

**Microstructural Characterization of FSW and FSSW Aluminum  
Alloy Plates**

by

Nor Naqiah Bt Mohd Nazari@Anuar

Dissertation submitted in partial fulfillment of  
the requirements for the  
Bachelor of Engineering (Hons)  
(Mechanical Engineering)

MAY 2011

Universiti Teknologi PETRONAS,  
Bandar Seri Iskandar,  
31750 Tronoh,  
Perak Darul Ridzuan.

## CERTIFICATION OF APPROVAL

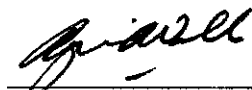
### MICROSTRUCTURAL CHARACTERIZATION OF FSW AND FSSW ALUMINUM ALLOY PLATES

by

Nor Naqiah Bt Mohd Nazari@Anuar

A project dissertation submitted to the  
Mechanical Engineering Programme  
Universiti Teknologi PETRONAS  
in partial fulfillment of the requirement for the  
BACHELOR OF ENGINEERING (Hons)  
(MECHANICAL ENGINEERING)

Approved by,



(Dr Azmi Bin Abdul Wahab)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2011

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



---

Nor Naqiah Bt Mohd Nazari@Anuar

## ABSTRACT

Friction Stir Welding (FSW) and Friction Stir Spot Welding (FSSW) are new techniques of welding. Since the process of friction stir welding (FSW) differs from friction stir spot welding (FSSW), the appearance and characteristics of the microstructure would be probably different. Therefore, the differences need to be characterized and documented. The main objective of the project is to characterize the microstructure of friction stir spot welding (FSSW) aluminum alloy and friction spot welding (FSW) aluminum alloy using Optical Microscopy (OM), Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM). This work focused on the weldment area of the welded plates. Both FSW and FSSW samples were prepared using the optimum parameters prior to undergoing metallography process. Both the friction stir welding (FSW) and friction stir spot welding (FSSW) are found to produce microstructural variations across the weld, due to the thermomechanical treatment involved in the process. Eventually, both samples are undergone Energy Dispersive X-ray Spectroscopy (EDS) to identify elemental composition of both sample.

## ACKNOWLEDGEMENT

*Alhamdulillah*, all praise to Allah and thank to Him, eventually my final report was finally completed and finished successfully on time as the final task for my Final Year Project.

I would like to record indebtedness to my supervisor, Dr Azmi Bin Abdul Wahab (Senior Lecturer of Universiti Teknologi PETRONAS). His tireless efforts and on-going support, advice as well as guidance are appreciated. Thanks also to Mr. Aiman and Mr. Umar Patthi (Post Graduate student) who delivered the best effort and co-operations in accomplishing my project task. Without their help, my project would not have been completed successfully. My appreciation is extended to mechanical technicians especially Mr. Anuar, Mr. Faisal, Mr. Irwan and Mr. Shahrul for their assistance and help in completing the experimental work.

I wish to acknowledge gratefully the help of all friends and staffs who contribute by offering needed tips, advices and endless cooperation. Though too numerous to mention individually, all have contributed toward the successful implementation of this final report. I offer my sincere apologies to anyone who may have been unintentionally excluded. Last but not least, thanks to my beloved family for their moral support and help throughout this project.

I hope that this report will give the readers some useful information associated with microstructural characterization of FSW and FSSW aluminum alloy plates.

## TABLE OF CONTENTS

<b>CERTIFICATION OF APPROVAL</b>									i
<b>CERTIFICATION OF ORIGINALITY</b>									ii
<b>ABSTRACT</b>									iii
<b>ACKNOWLEDGEMENT</b>									iv
<b>CHAPTER 1:</b>	<b>INTRODUCTION</b>								1
	1.1	Background of Study							1
	1.2	Problem Statement							2
	1.3	Objectives							2
	1.4	Scope of Study							2
<b>CHAPTER 2:</b>	<b>LITERATURE REVIEW</b>								3
	2.1	Aluminum Alloy							3
	2.2	Friction Stir Welding (FSW)							4
	2.3	Friction Stir Spot Welding (FSSW)							5
	2.4	Metallography							6
	2.5	Optical Microscopy (OM)							6
	2.6	Scanning Electron Microscopy (SEM)							7
	2.7	Field Emission SEM (FESEM)							8
	2.8	Energy-dispersive Spectroscopy (EDS)							9
<b>CHAPTER 3:</b>	<b>METHODOLOGY</b>								11
	3.1	Project Flow							11
	3.2	Sample Preparation							12
	3.3	Metallography							17
	3.4	Gantt Chart							21
<b>CHAPTER 4:</b>	<b>RESULTS AND DISCUSSION</b>								22
	4.1	Friction Stir Welding Micrographs							22
	4.2	Friction Stir Spot Welding Micrographs							31

<b>CHAPTER 5:</b>	<b>CONCLUSION AND RECOMMENDATION</b>	39
	5.1 Conclusion . . . . .	39
	5.2 Recommendations . . . . .	39
<b>REFERENCES</b>	. . . . .	40
<b>APPENDICES</b>	. . . . .	42

## LIST OF FIGURES

Figure 2.1: Schematic illustration of the friction stir welding process	-4-
Figure 2.2: Schematic illustrations of friction stir spot welding process	-5-
Figure 2.3: Principle of optical microscope	-6-
Figure 2.4: Difference in result between OM and SEM	-7-
Figure 2.5: Principle of FESEM	-8-
Figure 2.6: EDS system	-10-
Figure 3.1: Experimental work	-11-
Figure 3.2: Area of interest of FSW sample	-12-
Figure 3.3: Area of interest of FSSW sample	-13-
Figure 3.4: Automatic mounting machine	-13-
Figure 3.5: Grinding machine	-14-
Figure 3.6: Semi-automated polishing machine	-15-
Figure 3.7: Immersion of sample in the etchant	-16-
Figure 3.8: Optical microscope	-17-
Figure 3.9: Scanning Electron Microscope	-18-
Figure 3.10: FESEM machine	-19-
Figure 4.1: Schematic cross-section of FSW weld	-22-
Figure 4.2: OM Micrographs of FSW at three different spots	-23-
Figure 4.3: TMAZ and HAZ zones	-24-
Figure 4.4: Microstructure of (a) weld nugget, (b) base metal	-25-
Figure 4.5: Three different sections of FSW sample	-26-
Figure 4.6: The SEM micrograph at 3 different sections of FSW sample	-27-
Figure 4.7: FESEM micrograph at a and b sections of FSW sample	-28-
Figure 4.8: FESEM micrograph section c of FSW sample	-29-
Figure 4.9: EDS spectrum of FSW sample	-29-
Figure 4.10: Schematic cross-section of FSSW weld	-29-
Figure 4.11: OM Micrographs of FSSW at 3 different spots	-31-
Figure 4.12: Base metal of two plates	-32-
Figure 4.13: Lap joint microstructure	-33-
Figure 4.14: Microstructure of (a) weld nugget, (b)TMAZ, (c) HAZ	-34-
Figure 4.15: Three different sections of FSSW sample	-35-
Figure 4.16: SEM micrograph of FSSW sample	-35-



Figure 4.17: FESEM micrograph at 2 different sections of FSSW sample	-36-
Figure 4.18: EDS spectrum of FSSW sample	-37-

## **LIST OF TABLES**

Table 1: Final Year Project Semester 2 (2011) Gantt Chart	-21-
Table 2: Chemical composition of FSW sample	-30-
Table 3: Grain size of FSW sample	-30-
Table 4: Chemical composition of FSSW sample	-37-
Table 5: Grain size of FSSW sample	-38-

## **LIST OF ABBREVIATIONS**

FSW	Friction Stir Welding
FSSW	Friction Stir Spot Welding
Al	Aluminium
HAZ	Heat Affected Zone
TMAZ	Thermo-mechanically Affected Zone
OM	Optical Microscopy
SEM	Scanning Electron Microscope
FESEM	Field Emission Scanning Electron Microscopy
EDS	Energy dispersive X-ray Spectroscopy
SiC	Silica Carbide
HF	Hydrogen fluoride
HCl	Hydrochloric acid
HNO <sub>3</sub>	Nitric acid
H <sub>2</sub> O	Water
EDX	Engineering Design Exhibition
BM	Base Metal
Mg	Magnesium
Si	Silicon
C	Carbon

# CHAPTER 1

## INTRODUCTION

### 1.1 BACKGROUND OF STUDY

Friction Stir Welding (FSW) and Friction Stir Spot Welding (FSSW) are new techniques of welding. The techniques can produce joints utilizing equipment based on traditional machine tool technologies, and it has been used to weld a variety of similar and dissimilar alloys. Replacement of fastened joints with friction stir welded joints can lead to significant weight and cost savings, which would be an attractive propositions for many industries. <sup>[3]</sup> The welding process can be used to produce butt, corner, lap, T, spot, fillet and hem joints, as well as to weld hollow objects, such as tanks and tubes or pipes, stock with different thicknesses, tapered sections and parts with 3-dimensional contours. <sup>[2]</sup>

These types of welding have been used to weld all wrought aluminum alloys, and some of which are bordering on being classed as virtually unweldable by fusion welding techniques. Aluminum alloy (Al 6061) is widely used in numerous engineering applications including transport and construction where superior mechanical properties such as tensile strength, hardness are essentially required <sup>[1]</sup>. When using the conventional arc welding techniques, long butt or lap joints between Al 6061 and other aluminum alloys are particularly difficult to make without distortion because of high thermal conductivity and special welding procedures and high levels of welder skill are generally required. <sup>[3]</sup>

The welds are created by the combined action of frictional heating and mechanical deformation due to a rotating tool. <sup>[4]</sup> FSW and FSSW on aluminum alloys display several joints usually consist of four different regions. They are: (a) unaffected base metal, (b) heat affected zone (HAZ), (c) thermo-mechanically affected zone (TMAZ) and (d) stirred zone or weld nugget. <sup>[2]</sup> The formation of above regions is affected by the material flow behavior under the action of rotating non-consumable tool.

## **1.2 PROBLEM STATEMENT**

The joining of friction stir welding (FSW) and friction stir spot welding (FSSW) are butt joint and lap joint respectively. Since the welding process of friction stir welding (FSW) is differs from friction stir spot welding (FSSW), the behavior of the microstructure will be probably different from each other. Thus, the differences need to be identified. The previous studies mostly have been done on the characterization of friction stir welding only. For this project, characterization friction stir spot welding (FSSW) process is being included. The microstructures of the aluminum alloy of both samples were compared to the previous studies' results for a confirmation.

## **1.3 OBJECTIVES**

The objective of this project is to characterize the microstructure of friction stir spot welding (FSSW) aluminum alloy and friction spot welding (FSW) aluminum alloy using Optical Microscopy (OM), Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM).

