3$$

Time of exposure in hour

$$\begin{aligned} T &= \text{days} \times \text{hours} \\ &= 23 \times 24 \\ &= 552 \text{ hours} \end{aligned}$$

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

$$= \frac{3.45 \times 10^6 \times 8.720}{2888.053 \times 552 \times 2.65}$$

$$= 7.1211 \text{ mpy}$$

Table 4.6 shows the corrosion rate of all specimens. The corrosion rate was obtained by calculation using some formula. Corrosion rate in this table represented in mpy, means the mile per year. The unit of corrosion rate depends on the constant used during calculation.

Table 4.6: Corrosion rate

Specimens	Corrosion rate (mpy)
1	7.1211
2	3.5063
3	3.6394
4	2.8460
5	4.4630
6	4.1503
7	4.1779

Figure 4.16 show the corrosion rate of the specimens with difference time of aging process. The specimen 1 undergoes the maximum corrosion during immersion test which was 7.1272 mile per year. The specimen 4 show minimum corrosion rate means the heat treatment improve the corrosion resistance of aluminium alloys.

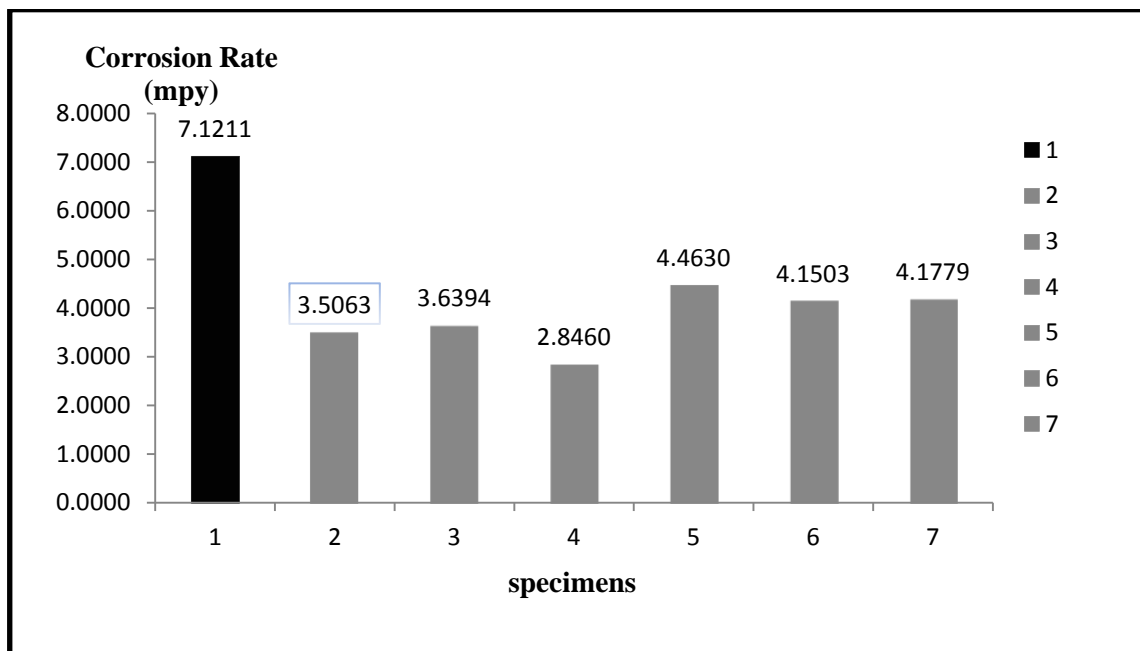


Figure 4.16: Graph of corrosion rate

The lowest corrosion rate showed by the specimen 4 with aging for 90 minutes which was 2.8485 miles per year and it is represent the optimum aging time to improve corrosion resistance. This analysis led to conclusion that the optimum aged could be achieved at 90 minutes aging time.

4.4.6 Relation between Hardness and Corrosion Rate

From the corrosion rate and hardness test result, the scatter plot was constructed to get the relation between corrosion rate and hardness of the specimens.

