INSITU PRODUCTION OF NANO METAL MATRIX COMPOSITE (NANO MMC's) USING FRICTION STIR PROCESSING (FSP)

By

MOHD RIDHWAN BIN MOHD NASHIR

DISSERTATION

Submitted to the Mechanical Engineering Programme
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Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

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(Dr Hasan Fawad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

SEPTEMBER 2012

CERTIFICATION OF ORIGINALITY

This is 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 acknowledgments, and that original work contained herein have not been undertaken or done by unspecified source or person.

(Mohd Ridhwan bin Mohd Nashir)

ABSTRACT

Metal matrix composite (MMC) is one of the technologies used in a big or small scale of manufacturing. MMC are produces by different method such as fabricated using Friction Stir Processing (FSP) method which is innovated from Friction Stir Welding which had been started in 1991. The composites fabricated possess good Young Modulus, good compressive strength and good compressive ductility. One of the common metals used in the process is Aluminium and it is combined with Copper during the process to produce Aluminium Copper Metal Matrix Composite.

The metal matrix will be fabricated using CNC vertical milling center and specially designed FSP tool. The MMC fabricated will be fabricated with different parameters during the process and they will be tested in several tests to show its enhancement in characteristics.

ACKNOWLEDGEMENT

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CHAPTER 1: INTRODUCTION

1.1 Project Background

Friction Stir Processing (FSP) is a new process adapted from Friction Stir Welding (FSW). Friction Stir Welding was developed in 1991 by The Welding Institute (TWI). The features of friction stir welding like low amount of heat generated, extensive plastic flow of material, very fine grain size in the stirred region, healing of flaws and casting porosity, random misorientation of grain boundaries in the stirred region and mechanical mixing of the surface and subsurface layers used to developed the process. This process can be applied to produce the Metal Matrix Composites (MMC's), which have better properties as compared to the base material. [1]

1.2 Problem Statements

MMC can be produced by different method such as Liquid Metallurgy and Powder Metallurgy. The main problem associated with liquid in homogenous distribution of reinforcement, reaction of base material, experiment, and oxidation and hydrogen porosity. Powder metallurgy is slow and experience process and gives a course situation.

The MMC using FSP, have these advantages of removing these problems and it had given a better and verified structure. Due to these advantages FSP being studied for producing MMC.

1.3 Objectives and Scope of Study

1.3.1 To use experimental set up for manufacturing Nano Metal Matrix Composite of Aluminium and Copper, $Al\text{-}Al_2Cu$ using Friction Stir Processing.

1.3.2 To analyze the Nano Metal Matrix Composite by using material characterization techniques.

CHAPTER 2: LITERATURE REVIEW

Metal matrix composites (MMC's) consist of a metal matrix and a reinforcement which improves the mechanical properties of the composite. The principal matrix materials for MMC's are aluminium and its alloys. Other materials include, metal like Copper, Zinc or lead matrix used for specialized applications.[2] MMC's give many advantages over monolithic alloys such as major weight savings due to higher strength-to-weight ratio, exceptional dimensional stability, higher elevated temperature stability and significantly improved cyclic fatigue characteristics. Generally there are three types of MMC which are particle reinforced MMCs, short fibre or whisker reinforced MMCs and continuous fiber or sheet reinforced MMCs. [3]

Friction Stir Processing is a novel approach recently applied for the production of the insitu & post situ. This process is being used for production of insitu MMC's in two ways. One way is the production of composites coating on the surface of base material and other is to produce the bulk MMC's. Similarly reinforcements can be added to the system in two ways one is in the form of ceramic particles like SiC or Al_2O_3 and second way is producing the insitu intermetallic articles during the FSP.

CHAPTER 3: METHODOLOGY

3.1 Project Work

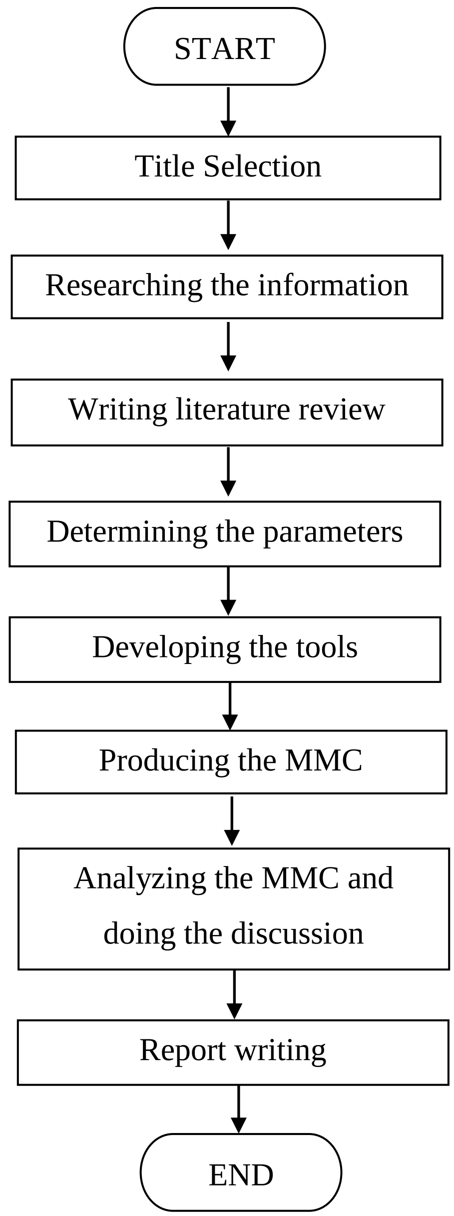

Figure 1: Project Activities Flow Chart

3.2 Research Methodology

Research is a method taken in order to gain information regarding the major scope of the project. The sources of the research cover the handbook Frictional Stir Processing, e-journal, and several trusted link.