## **1.4 SCOPE OF STUDY**

The project will be focusing on the microstructure of the aluminum alloy when it is subjected to two different welding process which are friction stir welding and friction stir spot welding. The project was focusing on the weldment area of the weld plates. Specifically, the microstructure studies aimed at characterization of four different regions, which are unaffected base metal, heat affected zone (HAZ), thermo-mechanically affected zone (TMAZ) and stirred zone or weld nugget.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 ALUMINUM ALLOY**

Aluminum's high usage in engineering is based on characteristics such as light weight, nonrusting properties, reasonably good strength and ductility, easy fabrication, modern metallurgical control of structure and properties, and favorable economics. [6] Aluminum and its alloy are characterized by a relatively low density. Some of alloys are easily to be formed by virtue of high ductility, which this is evidenced by the thin aluminum foil sheet into which the relatively pure material be rolled. [17]

Generally, aluminum alloy are divided into some of categories such as wrought and casting alloy. The major alloying elements used in aluminum are silicon, magnesium and copper. [6] Aluminum alloy is easily welded and joined by various commercial methods. Wrought aluminum alloys (used for rolled sheet or shapes, forgings or extrusions) have standard alloy composition and temper designation systems. These alloys exhibit good weldability when the magnesium content is below 5%. [6]

For aluminum-based alloys, it is possible to make joints where the strength of the weld is comparable to that of the base metal alloy friction stir spot welding is better compared to the conventional fusion welding processes. Aluminum alloys are difficult to be fusion-welded due to the requirements of (i) gas shielding of weld pool, and (ii) removal of oxide layers prior to ordering the welding process. In friction stir spot welding melting is avoided, the energy input used for friction stir spot welding is considerably low. Consequently, the Heat Affected Zone (HAZ) and residual stresses associated with the welds can be relatively small.

## 2.2 FRICTION STIR WELDING (FSW)

Friction stir welding (FSW) is a new technique for joining aluminum alloys. Invented in 1991 at The Welding Institute <sup>[9]</sup>, this technique results in low distortion and high joint strength compared with other techniques, and is capable of joining all aluminum alloys. <sup>[8]</sup> Friction stir welding (FSW) is a solid-state, hot-shear joining process in which a rotating tool with a shoulder and terminating in a threaded pin moves along the butting surfaces of two rigidly clamped plates placed on a backing plate as shown in Figure 1. <sup>[10]</sup>

Frictional heat, generated mostly under the tool's shoulder, softens the material. The shoulder also acts to contain the softened material which is forced to the back of the tool, in the process becoming consolidated to form a solid phase weld. Providing the components are adequately restrained, a high quality solid phase weld is formed following considerable hot working of the material at the joint. <sup>[11]</sup> The process is shown as the Figure 2.1 below:

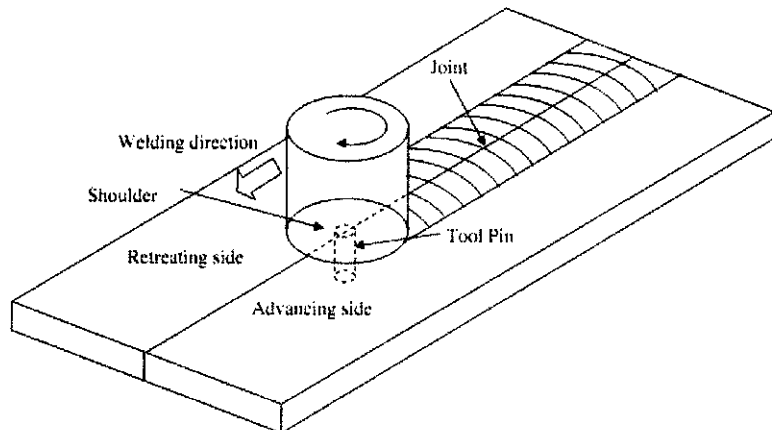


Figure 2.1: Schematic illustration of the friction stir welding process<sup>[10]</sup>

The solid-phase weld formation produced by FSW provides three important metallurgical advantages when compared to fusion welds in aluminum alloys; first, joining in the solid-phase eliminates cracking; second, there is no loss of alloying elements through weld metal evaporation and the alloy composition is preserved; and finally, the crushing, stirring and forging action of the welding tool produces a weld metal with a finer grain structure than that of parent metal. <sup>[11]</sup>

### 2.3 FRICTION STIR SPOT WELDING (FSSW)

Friction stir spot welding (FSSW) is a process variant of friction stir welding, which produces joints by plunging a rotating tool comprising a pin and a shoulder into two sheets and then retracting the tool leaving a keyhole depression. A rotating tool with a probe pin plunges into the upper sheet and a backing tool beneath the lower sheet supports the downward force. The downward force and the rotational speed are maintained for an appropriate time to generate frictional heat. Then, heated and softened material adjacent to the tool deforms plastically, and a solid-state bond is made between the surfaces of the upper and lower sheets. [5]

The FSSW process is shown in the Figure 2.2. The process of tool penetration essentially determines heat generation, plasticized material formation pin, joint formation and weld mechanical properties, since the whole FSSW process typically lasts from two seconds to the maximum of five seconds. [7]

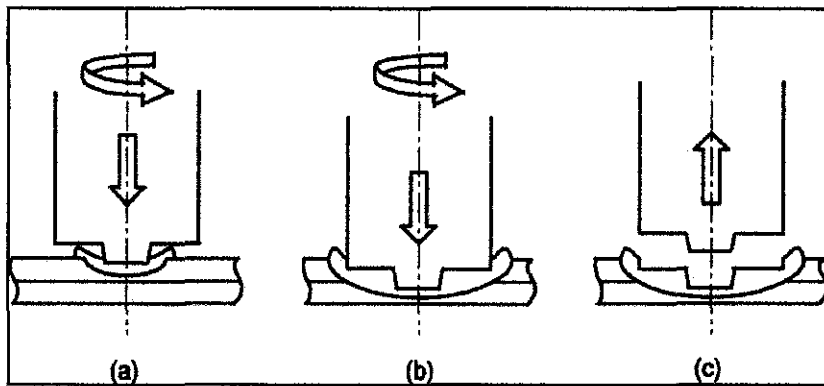


Figure 2.2: Schematic illustrations of friction stir spot welding process: (a) plunging; (b) bonding; (c) drawing out<sup>[7]</sup>

## **2.4 METALLOGRAPHY**

Metallography is the study of the structure of metals and alloys, particularly using microscopic (optical and electron) and X-ray diffraction techniques. <sup>[12]</sup> Metallography has been an invaluable tool for the advancement of science and industry for over one hundred years. Metallography is used to reveal the microstructure of metals, which is affected by alloy composition and processing conditions; including cold working, heat treatment and welding. <sup>[13]</sup>

## **2.5 OPTICAL MICROSCOPY (OM)**

Microscopic examination of polished or etched surfaces can reveal such information as size and shape of grains, distribution of structural phases and nonmetallic inclusions, micro-segregation, and other structural conditions. The magnifications of the microscope are usually ranging from about 100 to 1,500. Polarized light is useful to reveal grain structure, detect preferred orientation, examine oxide surface films, and identify phases of different composition.



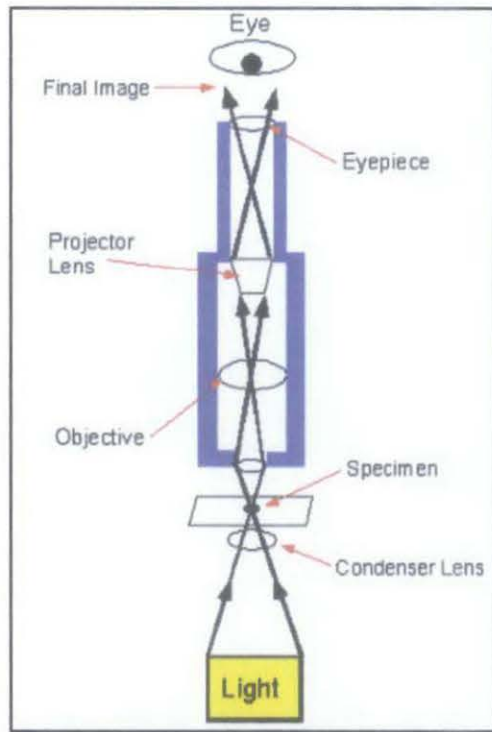


Figure 2.3: Principle of optical microscope <sup>[21]</sup>

From the Figure 2.3 above, it shows that the light passes through a prepared specimen on a slide and the image is magnified through a series of lenses. <sup>[21]</sup>

## 2.6 SCANNING ELECTRON MICROSCOPY (SEM)

The first scanning electron microscope (SEM) debuted in 1938 (Von Ardenne) with the first commercial instruments around 1965. <sup>[14]</sup> Its late development was due to the electronics involved in "scanning" the beam of electrons across the sample. Electron microscopes are scientific instruments that use a beam of energetic electrons to examine objects on a very fine scale. Electron microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light. <sup>[14]</sup> Figure 2.4 below shows the difference of micrograph produces by optical microscope and scanning electron microscope. The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample. <sup>[14]</sup>



Figure 2.4: Difference in result between Optical Microscope (left) and Scanning Electron Microscope (right)<sup>[12]</sup>(taken from Goldstein et al. 1992)

In electron microscopes a beam of electrons instead of a beam of light is directed onto the specimen; because only a highly energetic electron beam will pass through metal films thicker than about 0.05 micron (1 micron equals 0.001 millimeter), a microscope specimen replica of the surface is ordinarily made.<sup>[12]</sup> Backscattered electron images in the SEM display compositional contrast that results from different atomic number elements and their distribution. Energy Dispersive Spectroscopy (EDS) allows one to identify what those particular elements are and their relative proportions (Atomic % for example).<sup>[15]</sup>

## 2.7 FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)

FESEM is microscope that works with electrons (particles with a negative charge) instead of light. These electrons are liberated by a field emission source. The object is scanned by electrons according to a zig-zag pattern.<sup>[18]</sup> Electrons generated by a Field Emission Source are accelerated in a field gradient under vacuum condition. The beam focusing onto the specimen after passes through Electromagnetic Lenses. Different types of electrons are emitted from the specimen as result of this bombardment. A detector catches the secondary electrons and an image of the sample surface is constructed by comparing the intensity of these secondary electrons to the scanning primary electron beam. Finally the image is isplayed on a monitor. The process is shown as in the Figure 2.5 below.<sup>[22]</sup>

FESEM produces a cleaner image, less electrostatic distortions and spatial resolution lower than 2nm. That is 3 to 6 times better than conventional SEM. Smaller-area contamination spots can be examined at electron accelerating voltages compatible with Energy Dispersive X-ray Spectroscopy. Reduced penetration of low kinetic energy electrons probes closer to the immediate material surface. High quality, low voltage images are obtained with negligible electrical charging of samples.<sup>[18]</sup>