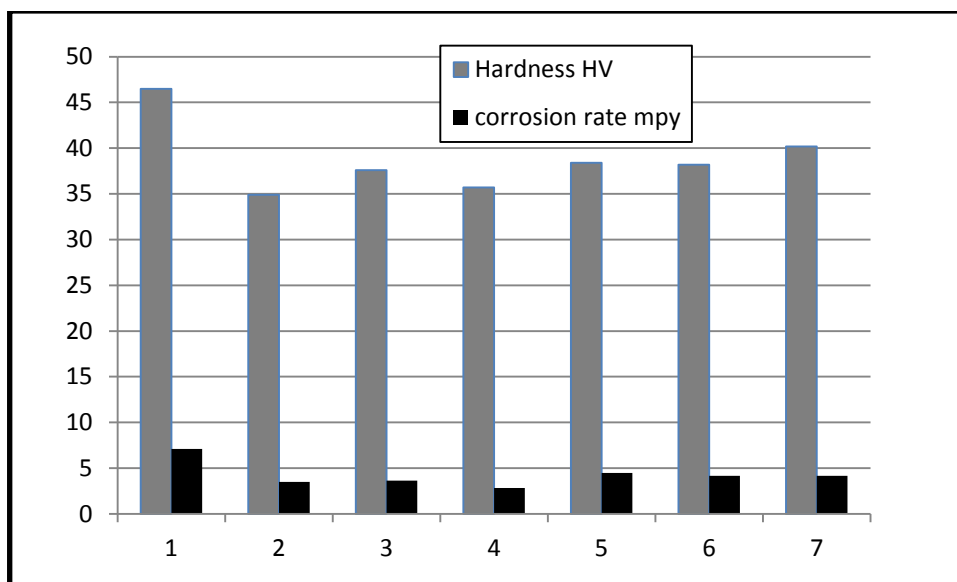


Figure 4.17: Relation between hardness and corrosion rate

According on the Figure 4.17, the relation between the corrosion rate and hardness can be established. Based on the graph, specimen 1 shows the highest corrosion rate and hardness. After heat treated, both the values of hardness and corrosion rate significantly decreased. When time of aging increase, the hardness slightly increase followed by corrosion rate. After that, the hardness slightly decrease and corrosion rate also decrease. From the variation of hardness, we can conclude, corrosion rate is increase when hardness increases and decrease when hardness is decreased.

The grains size produce after solution heat treatment also contributes to the hardness changes. With increasing of aging time, the grains size appears to become smaller. Thus, the hardness of specimens also increased. The corrosion rate also depends on the microstructure changes. Before solution heat treatment, the grains appear more clearly and size of grains also greater. Therefore, the corrosion rate is greater. After solution heat treatment, the grains size become smaller due to the distribution of precipitate and decreased the corrosion rate.

The hardness of specimens was greater when the precipitates were small and finely dispersed. Therefore, to gain the hardness, the specimens should be heat treated to produce a fine dispersion of small precipitates. From the graph, the corrosion rate increased with increasing the hardness. Hence, the increasing of hardness will affect the corrosion rate to increase.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 INTRODUCTION

This chapter will conclude the research and briefly discussed about the recommendation that can applied for the future works in order to improve the result and experimental procedures. The conclusions were done according to the result obtain in Chapter 4 and previous study.

5.2 OBJECTIVES ACHIEVED

The main objective of the study is to investigate the effect of heat treatment on the corrosion resistance of aluminium alloys 5083 by using immersion test method. This objective has been achieved since the experiments have been done and the results of the corrosion rate have been calculated. The corrosion type occurs after immersion test has been determined and the effect of solution heat treatment on the hardness also has been measured by using Vickers hardness test. Some analysis regarding the solution heat treatment and corrosion rate have been discussed in chapter 4.