The steps of research:

3.2.1 Gain information of frictional stir processing and its application in industry.

3.2.2 Discuss with Supervisor and Graduate Assistant on the parameters of the process.

3.2.3 Decide the analysis that will be used to evaluate the MMC produced

3.3 Activities/Gantt Chart and Milestone

Detail/Week	1	2	3	4	5	6	7	Mid-Semester Break	8	9	10	11	12	13	14
Selection of Project Topic															
Preliminary Research Frictional Stir Processing															
Literature Survey															
Discussion of parameters used in the process															
Submission of Extended Proposal Defence															
Proposal Defence Presentation															
Test rig preparation															
List down all the materials needed for manufacturing MMC															
Order the materials needed for manufacturing MMC															
Submission of Interim Draft Report.															
Submission of Interim Report.															

Figure 2: Gantt chart and Key Milestone for FYP 1

Detail/Week	1	2	3	4	5	6	7	Mid-Semester Break	8	9	10	11	12	13	14	15
Project work continues																
Submission of progress report									●							
Project work continues																
Pre SEDEX poster and presentation												●				
Submission of draft report													●			
Submission of dissertation (soft bound) and technical paper														●		
Oral presentation															●	
Submission of dissertation (hard bound)																●

Figure 3 : Gantt chart and Key Milestone for FYP 2

3.4 Material used for the tool and sample

After some discussions done with Supervisor and GA, we decided to use 2 types of materials as the sample and 1 type material as the tool.

Material for sample:

3.4.1. Aluminium plate (Al 1100)

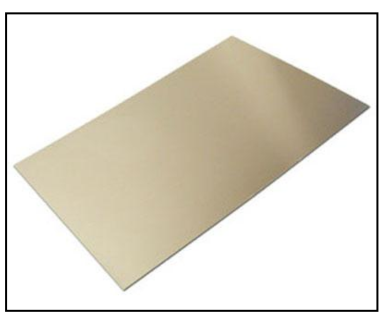

Figure 4 : Aluminium plate

Al 1100 has relatively low strength but excellent in formability, weldability and corrosion resistance. The percentage of Al contained in Al 1100 approximately 99%.

Grade	Chemical Composition %										
	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Other		Al
									Each	Total	
1100	0.95 max		0.05-0.2	0.05 max	-	-	0.1 max	-	0.05 max	0.05 max	99.00 min

Table 1: Chemical composition of Al 1100

Grade	Temper grade	Tensile test			
		Thickness (mm)	Tensile strength (N/mm^2)	Proof strength (N/mm^2)	Elongation %
1100	H14	0.2 or over, up to and incl. 0.3	120 min 145 max	-	1 min
		Over 0.3, up to and incl. 0.5		-	2 min
		Over 0.5, up to and incl. 0.8		-	3 min
		Over 0.8, up to and incl. 1.3		95 min	4 min
		Over 1.3, up to and incl. 2.9		95 min	5 min
		Over 2.9, up to and incl. 12		95 min	6 min

Table 2 : Mechanical properties of Al 1100

3.4.2. Copper strip

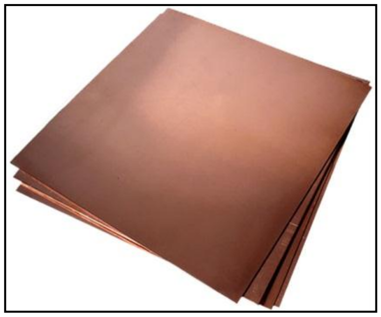

Figure 5: Copper strip

Material for tool:

3.4.3 Stainless steel

Figure 6 : FSP tool (stainless steel)

Stainless steel used because of high resistance to temperature and pressure and availability in market.

3.5 Machine and tools used

3.5.1 Bridgeport CNC Milling Machine

Figure 7 : Bridgeport CNC Milling Machine

3.5.2 FSP tools

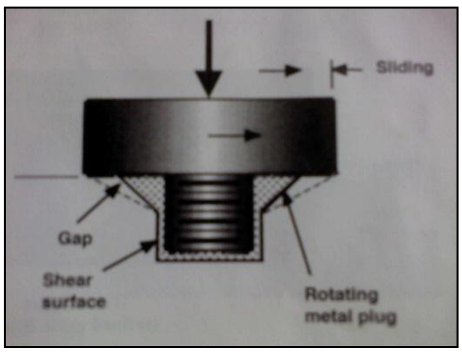

Figure 8 : FSP tool diagram

This tool is going to be used in the project. The tools will be prepared using CNC Lathe machine at Block 21 using stainless steel as the material. The tool is very important it needs to withstand high speed and high temperature during the process.