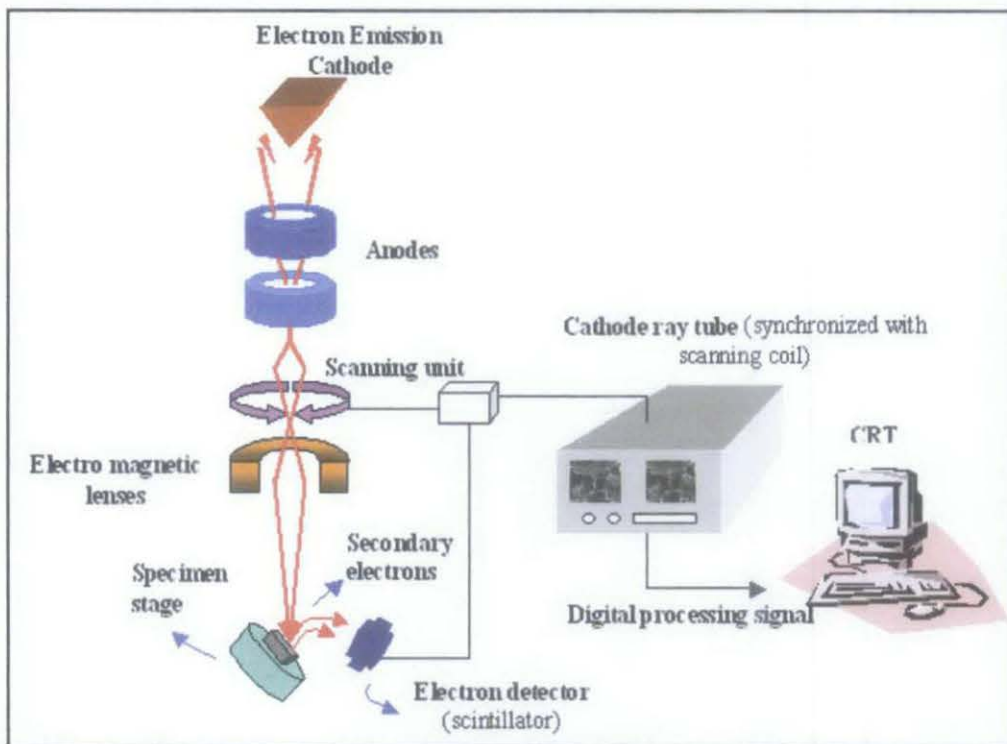


Figure 2.5: Principle of FESEM [22]

## 2.8 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDS)

Energy dispersive X-ray Spectroscopy (EDS) is an analytical technique that qualitatively and quantitatively identifies the elemental composition of materials analyzed in SEM.<sup>[19]</sup> Qualitative analysis is the process of identifying which elements are present in a sample. Quantitative analysis seeks to establish not only the identities of the elements present in a sample, but also their concentrations, together with an indication of the confidence that can be placed in the computed results.<sup>[20]</sup>

When the electron beam of the SEM is scanned across the sample, it generates x-rays from the atom in the top two microns. The energy of each x-ray is characteristic of the atom from which is escaped. The EDS system collects the x-rays, sorts them by energy and displays the number of x-rays versus their energy. This qualitative EDS spectrum can be either photographed or plotted.<sup>[19]</sup>

EDS system in Figure 2.6 above shown the array of components from detector to multichannel analyzer that assembles the information contained in the x-ray signals into a convenient x-ray spectrum.<sup>[27]</sup> There is sample constraint whereby the sample can be up to 15cm x 10cm x 75cm in size. The sample must be compatible with a  $10^{-6}$  torr vacuum, for example non-volatile and not susceptible to electron beam induced damage.<sup>[19]</sup>

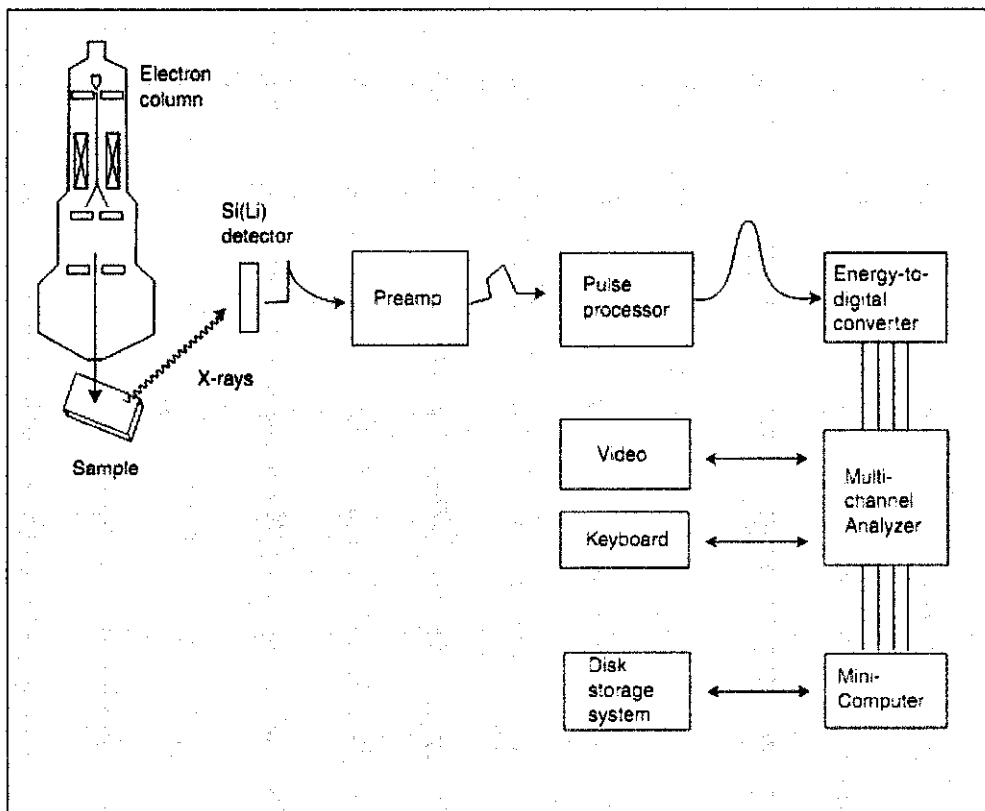


Figure 2.6:EDS system<sup>[20]</sup>



## CHAPTER 3 METHODOLOGY

### 3.1 PROJECT FLOW

The project is being divided into two phases. The first phase of the project is research work on welding process, the microstructure of aluminum and the metallography process. All information about the materials, equipment and experimental procedure were gathered in the first semester of the final year project. The second phase of the project is experimental work. It is divided into two parts, which are sample preparation and metallography process. All the experimental work is done in Block 17 of Mechanical Engineering Building with the assistance of mechanical technicians. The flow of the experimental work is as the figure below:

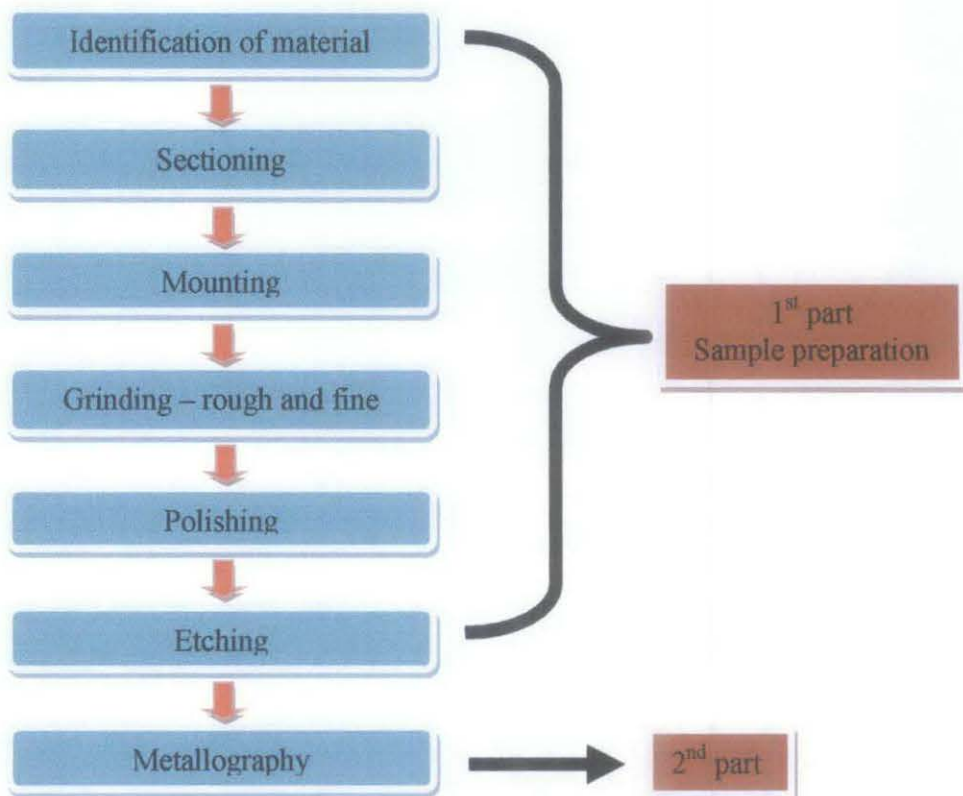


Figure 3.1: Experimental work

## 3.2 SAMPLE PREPARATION

Metallographic sample preparation is the crucial part before doing other tasks on the samples. To achieve true representation of the materials' microstructure, preparation must be carried out accurately with a clear understanding of what must be accomplished during each stage<sup>[13]</sup>. It is the key to obtain an accurate interpretation of a microstructure as it will be representative of the material being examined. The preparation method included steps listed as follows<sup>[13]</sup>:

### 3.2.1 Sectioning

Sectioning is the first step of sample preparation whereby it is a process of removing the representative sample from the parent piece. Abrasive cutting offers the best solution to eliminate or minimize heat and deformation. The materials were sectioned based on the direction of welding. Both figures below show the area of interest of FSW and FSSW respectively.

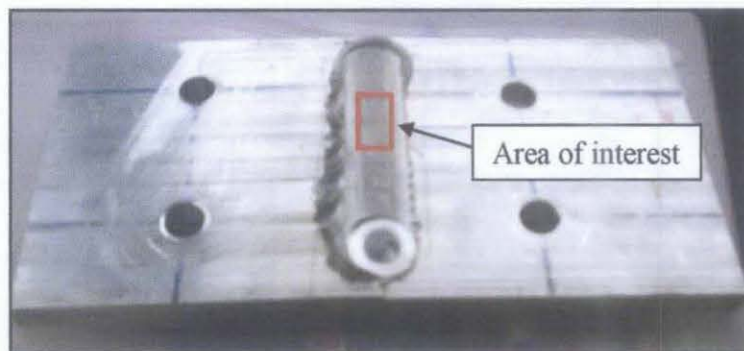


Figure 3.2: Area of interest of FSW sample

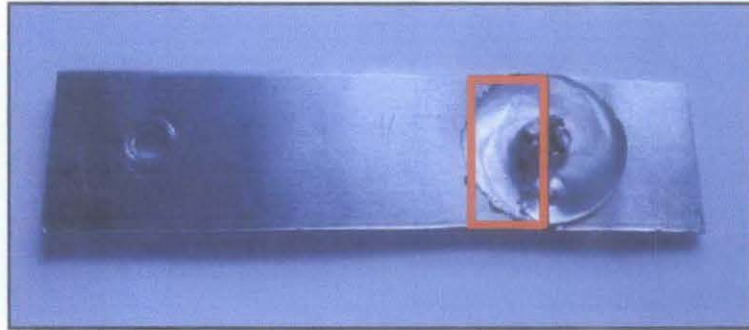


Figure 3.3: Area of interest of FSSW sample

### 3.2.2 Mounting

Mounting is a process of embedding the sample in plastic medium for ease in manipulation and to prevent fragility, edge preservation or edge retention. Hot compression mounting process had been used. It can preserve edge and minimize the shrinkage. Mounting medium that was used in this project is diallyl phthalate mineral blue in powder form. Thermosetting material had been used to mount the sample with the molding temperature and molding pressure of 300-360 °F and 3800-4400 psi respectively.