5.3 CONCLUSION

As conclusion, the investigations of the effect of heat treatment on the corrosion resistance of aluminium alloys have been done successfully. The principle conclusions are summarized as followed:

- i. The corrosion resistances of aluminium alloys increased after solution heat treatment process. It was observed that different aging times correspond to different corrosion rate. Lowest corrosion rate was found at 90 minutes at 2.8460mpy while the control specimen shows highest corrosion rate at 7.1211mpy.
- ii. The hardness of the aluminium alloys decreased after solution heat treatment process with different value at different aging time. The optimum time of aging process which produces the maximum hardness was found at 240 minutes while minimum hardness was found at 30 minutes.
- iii. The types of corrosion occurred on the surface of the aluminium alloys after immersion in sulphuric acid for 23 days was found to be uniform corrosion. This type of corrosion occurs due to the reaction between aluminium alloys and sulphuric acid.

5.4 RECOMMENDATIONS

In order to improve the investigation of the effect of heat treatment on the corrosion resistance of aluminium alloys for the future study, there is some aspects should be taken into consideration:

- i. The type of solution used during immersion test should be same as the real situation in the problems statement but it will take longer time to get the weight lost due to the characteristic of Al 5083 which is very excellent in the marine environment.
- ii. Other method should be used to measure the corrosion rate such as polarization method.
- iii. The weighing scale with high decimal should be used during taking the measurement of the specimen to get the accurate result of the weight loss during corrosion test. This experiment used only three decimal weighing scales which is no really sensitive with small changes.
- iv. Scanning Electron Microscope (SEM) and Energy Dispersive X-ray analysis (EDX) should be used together during microstructure analysis to get the better understanding of the effect of solution heat treatment process on microstructures changes and formed precipitates and corrosion product.

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APPENDIX A1

DATA OF EXPERIMENT

Table 6.1: Vickers hardness test (HV)

Specimens	Data										Average
1	44.6	45.0	48.3	45.6	47.6	47.3	47.6	46.7	44.7	48.0	46.5
2	35.4	37.4	34.1	33.1	32.5	35.8	34.1	37.2	35.3	34.1	34.9
3	37.1	38.0	39.8	37.6	36.5	35.9	37.8	37.0	39.1	37.5	37.6
4	35.7	37.5	34.8	35.6	34.0	37.6	34.7	35.4	36.6	35.0	35.7
5	36.6	37.4	40.6	40.9	35.7	38.4	36.6	37.5	39.1	41.3	38.4
6	38.3	35.7	40.6	38.7	37.4	35.1	41.4	37.2	39.3	38.0	38.2
7	42.0	41.9	41.0	40.2	40.1	39.7	40.3	39.0	40.4	37.5	40.2

Table 6.2: Dimension of the specimens

Specimens	Length (mm)	Width (mm)	Thickness (mm)	Total area (mm)
1	42.10	29.40	2.80	2888.053
2	41.75	30.70	2.90	2996.619
3	41.25	28.90	2.90	2804.079
4	38.95	30.80	2.95	2824.196
5	39.20	31.10	2.95	2866.361
6	39.20	31.10	2.95	2866.361
7	41.15	30.00	2.80	2879.613

Table 6.3: Weight before immersion test

Specimens	Weight 1 (g)	Weight 2 (g)	Weight 3 (g)	Average (g)
1	8.920	8.919	8.920	8.920
2	9.829	9.830	9.830	9.830
3	8.672	8.670	8.671	8.671
4	9.142	9.142	9.142	9.142
5	9.314	9.314	9.313	9.314
6	9.128	9.129	9.128	9.128
7	8.640	8.640	8.640	8.640

APPENDIX A2

DATA OF EXPERIMENT

Table 6.4: Weight after immersion test

Specimens	Weight 1 (g)	Weight 2 (g)	Weight 3 (g)	Average (g)
1	0.200	0.200	0.200	0.200
2	5.379	5.373	5.374	5.375
3	4.345	4.344	4.342	4.344
4	5.734	5.734	5.735	5.734
5	3.890	3.891	3.890	3.890
6	4.083	4.084	4.084	4.084
7	3.540	3.539	3.538	3.539