3.5.3 Field Emission Scanning electron microscope (FESEM)

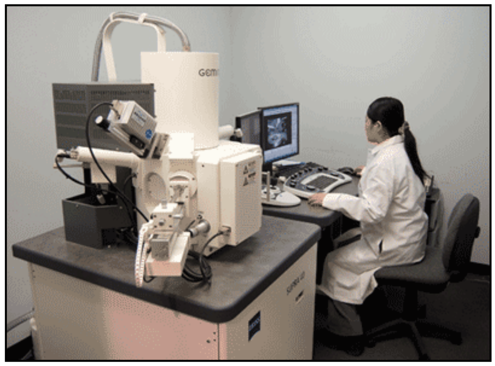

Figure 9 : FESEM equipment

3.5.4 Optical Microscope

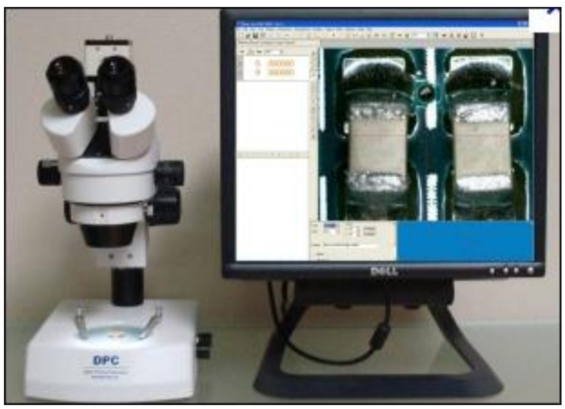

Figure 10 : Optical Microscope equipment

3.5.5 X-ray Diffraction (XRD)

Figure 11 : XRD machine

3.6 Procedure for producing metal matrix composite

Bridgeport CNC Milling machine used to produce metal matrix composite using Friction Stir Processing method. There are 2 components need to prepared before starting the processes which are jig and tool. Jig is used to clamp the aluminium plates and support them during the process while tool used to weld the joint.

Process of FSP using Bridgeport machine is done as follows:

1. Two aluminium plates are joining as lap joint and they are placed inside the jig and it will be tighten up using the bolt and nuts at the jig.
2. The jig then been place inside the Bridgeport Milling Machine and it is clamped tightly.
3. The parameters are set up before start the process.
4. The safety door will be closed as the process begins.
5. After the process finish, let it cool for a few minutes before taking out the samples.

Figure 12 : Experiment jig

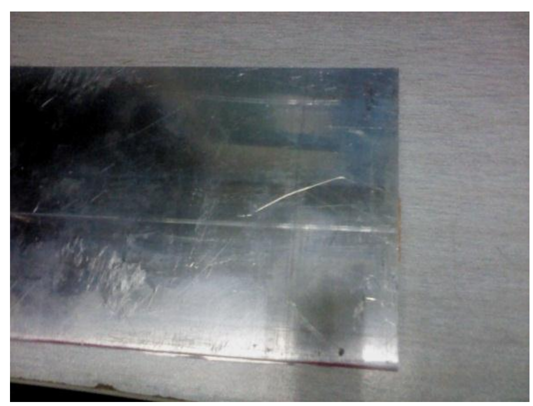

Figure 13 : Al 1100 plate

Figure 14 : FSP process

3.7 Sample preparation for microstructure testing

To observe the microstructure of the Metal matrix composite produced, there are 4 steps must be done to reveal the microstructural information of the metal matrix composite,

1. Cutting
2. Grinding
3. Polishing
4. Etching

3.7.1 Cutting

Cutting is a process to get the part of the aluminium which we want to observe. The aluminium plates are cut into 4 parts. After that the each of the parts are cut again at the middle into small pieces to get the metal matrix composite. EDM wire cut machine used to cut the samples in order to get precise shape and reduce the mechanical damage on the sample.

Figure 15 : Nano Metal Matrix Composite after been cut

3.7.2 Grinding

After finish cutting the samples, the metal matrix composite then prepared for grinding. The purpose of grinding the sample is to minimize the thickness of damaged layer from the sectioning process. The samples are grinding using SiC paper and water as lubricant. The grade of grinding paper used is 400 grits (grains per square inch).

Figure 16 : Grinding process

3.7.3 Polishing

Then the grinded samples are going to continue with the next process called polishing. Lubricant is use is Metadi fluid and Diamat paste. Firstly the tri-cloth is sprayed with Metadi fluid, then after that Diamat paste is put on the surface of the samples. The polishing process is done once the mirror-like image appeared.