Figure 3.4: Automatic mounting machine

The mounting medium is being put inside the automatic mounting machine as in the Figure 3 above. A heat time of 3 minutes, cool time of 5 minutes and pressure of 4000psi were used for both samples. The medium was being pre-heated first before starting the heating process. It will heat the mounting medium for 3 minutes, followed by cooling process. Releasing agent is one of the important elements that need to be applied to the upper part of mold before start mounting as it is used for mold release.

### 3.2.3 Grinding

There are two types of grinding process, which are coarse grinding and fine grinding. Coarse grinding is a process to produce an initial flat surface. Fine grinding is a process to remove the zone of deformation caused by sectioning and coarse grinding and limit the depths of deformation during this stage by proper abrasive size sequencing.



Figure 3.5: Grinding machine

Mechanical grinding was performed in successive steps using SiC abrasive papers of different grit sizes, usually 180, 220, 320, 600, 800, 1000, 1200, 2400 grit. The starting grit depends on the type of cut surface to be removed. A wet grinding (water) to flush away these particles and a small pressure on the specimen were recommended since the abrasive particles embedded easily into soft aluminum alloys. The sample was placed on the surface



of the grinding paper, and the grinding process continued until the surface of the samples is shining. During the grinding process, the handling the samples is important, if not, the surface will not be flat, and this will affect the image in the optical microscope.

### 3.2.4 Polishing

After both grinding process, polishing needed to be done. The recommended polishing procedure depended upon the hardness and ductility of the specimen. Polishing is used to remove the deformation zone produced by fine grinding. It is also used to produce a shining surface or scratch-free surface to ease the characterization process.

The polishing process is accomplished primarily with diamond abrasives ranging from 9 micron down to 0.25-micron diamond. Polycrystalline diamond because of its multiple and small cutting edges, produces high cut rates with minimal surface damage, therefore it is the recommended diamond abrasive for metallographic rough polishing on low napped polishing cloths.



Figure 3.6: Semi-automated polishing machine

The sample was polished using the semi-automated polishing machine as in the Figure 3.6 above. The polishing process was done for a longer time to get the best result.

### 3.2.5 Etching

Etching is defined as preferential attack of a metal surface with an acid or basic chemical solution to reveal structural details. It is to obtain sufficient contrast between phases. It is clear for example that the grain structure cannot be easily revealed in every alloy. The etching reagent for the aluminum alloy sample is Keller's reagent.

Keller's reagent consists of 2ml HF (48%), 3ml HCl, 5ml HNO<sub>3</sub> and 190ml H<sub>2</sub>O. Chemical etching selectively attacks specific microstructural features. It generally consists of a mixture of acids or bases with oxidizing or reducing agents. The proper protective garb such as glasses, gloves, and apron should be used when pouring, mixing, or etching. For weighing, mixing, containing, and storage of solutions proper devices had been used. The measurement of each the chemical need to be precisely done, because it will affect the reading of the microstructure on the surface.



Figure 3.7: Immersion of sample in the etchant (source: knoll website)

The sample was immersed in the composition for 10 to 20 seconds, and then rinsed in a stream of water. The quality of the polishing influences the development of the true microstructure. A faulty preparation can lead to misinterpretation of the structure.

### 3.3 METALLOGRAPHY

#### 3.3.1 Optical Microscopy (OM)

The samples were viewed via optical microscope. There are several important features of the optical microscope as in the Figure 3.8 below are lenses, eyepieces (oculars), light source and camera. These parts are essential in acquiring the accurate images of the samples. It consists of two lens systems (combination of lenses) to magnify the image. Each lens has a different magnifying power. Light source or a beam is being used for focusing on the images.



Figure 3.8: Optical microscope

The magnification employed in this analysis is ranging from 50 times to 1500 times. The samples were etched before viewing the microstructure of the aluminum. The focus lens was adjusted according to the image created in the computer. The picture is taken spot by spot, in order to make sure that the surface is fully viewed.

### 3.3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy analysis was applied after the optical microscopy analysis. The model for metallurgy optical microscope is Zeiss and the magnification employed is ranging from 100 times to 3000 times. Minimal preparation includes acquisition of a sample that will fit into the SEM chamber and the samples need to be coated before doing the analysis. The pictures were taken based on the three parts that I have divided before the experiment. The equipment of SEM is as shown in Figure 3.9 below.



Figure 3.9: Scanning Electron Microscope

In general, for the scanning electron microscope (SEM) the sample preparation is similar to the one for optical microscopy. The different results in observations are eventually attributed to the technique that is based on the interaction between electrons and metals and not on visible light.



### 3.3.3 Field Emission Scanning Electron Microscopy (FESEM)

In order to be observed with a SEM objects are first made conductive for current. This is done by coating them with an extremely thin layer (1.5 - 3.0 nm) of gold or gold-palladium. After the object has been covered by a conductive layer, it is mounted on a special holder. The magnifications for this process are ranging from 500 times to 3000 times.



Figure 3.10: FESEM machine

The object is inserted through an exchange chamber into the high vacuum part of the microscope and anchored on a moveable stage. In the virtual FESEM the object can be moved in horizontal and vertical direction on the screen by operating the arrows in the Position box. In the real microscope the object can be repositioned in the chamber by means of a joy stick that steers in left-right axis, or forward and backward. In addition, the object can be tilted (e.g. for stereo views), rotated and moved in Z direction (closer or further away to the objective lens).

### **3.3.4 Energy Dispersive X-ray Spectroscopy (EDS)**

A typical instrument is equipped with four computer control crystal spectrometers, containing a range of crystal such that the whole spectrum can be covered. An EDS system for preliminary qualitative analysis is fitted. An energy dispersive detector is mounted on to an existing scanning microscope since the instrument is ideally suited to an environment where accurate quantitative analysis is required from a large number of specimens on a routine basis. <sup>[23]</sup>

There are various methods in which qualitative analytical information obtained from suitable specimen can be presented and the method used is analysis entails obtaining data from a larger area of the specimen. <sup>[23]</sup>This only requires scanning the beam over a suitable area which might be 100 um x 100um, as the X-rays are detected.

Before start taking the readings, both specimen and standards must be flat polished with scratches less than 0.1 um. The x-ray spectrum of the specimen and standards containing the elements that have been identified in the specimen should be obtained. All measurements for a given element, in both the specimen and the standards, are being made at the same. <sup>[15]</sup>The images created then being analyzed based on the concentration of the element of interest.

### **3.4 GANTT CHART**

Below is the Gantt chart of this project for this semester. The important weeks that need to be alerted with are on week 11 until week 15. On these particular weeks, there were due date for submission of draft report, dissertation (softbound), technical paper and dissertation (hardbound). In week 11, there was pre-EDX. A thorough preparation need to be taken before the dateline.



## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 FRICTION STIR WELDING (FSW) MICROGRAPHS

##### 4.1.1 Optical Microscopy

The cross-section was hot-mounted, polished, and etched with the Keller's reagent for the optical microscopy (OM) at room temperature. The samples FSW and FSSW are being labeled as sample 1 and sample 2 respectively. The micrographs of the samples were taken separately for each region.

The examination of many FSW and FSSW joints in aluminum alloys has revealed that there are four major microstructural zones<sup>[20]</sup>, as indicated in Figure 4.1. The formation of the regions is affected by the material flow behavior under the action of rotating non-consumable tool.<sup>[21]</sup> The microstructure of the weld is complex and highly dependent on the position within the welded zone.<sup>[20]</sup>

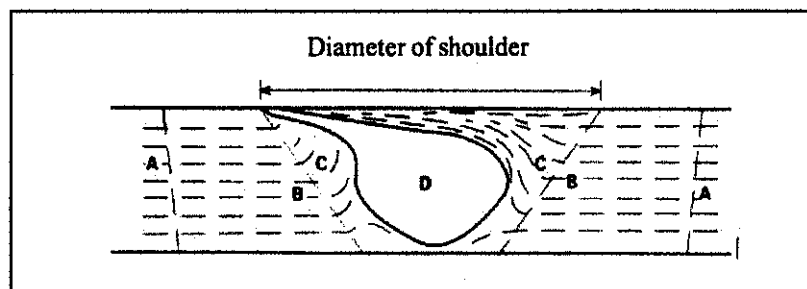


Figure 4.1: Schematic cross-section of FSW weld (A) - Base Metal (BM), (B) - Heat-affected Zone (HAZ), (C) - Thermo-mechanically Affected Zone (TMAZ) and (D) - stirred zone or weld nugget



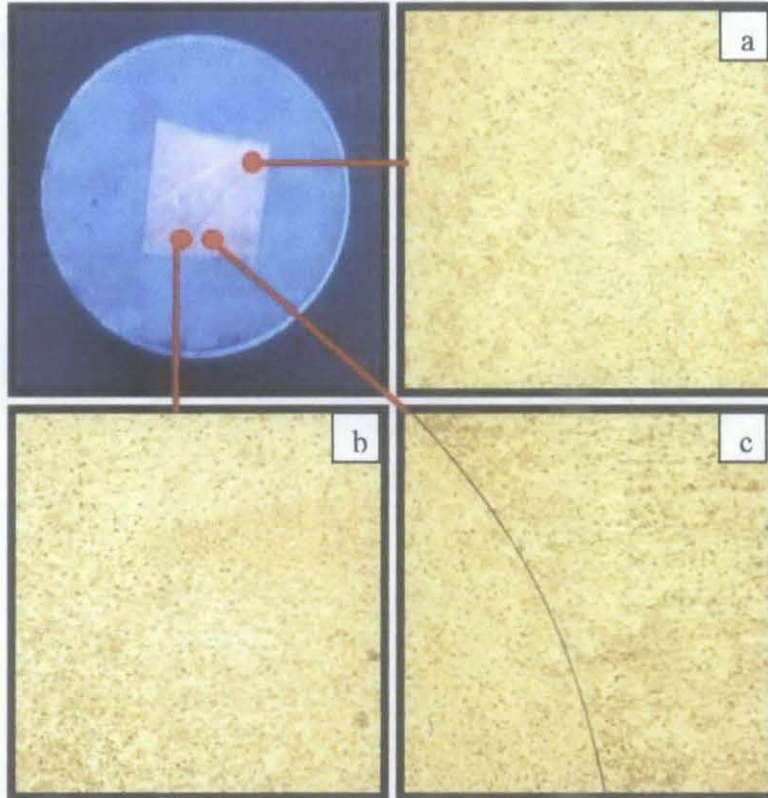


Figure 4.2: OM Micrographs of FSW at three different spots. The micrograph is at the magnification of 100 times.

Figure 4.2 above shows the micrographs of FSW sample at three different spots. They are (a) base metal (BM), (b) stirred zone and (c) heat affected zone (HAZ) and thermo-mechanically affected zone (TMAZ). These spots had been chosen based on the behavior of the microstructures. These microstructural zones will then be explained further.