Table 6.5: Weight loss after immersion test

Specimens	Weight loss (g)
1	8.720
2	4.455
3	4.327
4	3.408
5	5.424
6	5.044
7	5.101

Table 6.6: Corrosion rate (mpy)

Specimens	Corrosion rate (mpy)
1	7.1211
2	3.5063
3	3.6394
4	2.8460
5	4.4630
6	4.1503
7	4.1779

APPENDIX B1

EXPERIMENT PROGRESS

Table 6.7: Experiment progress for solution heat treatment






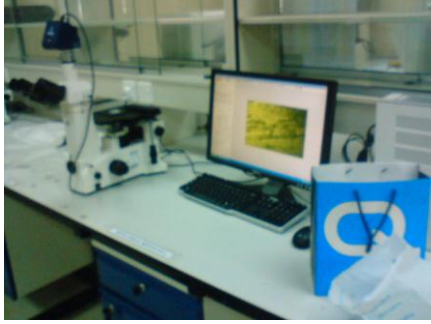
Figure	Description
	Aluminium sheet
	Cutting aluminium sheet
	Specimen after cutting process°C

Figure	Description
	Drilling process
	Grinding process
	Assembling process
	Solution heat treatment process

APPENDIX B2

EXPERIMENT PROGRESS



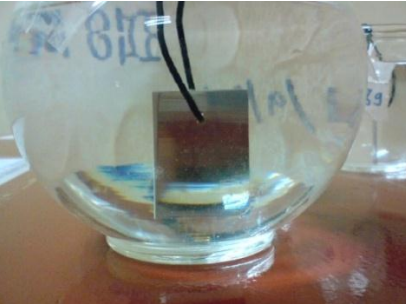

Table 6.8: Preparation for microstructures observation

Figure	Description
	Mounting process
	Polishing process
	Etching process
	Microstructure examination

APPENDIX B3

EXPERIMENT PROGRESS

Table 6.9: Experiment setup for immersion test

Figure	Description
	Specimen after polishing process
	Sulphuric acid 5% concentration
	Immersion test setup
	Immersion test

APPENDIX C1

GANTT CHART

ACTIVITIES		WEEKS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
RECEIVE TITLE FROM FACULTY	Planning	■														
	Actual	■														
PROBLEM STATEMENT	Planning		■	■	■											
	Actual			■	■	■										
LITERATURE REVIEW	Planning					■	■	■	■							
	Actual						■	■	■	■	■	■				
METHODOLOGY	Planning									■	■	■	■	■		
	Actual										■	■	■	■		
EXPERIMENT SETUP	Planning														■	■
	Actual															■
PRESENTATION	Planning															■
	Actual															■
COMPLETE DRAFT	Planning															■
	Actual															■

Figure 6.1: Gantt chart FYP 1

APPENDIX C2

GANTT CHART

ACTIVITIES		WEEKS																
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
EXPERIMENT SETUP	Planning	█	█	█														
	Actual		█	█	█													
EXPERIMENT PROCESS	Planning		█	█	█	█	█	█	█									
	Actual			█	█	█	█	█	█									
ANALYSIS	Planning				█	█	█	█	█									
	Actual				█	█	█	█	█	█	█							
DISCUSSION	Planning							█	█	█	█	█						
	Actual								█	█	█	█						
REPORT WRITING	Planning			█	█	█	█	█	█	█	█	█	█					
	Actual			█	█	█	█	█	█	█	█	█	█					
SUBMIT FIRST DRAFT	Planning													█				
	Actual													█				
CORRECTION ON FIRST DRAFT	Planning														█			
	Actual														█			
SUBMIT SECOND DRAFT	Planning															█		
	Actual															█		
PRESENTATION	Planning																	█
	Actual																	█
SUBMIT THESIS REPORT	Planning																	█
	Actual																	█

Figure 6.2: Gantt chart FYP 2