Figure 17 : Polishing solid paste

Figure 18 : Polishing process

3.7.4 Etching

The last process before the sample is been observe on microstructure is etching. Etching process is important to reveal the microstructure of the metal through selective chemical attack. Etchant used for aluminium called Keller’s reagent. Keller’s reagent is prepared accordingly to these compositions of solution:

- 95% of distilled water, H_2O
- 1% of hydrofluoric acid, HF
- 1.5% of nitric acid, HNO_3
- 2.5% of hydrochloric acid, HCl

The solutions are mixed together in fume chamber, and then once the etchant is ready, the samples are dip into the etchant for 20 seconds. Then they are washed with distilled water and let them dry before move to the next step.

CHAPTER 4: RESULT AND DISCUSSION

4.1 Parameter of the processing

There are some factors that must be considered before beginning producing MMC using FSP. These factors called processing parameter. Processing parameter is the parameter set up on certain setting of the CNC Milling machine and setting of the aluminium plates. The processing parameter include in this experiment are spindle speed, traverse speed, type of joint and the penetration depth.

4.1.1 Spindle speed

Spindle speed is the rate at which machine spindle rotates. It is measured in rpm. The designated tool is attached at the spindle to perform the process on the aluminium plates. The spindle speed set up for this experiment is 1200rpm.

4.1.2 Traverse speed

Traverse speed is the rate at which machine spindle moves horizontally. It is measured in mm/min. The tool attached at the spindle will rotate and it will move horizontally form one point to another point. The traverse speed set up for this experiment is 50mm/min.

4.1.3 Type of joint

In Friction Stir Welding, there are various methods how to join the aluminium plates. Each of type of joining has different purpose. The type of joint set up for this experiment is Lap Joint. The second aluminium plate will be place on the first aluminium plate and copper strip will be place at the middle of the plates and in between two aluminium plates.

4.1.4 Penetration depth

Penetration depth is the depth of the tool penetrates inside the plates. The depth decided accordingly to the thickness of the plates. Since the thickness of two aluminium plates is 10mm, 9.2mm is the best penetration depth set up for this experiment.

Parameter	Parameter's Value
Spindle speed	1200rpm
Traverse speed	50mm/min
Type of joint	Lap join
Penetration depth	9.2mm

Table 3 : Processing parameter

4.2. Microstructure analysis by Optical Microscope

Microstructural analysis is method of which its purpose to analyze surface of a material. The microstructure revealed by selective etching with appropriate acid solution. Keller's reagent used in this experiment to etch the surface of MMC. After the MMC has been etched using Keller's reagent, then its surface will be observed using optical microscope. Microstructural analysis was done to observe the formation of Aluminium and Copper. Aluminium and Copper are expected to mix together to form metal matrix composite. There are also possibilities that Copper strip will not mix with Aluminium due to its higher melting point than Aluminium. Surface of MMC is observed using optical microscope in magnification of 10X and 50X.