From Figure 4.2(c) above the heat affected zone (HAZ) and thermo-mechanically affected zone can be distinguished into two distinct areas, as shown in Figure 4.3 below:

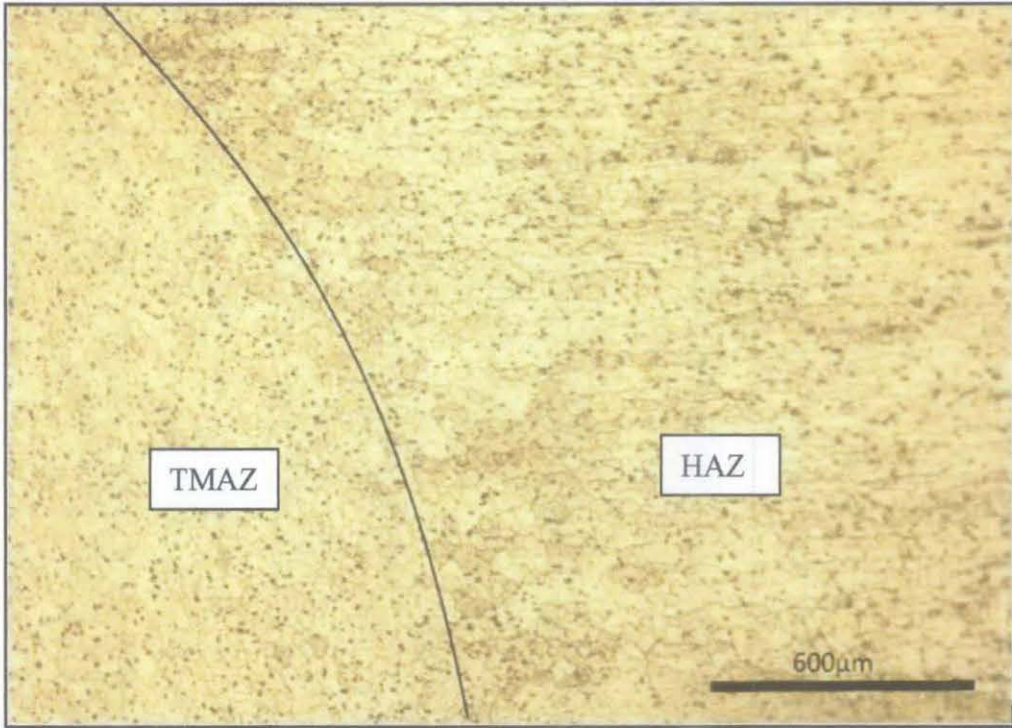


Figure 4.3: Thermo-mechanically Affected Zone (TMAZ) and Heat Affected Zone (HAZ) areas

The grains at Thermo-mechanically Affected Zone (TMAZ) were deformed into elongated and rotated grains due to the strain to which they were subjected during welding. The following zone is heat affected zone (HAZ) which is moving away in the direction of base material. <sup>[23]</sup>

The thermo-mechanically affected zone (TMAZ) is an area that has been plastically deformed by the friction stir welding tool, and the heat from the process will also have exerted some influence on the material. As aluminum behaves in a different manner to most other materials, it is possible to get significant plastic strain without recrystallization in this region, and there is a distinct boundary between the recrystallized zone and the deformed zones of the TMAZ. <sup>[20]</sup>

The heat affected zone (HAZ) lies further from the weld center. The material has experienced a thermal cycle, and modifications in mechanical properties and microstructure are noticed. However, no plastic deformation occurs in this zone. <sup>[20]</sup>



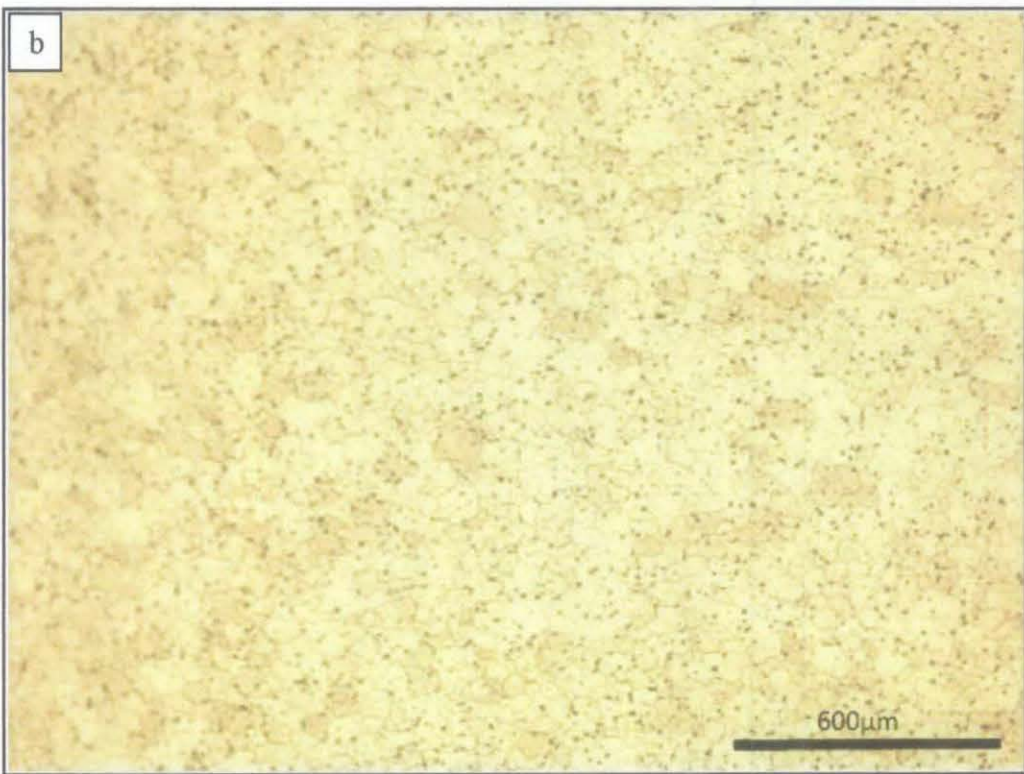
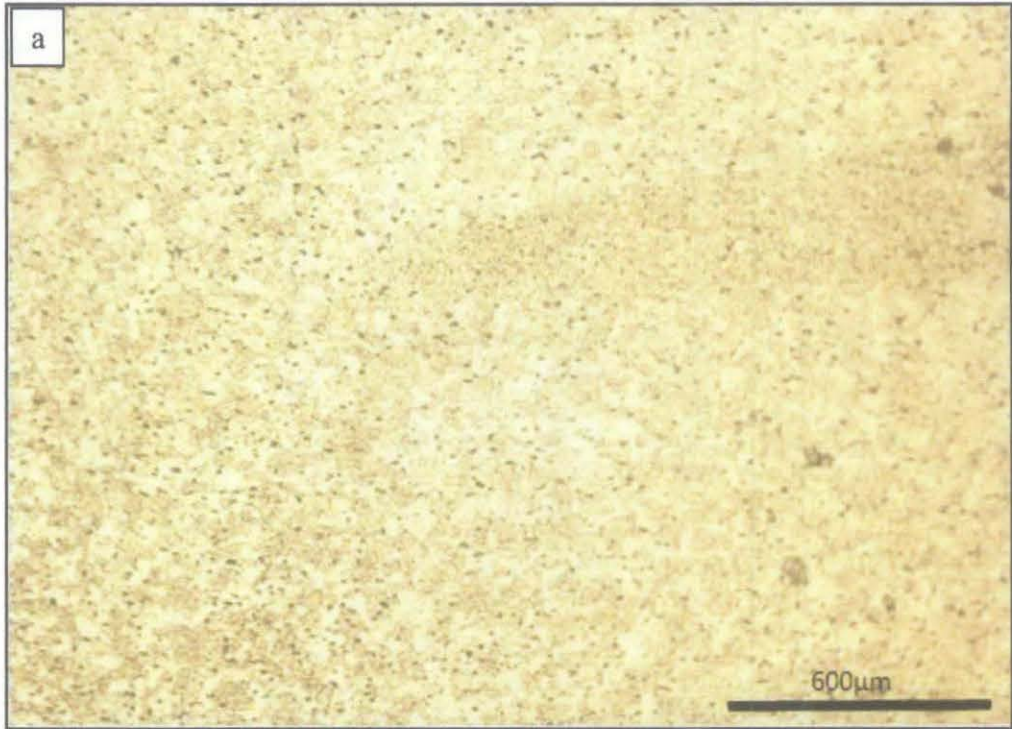


Figure 4.4: (a)Microstructure at weld nugget area, (b)Microstructure at base metal area

The fine grains are found in the weld nugget zone, in contrast to the base metal area where coarsened grains are observed. <sup>[20]</sup> Weld nugget region experiencing

intense plastic deformation and high-temperature exposure and characterized by fine and equiaxed recrystallized grains. It has been attributed to friction heating and plastic flow. The process also produced a softened region in the weld nugget which may be due to the dissolution and growth of possible precipitates. [22]

The parent material has not been deformed and may have experienced a thermal cycle from the weld but has not been affected by the heat in terms of microstructure or mechanical properties. [20]

The cross-section has the typical aspect of a friction stir-welded zone, whose most prominent feature is the presence of a weld nugget zone characterized by fine equiaxed recrystallised grains. [24] In TMAZ region, the material has been plastically deformed by the FSW tool, and the heat from the process will also have exerted some influence on the material. While in heat affected zone (HAZ) the material has experienced a thermal cycle which has modified the microstructure properties. However, there is no plastic deformation occurring in this area.

#### 4.1.2 Scanning Electron Microscopy (SEM)



Figure 4.5: Three different sections of FSW sample

The FSW sample was being divided into three sections for the ease of microscopy process. Figure 4.5 above shows the three different sections of the FSW sample. The pattern of the micrographs is differing to each other. It will be explained further based on the micrographs as in Figure 4.6 below.



Different patterns of the microstructures can be observed in the Figure 4.6 above. Figure 4.8a shows the microstructure is more elongated than the microstructure in Figure 4.6b and Figure 4.6c.

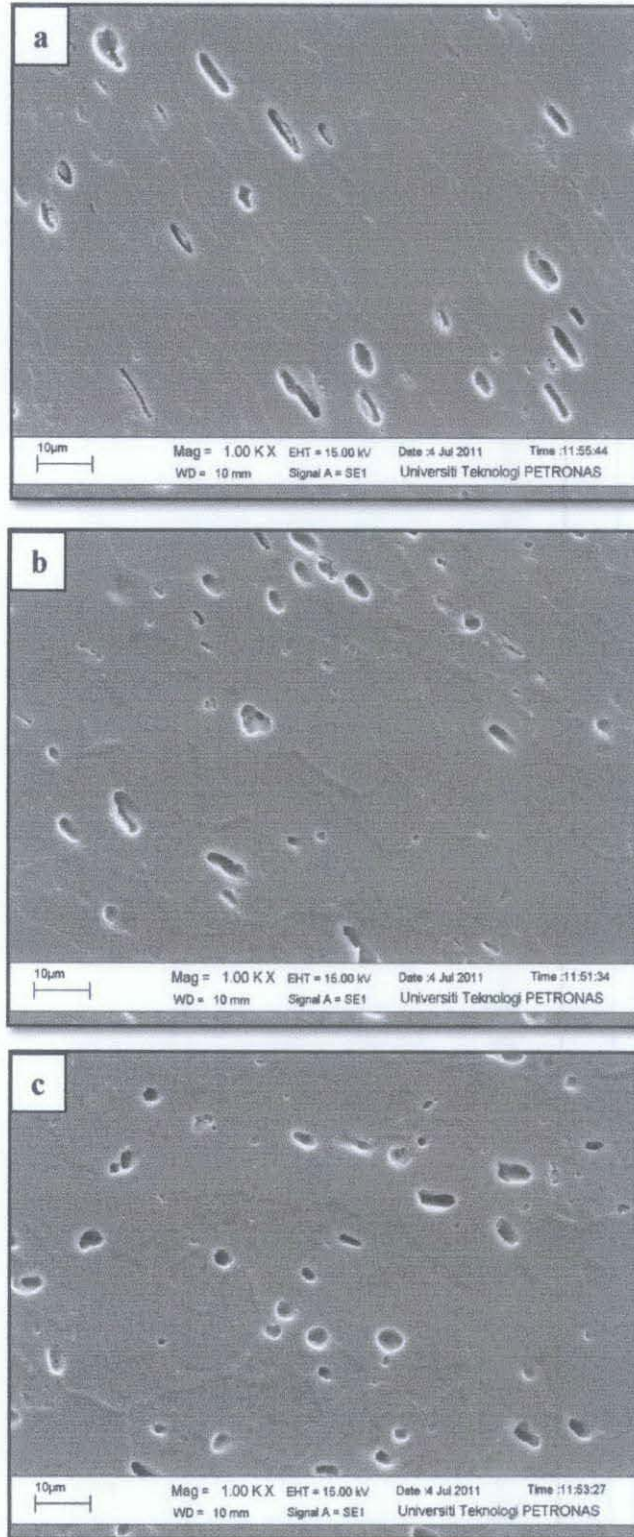


Figure 4.6: The SEM micrograph at three different sections of FSW sample

### 4.1.3 Field Emission Scanning Electron Microscopy (FESEM)

Figure 4.7 and Figure 4.8 below shows the micrograph at three different sections of FSW sample. The micrograph is being observed at the magnification of 500 times. The grain boundary at Figure 4.7a and Figure 4.7b is the same, but the latter is having more voids.

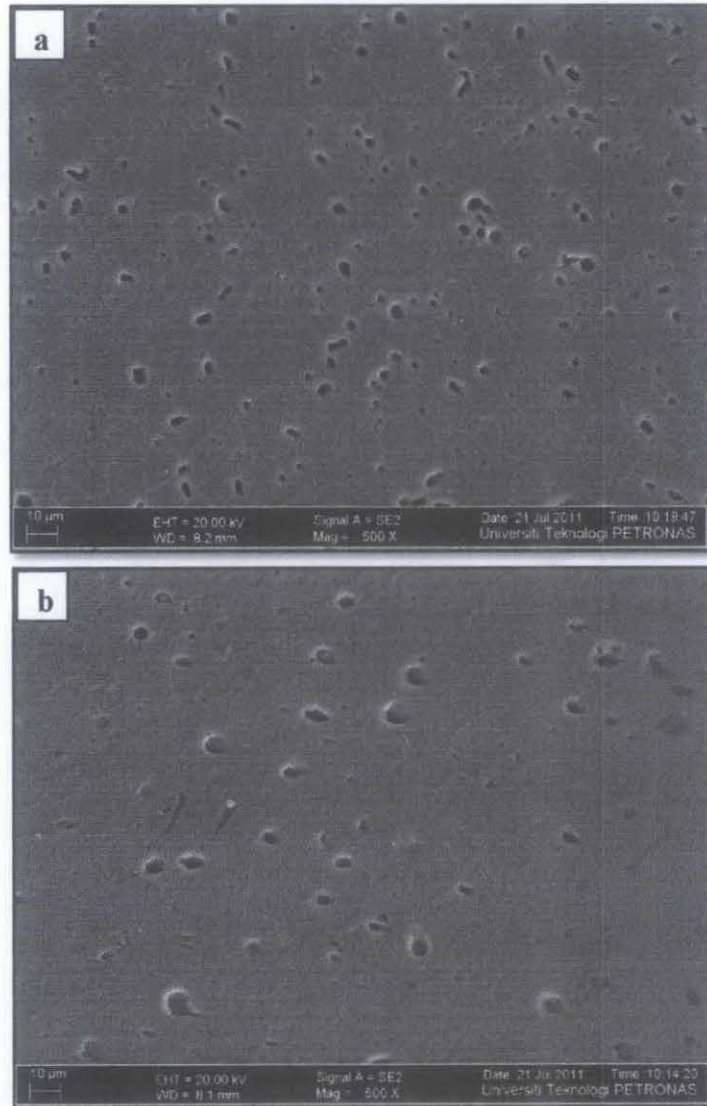


Figure 4.7: The FESEM micrograph at a and b sections of FSW sample

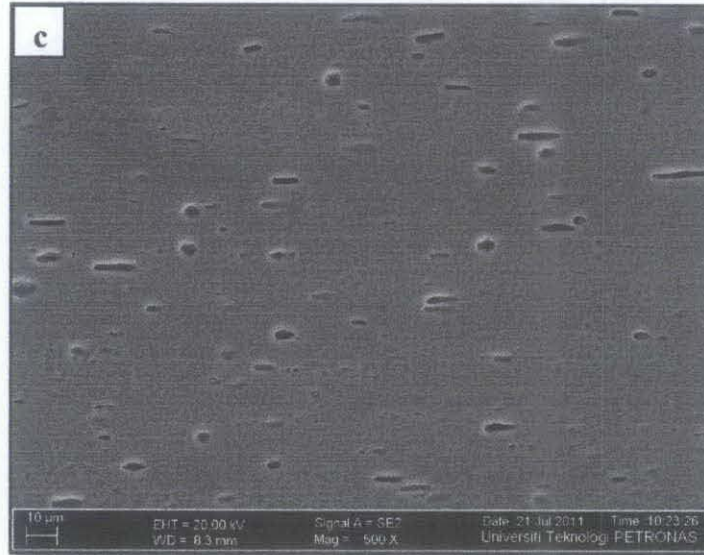


Figure 4.8: The FESEM micrograph section c of FSW sample

The Figure 4.8 above shows the FESEM micrograph at section c. the grain boundaries are more elongated than the grain boundaries in Figure 4.7a and Figure 4.7b. It is probably happened due to the heat created during the welding process.

#### 4.1.4 Energy Dispersive X-ray Spectroscopy (EDS)

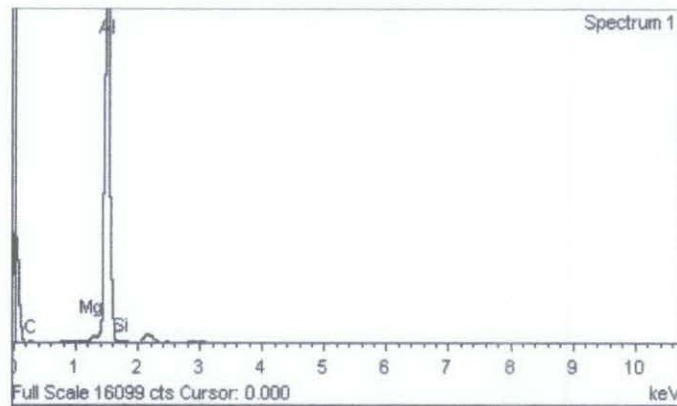


Figure 4.9: EDS spectrum of FSW sample

The EDS analysis of the chemical composition proved the FSW sample is composed of Mg, Al and Si within the band of the 6061 alloy. These phases are probably the typical ones for the alloys of 6xxxx series. It is used widely for hot extrusions for instance window or door frames.<sup>[25]</sup>



Table 2: Chemical composition of FSW sample

Element	Weight%	Atomic%
C K	17.41	32.12
Mg K	0.58	0.52
Al K	81.38	66.85
Si K	0.63	0.50
Totals	100.00	

#### 4.1.5 Grain Size Determination

A specimen must be properly prepared to reveal the grain structures, which is photographed at a magnification of 100X. The method used for estimating the grain size number is grain counting method, whereby the number of grains per unit area is counted directly. The average number of grains per square inch at a magnification of 100X is related to grain size number according to the equation below:

$$N' = 0.155 \times 10^{(0.301)(n-1)}$$

$N'$  represents the average number of grains per square centimeter at magnification of 100X and  $n$  the grain size number. Based on the equation above, these two parameters are related to each other. Calculation has been made based on the equation above.

The result is shown as in Table 2 below:

Table 3: Grain size of FSW sample

Regions of welding	Calculation of n
1. Base metal	$n = 1 + \frac{\log 10.5 - \log 0.155}{0.301} = 7.08$
2. TMAZ	$n = 1 + \frac{\log 14.5 - \log 0.155}{0.301} = 7.55$
3. HAZ	$n = 1 + \frac{\log 11.5 - \log 0.155}{0.301} = 7.21$
4. Weld nugget	$n = 1 + \frac{\log 16 - \log 0.155}{0.301} = 7.69$



## 4.2 FRICTION STIR SPOT WELDING (FSSW) MICROGRAPHY

### 4.2.1 Optical Microscopy (OM)



Figure 4.10: Schematic cross-section of FSSW weld <sup>[5]</sup>

The four different regions are still applicable in identifying the behavior of the microstructure although the joining for FSSW is lap joint as in Figure 4.10 above. The different regions of FSSW sample are presented as in Figure 4.11 below. Each of the spots were being chose based on the significant of microstructure behavior.

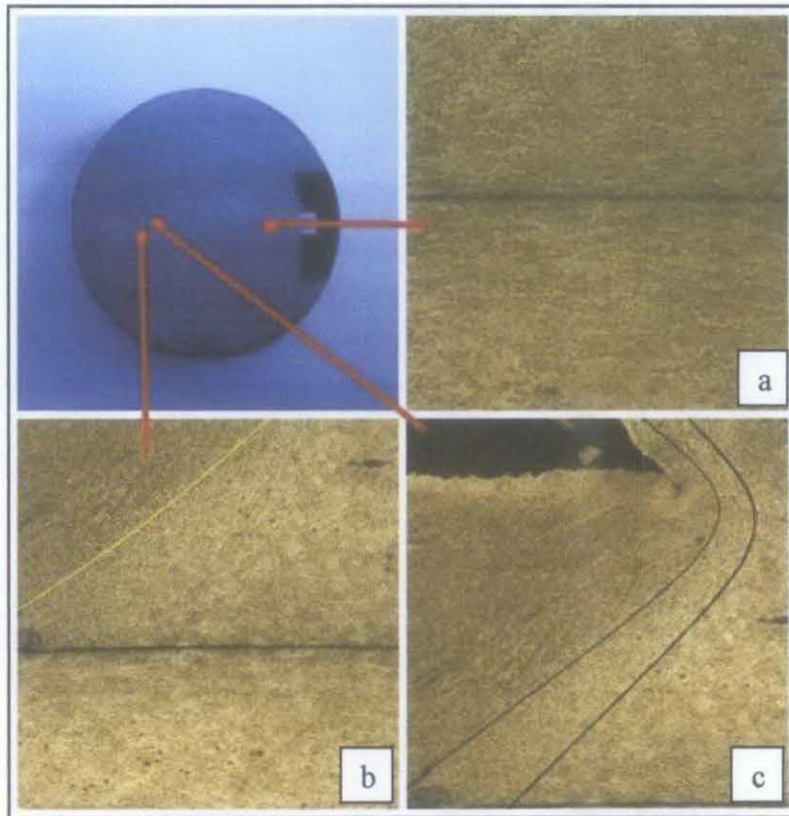


Figure 4.11: OM Micrographs of FSSW at 3 different spots

Each of the micrographs in the Figure 4.11 shows two or three different regions of weldment area. These differences will be explained further as in the figures below.