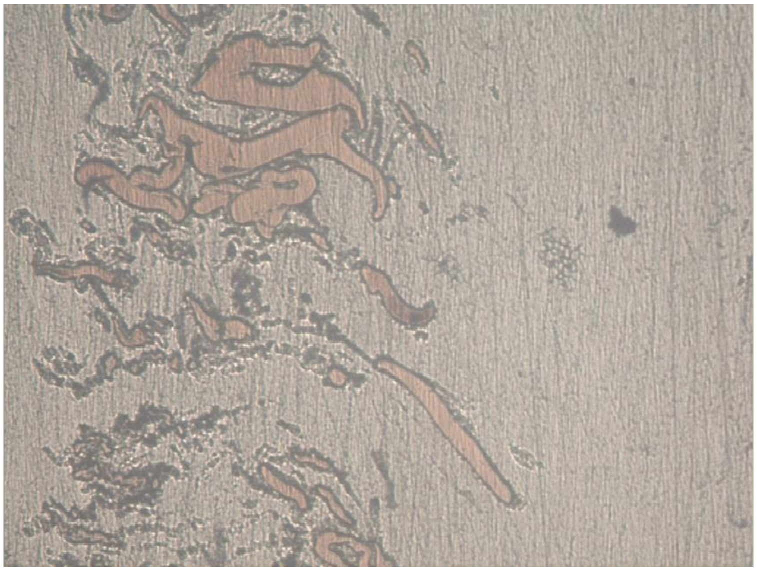

Figure 19 : Microstructure image of MMC surface with 10X magnification

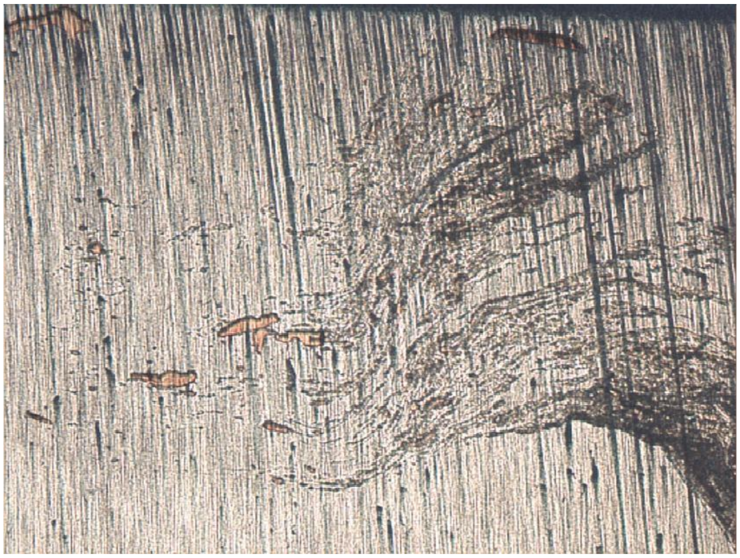

Figure 20 : Microstructure image of MMC surface with 50X magnification

The gold color portion in the image shows the Copper which did not mix with Aluminium while the other part of the image is Aluminium and metal matrix composite of Aluminium and Copper. There is metal matrix composite produce but in very small size which is in nano size. The microstructure image of MMC cannot be viewed underneath the optical microscope because the size is very small and if the magnification of the optical microscope is increased, the view is not clear and it only display black image.

It is seen from the image that the Copper strip is distributed randomly throughout the surface of aluminium. It is because the copper strip is not mix completely with aluminium and the movement of the tool is not uniform.

4.3 Characterization of Nanometal matrix composite using X-ray diffraction.

X-ray diffraction or XRD used to detect the presence of Aluminium Copper compound inside the sample. The limitation of optical microscope can be overcome by using XRD. XRD will show all the elements presence in the sample. All the possible elements present in the sample filtered to the specification.

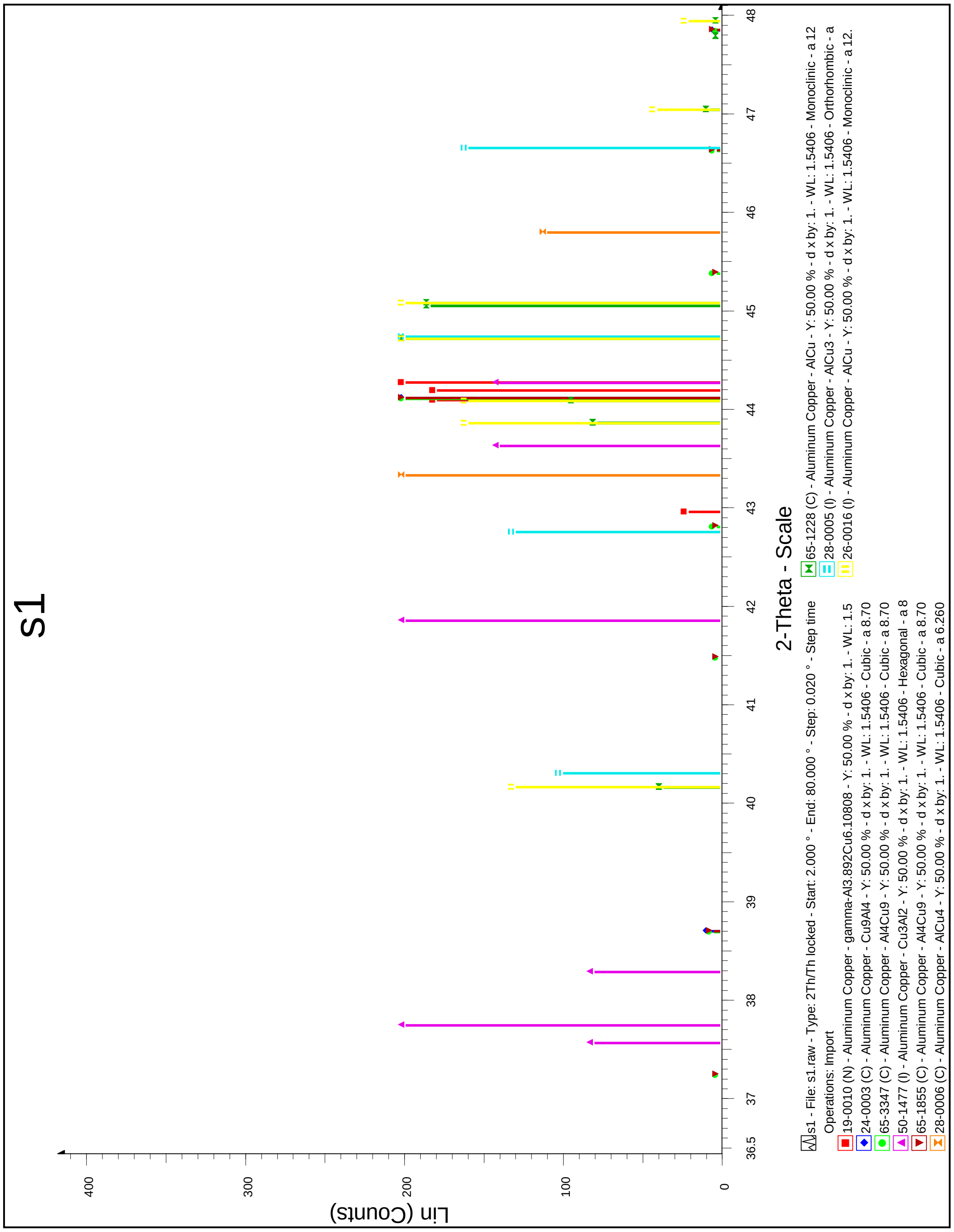

Figure 21 : XRD graph

The graph shows all the possible compound of Aluminium Copper inside the sample. From the XRD test, it is shown that Nano Metal Matrix Composite of Aluminium Copper formed. There is some information that XRD could not provide such as the composition of the elements in the sample.