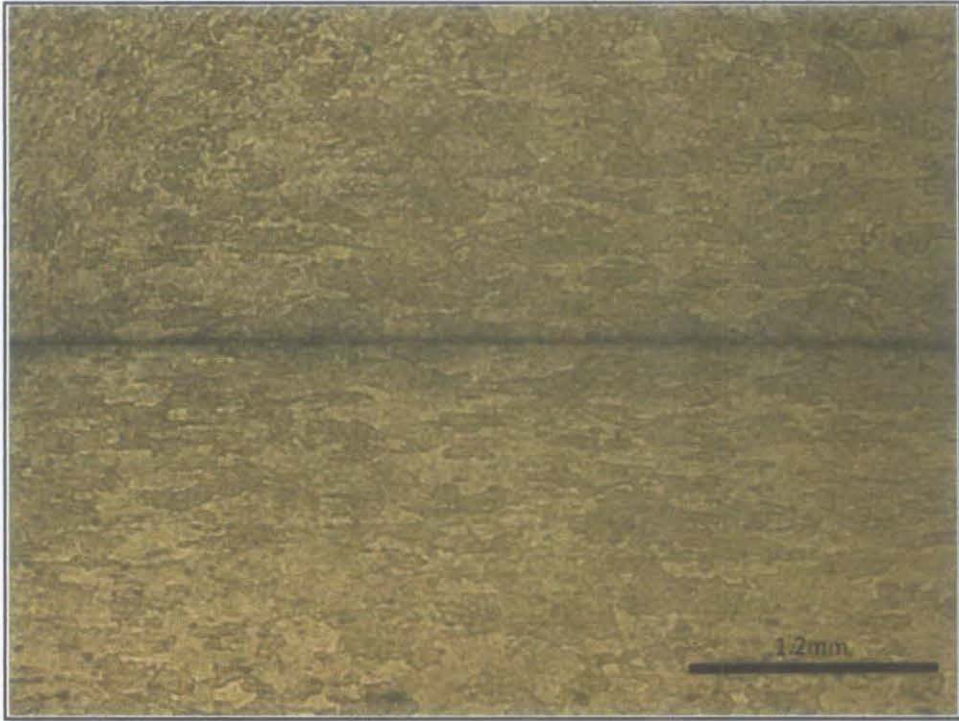


Figure 4.12: Base metal of two plates

Figure 4.12 above represents the base metal of two plates in FSSW. The microstructure at the base metal is unaffected from the welding process. This is material remote from the weld, which has not been deformed, and which although it may have experienced a thermal cycle from the weld is not affected by the heat in terms of microstructure or mechanical properties.

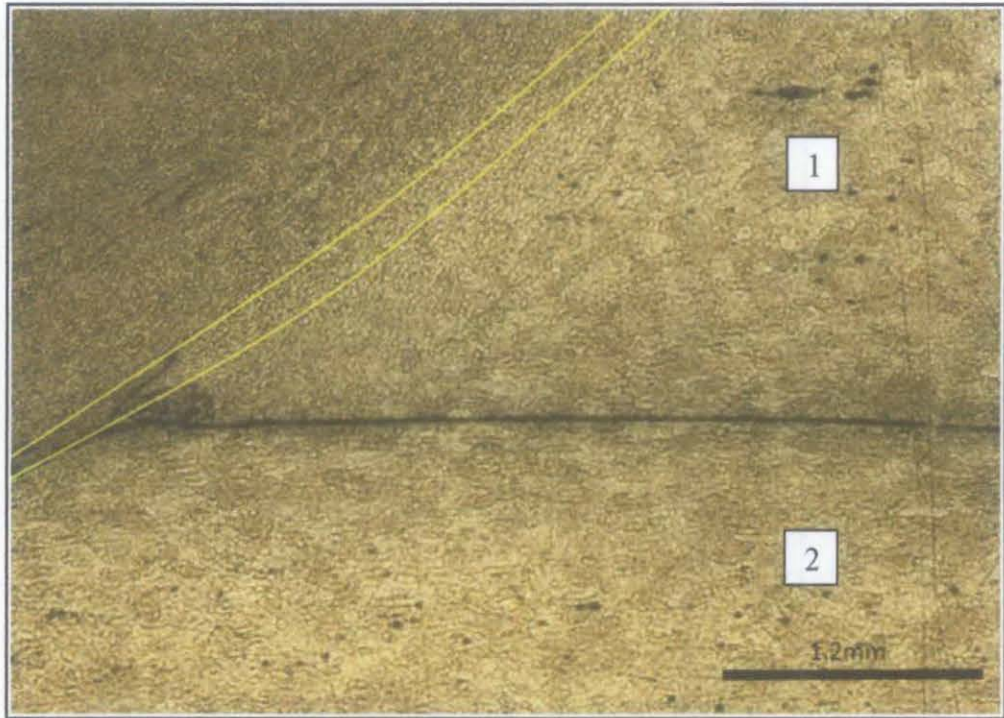


Figure 4.13: Lap joint microstructure

The line with the black color shows in the Figure 4.13 above shows that the plates are being lapped. Plate 1 is being lapped on the plate 2. The border line will disappear once they are introduced to welding process. It can be seen at Figure 4.13 the region which is below yellow line. The yellow line is being drawn to distinguish the area of thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ).



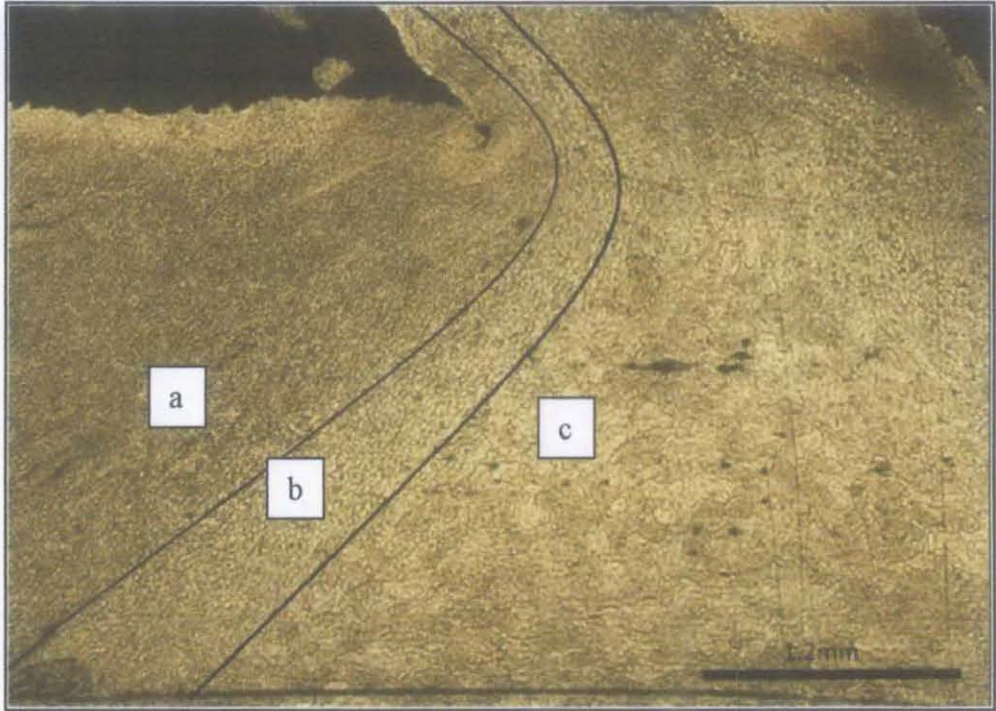


Figure 4.14: (a) weld nugget, (b) Thermo-mechanically Affected Zone (TMAZ) and (c) Heat Affected Zone (HAZ)

From the Figure 4.14 above, there are three distinct areas that can be distinguished. Two lines had been drawn in order to show the difference of the microstructure. The region of a, b and c shows the region of weld nugget, thermo-mechanically affected zone (TMAZ) and heat affected zone respectively.

#### 4.2.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) photographs are taken in 1000X scales from scanning electron microscope with video capture. The FSSW sample also was being divided into three sections for the ease of microscopy process. Figure 4.9 below shows the three different sections of the FSSW sample. The pattern of the micrographs is differing to each other. It will be explained further based on the micrographs as in Figure 4.8 below.

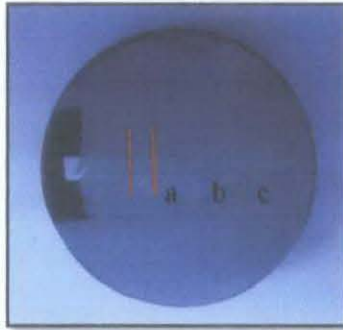


Figure 4.15: Three different sections of FSSW sample

From the SEM process, the micrographs at section a and c, were not showing any significant pattern. However at the b section, the microstructure had a pattern of dimple-like. It is probably due to the heat and friction of the welding process.

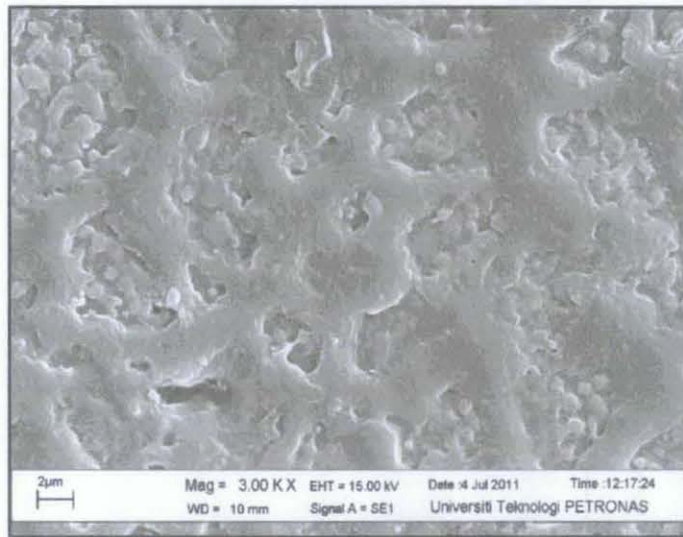


Figure 4.16: The SEM micrograph of FSSW sample

#### 4.2.3 Field Emission Scanning Electron Microscopy (FESEM)

The sample was being divided as in the Figure 4.9. The micrographs of FSSW at three sections are shown as in Figure 4.13 below. From Figure 4.13a is aiming to investigate the boundary between the upper and lower plates in detail. It shows the microstructure of two different plates were being lapped. The boundary is in zigzag manner can be seen clearly. The micrograph in Figure 4.13b is located at the weldment area. The microstructure shows the dimple-like pattern as in the Figure

4.10. It is possibly caused by the welding process. The micrograph at c-region was not showing any significant microstructure because of the coating problem. Consequently, the microstructure cannot be defined.