4.4 Composition Determination of Nano Metal Matrix Composite using FESEM

Field emission scanning electron microscopy used to observed more detail on the sample. There are 3 tests that will be carried out using FESEM which is morphology, EDX and elementary mapping.

4.4.1 Morphology image of Nano Metal Matrix of Aluminium Copper

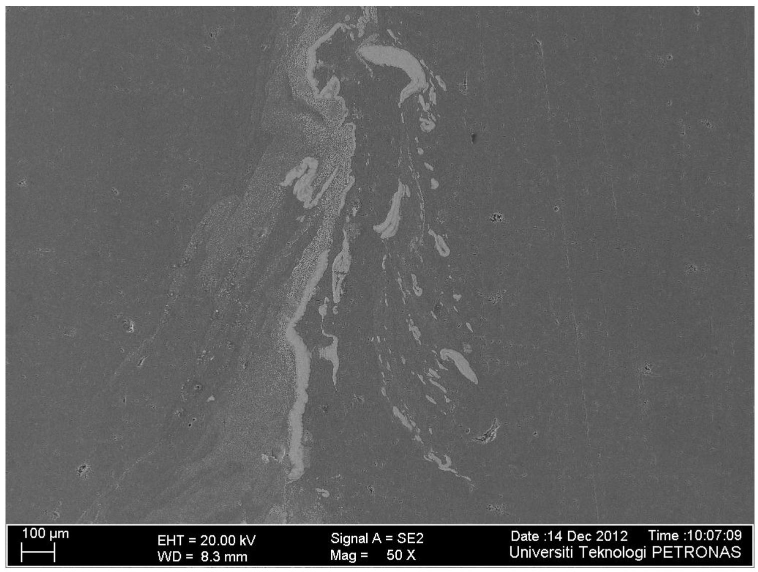

Figure 22 : FESEM image of Nano MMC Al Cu (50X magnification)

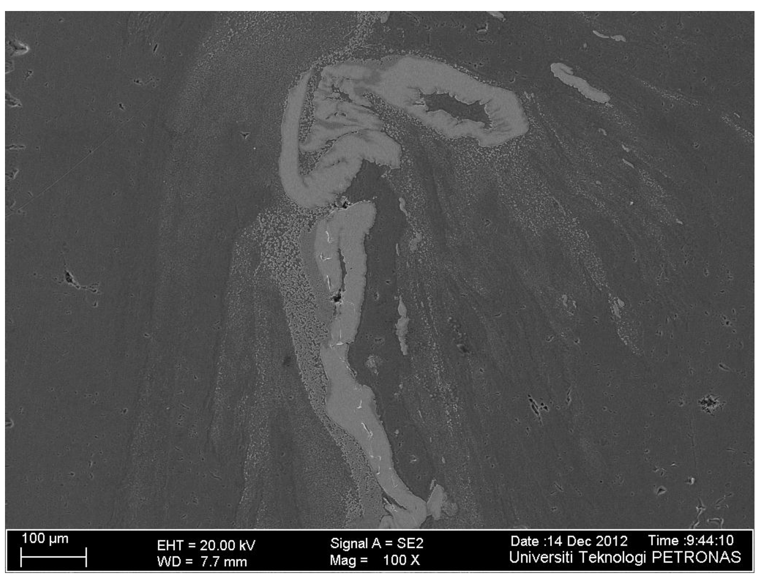

Figure 23 : FESEM image of Nano MMC Al Cu (100X magnification)

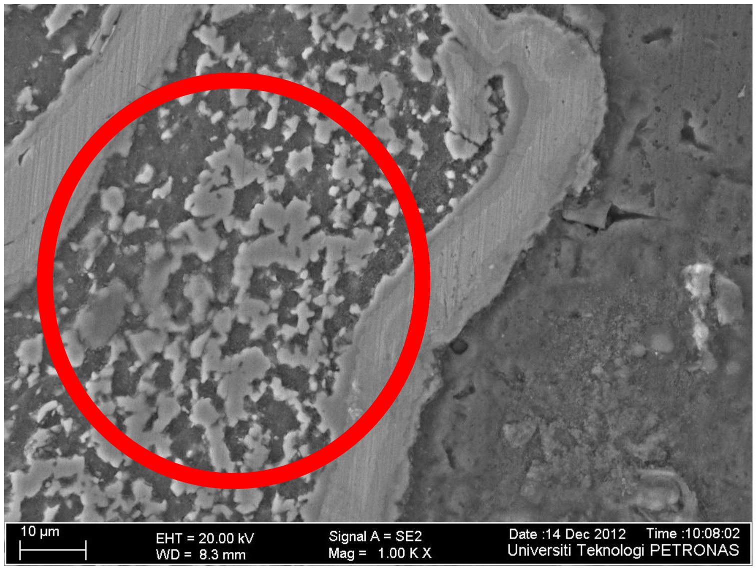

Figure 24 : FESEM image of Nano MMC Al Cu (1000X magnification)

The darker colour region in the image shows the Aluminum and the less dark colour region in the image is Copper. The Nano Metal Matrix Composite of Aluminium Copper formed at the region marked in the figure 24.

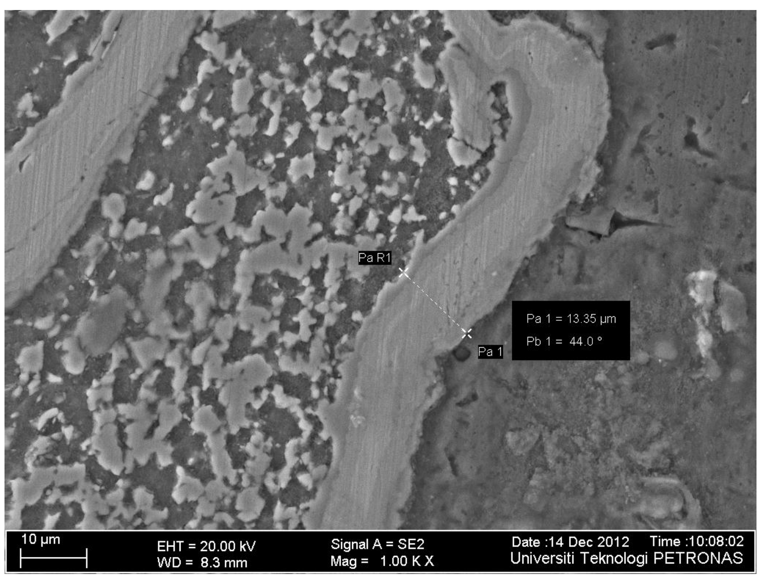

Figure 25 : FESEM image that shows the thickness of Copper strip after FSP

From the image, the thickness of the Copper strip remains after FSP is 13.35μm. The thickness of the copper strip before the process is 0.1mm.

$$\%\ Copper\ disperse = \frac{Initial\ thickness - Final\ thickness}{Initial\ thickness} X\ 100\%$$

$$\%\ Copper\ disperse = \frac{1x10^{-4}m - 1.335x10^{-5}m}{1x10^{-4}m}\ X\ 100\% = 86.65\%$$

86.65 % Copper strip had been disperse as a result of FSP. Part of the dispersive are mix with aluminium to form Nano Metal Matrix Composite while the part remains as Copper in very small size.

4.4.2 Energy dispersive x-ray (EDX) and EDX mapping micrograph

EDX is a technique used to investigate the elemental analysis of a sample. It relies on the investigation of an interaction of some source of x-ray excitation and a sample. EDX is used to analyze the elemental analysis of Nano Metal Matrix Composite of Aluminium Copper. From the FESEM image of the sample, 12 points mark on the surface of the sample to perform elemental analysis at different point on the sample. The writing on the image shows the position of marking point.

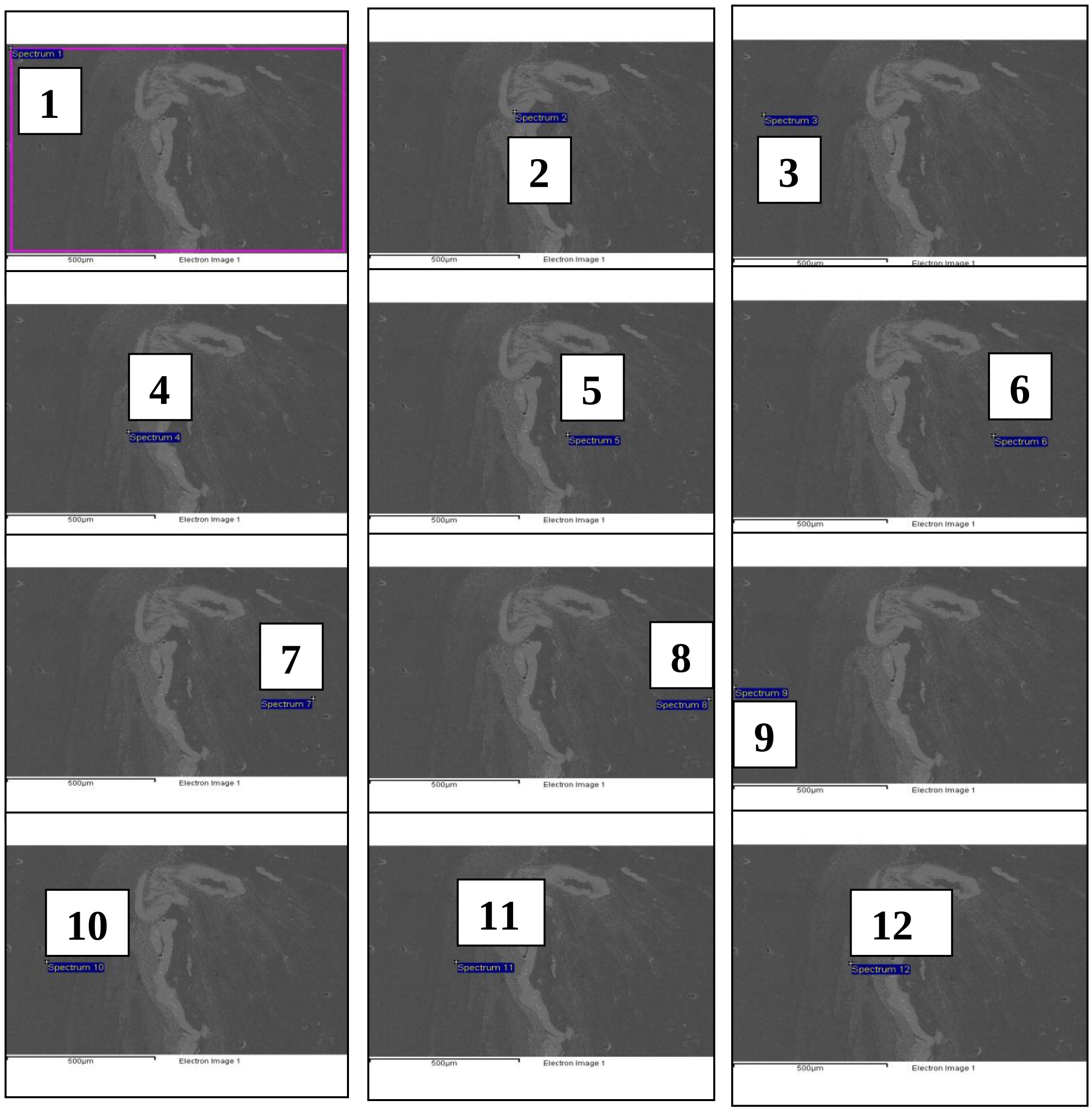

Figure 26 : Multiple spectrum point on the sample image for EDX analysis

Spectrum No.	Atomic percentage	
	Al	Cu
1	71.87	3.74
2	1.49	57.59
3	100	0
4	67.86	1.68
5	52.59	4.6
6	71.78	0.71
7	100	0
8	90.7	0
9	99.73	0
10	100	0
11	76.57	0.48
12	69.26	1.35

Table 4 : Table of elemental composition of Al and Cu

According to the figure 26 and the table 4, it shows the some point on the sample with the elemental composition of aluminium and copper at that point. The copper composition at the middle of the sample is higher because copper strip is positioned at the middle before the process. After the process, the composition of the copper does not concentrated at the middle but it spread at any orientation on the surface, thus the composition of the copper decreased at the middle and it exist at the sides of the surface.

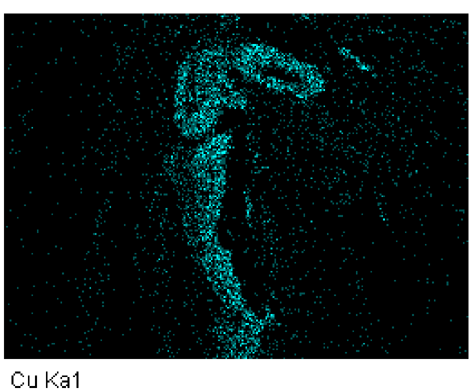

Figure 27 : Elemental mapping of Cu

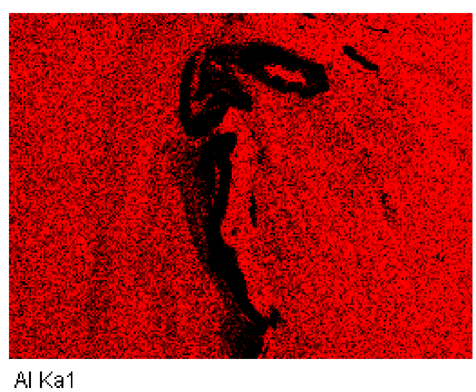

Figure 28 : Elemental maaping of Al

From the elemental mapping image of aluminium and copper, we can see the distribution of the copper and and aluminium on the sample. Thus, Nano Metal Matrix Composite Aluminium Copper formed but it formed non uniformly.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

With the analysis done in the result section, it is safe to conclude that Nano Metal Matrix Composite of Aluminium Copper can be produce by using Friction Stir Processing. The sample is been cut into small pieces for characterization test. It is proof that Nano Metal Matrix Composite of Aluminium Copper produce from optical microscopic, XRD and FESEM test. Nevertheless it is form non uniformly and it is hard to determine the specified Nano Metal Matrix Composite of Aluminium Copper.

5.2 Recommendation

For future work, it is recommended to measure the ratio of the Aluminium and Copper used so that Copper will mix completely with Aluminium. High melting temperature of Copper compared to Aluminium make the composite hardly to form if the ratio of both metals does not take into account.

The micrograph of Aluminium and Copper should be taken before the process as a comparison after the process. The sample should be produced in some numbers so with different processing parameter. The properties of the metal matrix composite can be studied by setting various processing parameter.

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APPENDICES