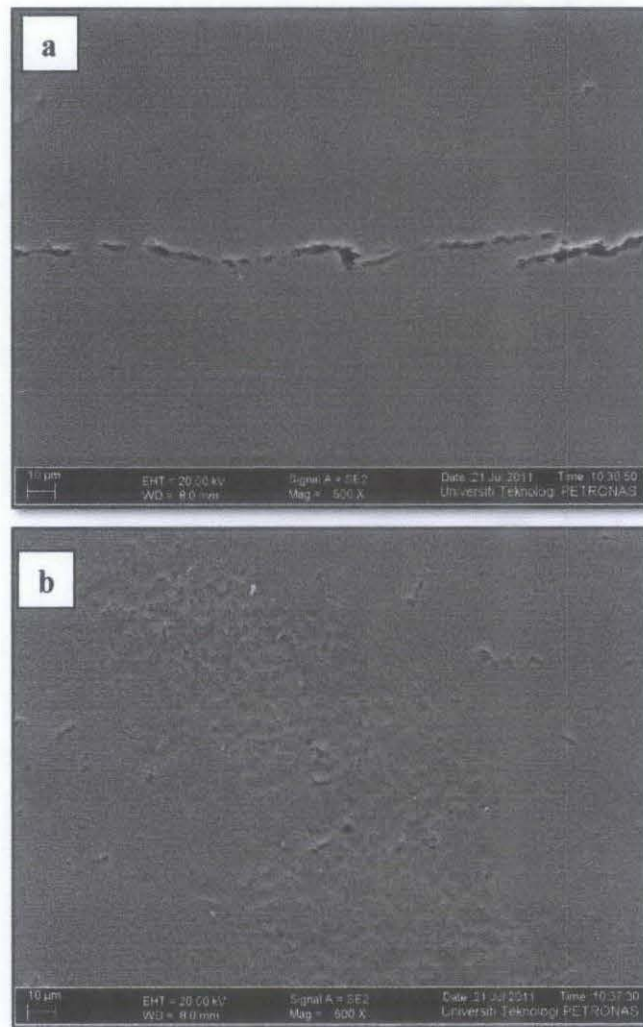


Figure 4.17: The FESEM micrograph at two different sections of FSSW sample

#### 4.2.4 Energy Dispersive X-ray Spectroscopy (EDS)

The EDS analysis of the chemical composition of the FSSW sample shows that the material is composed of aluminum solitary. The sample is in the series of 1xxx aluminum. It is usually softer than the alloys of 6xxx series. It is rarely been used in the industry. <sup>[25]</sup>

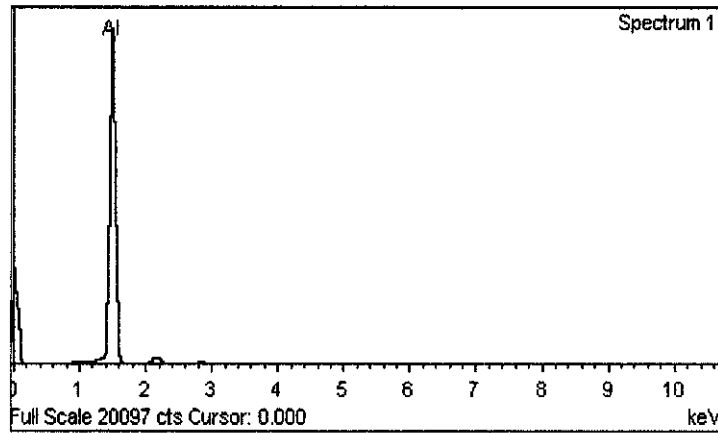


Figure 4.18: EDS spectrum of FSSW sample

Table 4: Chemical composition of FSSW sample

Element	Weight%	Atomic%
C K	15.80	29.66
Al K	84.20	70.34
Totals	100.00	

#### 4.2.5 Grain Size Determination

Planimetric Procedure is an example of grain counting method. This method is described in Section 9 of ASTM E112. The basic steps of the procedure are as follows:

- Inscribe a circle (or other shape) of known area,  $A$ , on an image of magnification,  $M$
- Count the number of grains (completely within the area  $A$ )

- c. Count the number of grains (partially within the area A)
- d. Divide the result in (c) by 2
- e. Add the result in (d) and (b)
- f. Divide the result in (e) by area A
- g. Convert the result in (f) to grains/in<sup>2</sup> @ 100X
- h. Use the definition of ASTM grain size number to determine n

The result in (f) will be given in grains per unit area, measured on the image of magnification, M. to convert this to the 100X equivalent, multiply the result by  $(M/100)^2$ . Area of the square shape is 0.152 in<sup>2</sup> and the magnification of the micrographs is 50X. Based on the calculation above, the result is obtained as in the table below:

Table 5: Grain size of FSSW sample

Regions of welding	Value of n
1. Base metal	5.04
2. TMAZ	5.80
3. HAZ	5.47
4. Weld nugget	6.00



## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 CONCLUSION**

Friction stir welding (FSW) and friction stir spot welding (FSSW) were found to produce microstructural variations across the weld, due to the thermomechanical treatment involved in the process. Optical Microscopy (OM), Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM) had been done successfully. From OM results, we can distinguish the microstructure into four different regions; base metal, TMAZ, HAZ, and weld nugget that is targeted. However, for SEM and FESEM, the microstructure can be captured only at certain spots. It only can be distinguished by the difference in pattern. It was due to lack of sample preparation before doing the SEM and FESEM. EDS also had been done to identify the elemental composition of both FSW and FSSW samples. From the results obtained, the material of FSW is aluminum alloy of 6xxx series, while the material of FSSW is pure aluminum.

#### **5.2 RECOMMENDATION**

This study has been concentrated on the investigation in properties of friction stir welding and friction stir spot welding with similar metals. In future, the following considerations could be improved:

- Inspecting the microstructure at different angle of weldment area.
- Different welding speeds could be applied.
- Both processes may be applied to more different materials. Such as brass, stainless steel and other materials.

## REFERENCES

- 1) *A Comparative Study on the Microstructures and Mechanical Properties of Al 6061 Alloy and the MMC Al 6061/TiB<sub>2</sub>/12P*
- 2) M.Sivashanmugam, S.Ravikumar, T.Kumar, V.Seshagiri Rao, D.Muruganandam, *A Review on Friction Stir Welding for Aluminium Alloys*
- 3) Indira Rani M., Marpu R. N. and A. C. S. Kumar, *A Study of Process Parameters of Friction Stir Welded AA 6061 Aluminum Alloy O and T6 Conditions*
- 4) Murat Dilek Çukurova, *Mechanical Properties of Different Aluminum Alloys Joined by Friction Stir Welding*, Uni Institute of Natural and Applied Sc
- 5) Tsung-Yu Pan, Armando Joaquin, Daniel E. Wilkosz, Larry Reatherford, John M. Nicholson, *Spot Friction Welding for Sheet Aluminum Joining*
- 6) Henkel and Pense *Structure and Properties of Engineering Materials*
- 7) C. B. Smith\*, J. F. Hinrichs\*, P. C. Ruehl\*, *Friction Stir and Friction Stir Spot Welding - Lean, Mean and Green*, \*Friction Stir Link, Inc. W227 N546 Westmound Dr., Waukesha, WI 53186
- 8) *Material Flow Behavior during Friction Stir Welding of Aluminum*  
SUPPLEMENT TO THE WELDING JOURNAL, Sponsored by the American Welding Society and the Welding Research Council, JULY 1999
- 9) Thomas, W. M., *Friction Stir Butt Welding*. U.S. Patent No. 5,460,317, et al. 1991.
- 10) C. J. Dawes, *Recent Advances in Friction Stir Welding – Process, Weldment Structure and Properties* R. Nandan, T. DebRoy and H. K. D. H. Bhadeshia† August 14, 2008
- 11) *Friction Stir Welding* , The Welding Institute, Abington Hall, Cambridge adapted for TALAT by Roy Woodward, Birmingham and Christian Leroy, EAA
- 12) from the Encyclopedia Britannica
- 13) *Metallography Principles and Procedures* by LECO Corporation
- 14) *Topics 3b,c Electron Microscopy*, University of Tennessee, Dept. of Materials Science and Engineering

- 15) Bob Hafner, *Energy Dispersive Spectroscopy on the SEM: A Primer* by Characterization Facility, University of Minnesota—Twin Cities
- 16) René Guinebretière, *X-ray Diffraction by Polycrystalline Materials*
- 17) Shairulafizan bin Muhammad shamsusddin, *Heat Treatment process of Aluminum Alloy to minimize the precipitate free zones and its effect to wear resistance*
- 18) *Information on the FESEM (Field-emission Scanning Electron Microscope,* Radboud University Nijmegen
- 19) *Energy-dispersive X-ray Spectroscopy (EDS),* Analytical Answers, Inc
- 20) *Energy-dispersive X-ray Microanalysis An Introduction,* NORAN Instruments Middleton, Wisconsin
- 21) Prevention Training Ctr. Washington, *Principles of Light Microscopy* St. Louis STD/HIV University School of Medicine St. Louis, Missouri
- 22) New Mexico Tech website
- 23) Peter J. Goodhew, John Humphreys and Richard Beanland, *Electron microscopy and analysis*, third edition, Taylor and Francis
- 24) V. Soundarajan, M. Valant and R. Kovacevic, *An Overview of R&D in Friction Stir Welding*, SMU
- 25) K. Mroczka \*, J. Dutkiewicz , L. Lityńska-Dobrzyńska , A. Pietras, *Microstructure and properties of FSW joints of 2017A/6013 aluminium alloys sheets*
- 26) Adamowski \*, C. Gambaro, E. Lertora , M. Ponte , M. Szkodo, *Analysis of FSW welds made of aluminium alloy AW6082-T6*, J
- 27) M. DI Paola, A. Falchero & M. Cabibbo, E. Evangelista, E. Meccla and S. Splgarelli, *Mechanical and Microstructural Characterisation of an aluminium friction stir-welded butt joint*

**APPENDIX I**  
**Parameter for sample preparation**

A) Data of machining Friction Stir Spot Welding (FSSW)

Date	Spindle speed (rpm)	Plunge feed rate (mm/min)	Move feed rate (mm/min)	Penetration
16/8/2010	4000	55	50	1.6
<b>16/8/2010</b>	<b>5000</b>	<b>55</b>	<b>50</b>	<b>1.6</b>
16/8/2010	6000	55	50	1.6

B) Parameter of mounting

Parameter	Sample 1	Sample 2
Heat time (min)	3.00	3.00
Cool time (min)	5.00	5.00
Pressure (psi)	4000	4000
Diameter of mould (mm)	30	30

APPENDIX II  
Micrograph of OM



Friction Stir Welding sample

- At 6<sup>th</sup> spot:



- At 7<sup>th</sup> spot:

