A STUDY ON THE MECHANICAL EFFECTS OF

GAS NITRIDING ON ALUMINIUM ALLOY 6061

FOR THE USE

IN MOBILE PHONE CASING

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Dissertation submitted in partial fulfilment of

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CERTIFICATION OF APPROVAL

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Approved by,

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January 2016

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 acknowledgments and that the work contained herein have not been undertaken or done by unspecified sources or persons.

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Muhammad Firdaus Bin Jinal Ahbidin

ABSTRACT

The constant demands for low weight and high strength mobile phone casing lead manufacturers to utilize aluminium for the material selection. In this study, heat treatment process will be conducted on Aluminium Alloy 6061 by means of gas nitriding process. In this process, the aluminium sample was heat treated at temperature 550℃ while at the same time nitrogen gas diffused into the surface material after being introduced into the tube furnace. The samples were then tested for its improved properties using Rockwell hardness testing machine and Revetest Scratch Tester for its hardness and scratch resistance. The microstructure test was carried out to examine the formation of nitride layer using Optical Microscope (OM) and Scanning Electron Microscope (SEM) built in with Energy Dispersive Spectrometer (EDS). The experimental results comprised data comparing with both nitrided and unnitrided samples.

ACKNOWLEDGEMENT

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND STUDY

Metal comes out as the most popular material option for mobile phone casing as its shiny and sturdy looks gives a premium feel to the consumer. Aluminium crafted body have received immense praise as we can see the increasing demand for aluminium casing in numerous models such as iPhone, HTC One Series and many more. The high quality looks of aluminium with precisely-crafted unibody designs giving luxury and exclusive sensation to the owners. Despite the luxurious looks, with rough usage or condition, aluminium casing is proven to experience scratches, bending and wearing of corrosion coatings. In this scenario, it is clear that crucial properties such as hardness will affect the wear resistance of the casing to ensure durable appealing look of the smartphone. Increasing the hardness will increase the wear resistance. These properties can be improved using many techniques and one them is heat treating. Gas nitriding is classified as heat treating process which suitable in enhancing surface hardness of steels containing nitride-forming elements such as chromium, molybdenum, vanadium and aluminium. Aluminium Alloy 6061 is selected as the project material since this alloy is commonly manufactured as the smartphone casing.

1.2 PROBLEM STATEMENT

1.2.1 Aluminium has low hardness and wear resistance.

The hardness of aluminium is relatively low due to its low density. It possess poor wear resistance which affects the smartphone handling in rough daily activities.

1.2.2 Additional protective cover equipped is less user-friendly.

Adding protective cover undeniably protect the smartphone body from impact that can damage the internal components. However, this accessory make the smartphone more bulky and inconvenient to be carried in the pocket. Furthermore, equipping protective cover hides the sleek design of the smartphone.

1.3 OBJECTIVES

With regards to the problems stated, this study was carried out to achieve these following objectives:

1. To perform gas nitriding process on aluminium.
2. To study the mechanical effects of gas nitriding on the surface hardness, microstructure and wear resistance of the aluminium.
3. To compare the hardness and wear resistance of nitrided with unnitrided aluminium.

1.4 SCOPE OF STUDY

1.4.1 Conducting gas nitriding process on aluminium samples.

- Specimen used is a same material as smartphone casing and cut according to the size stated in ASTM standard for scratch and hardness testing.

1.4.2 The study focused on microstructure, hardness and wear resistance of nitrided and unnitrided aluminium.

- This study will determine the changes in microstructure, hardness and relationship with wears resistance for unnitrided and nitrided sample.

1.4.3 Carry out the scratch and hardness testing using ASTM Standard.

- All procedures for scratch and hardness testing will be following the ASTM standard to obtain credible results.

1.4.4 Performing microstructure testing using OM and SEM

- Microstructure testing will be conducted to review the formation of nitride layer on the sample's surface after the nitriding process using SEM.

1.5 PROJECT SIGNIFICANCE

Aluminium exhibits a high commercial value in smartphone production due to its lightweight and sturdy properties which make it suitable to be designed as a smartphone casing. Since smartphone crafted from the aluminium is costly, improving the surface hardness will increase the time worth value of money for the consumer to buy. Performing gas nitriding on aluminium casing smartphone will increase the hardness and wear resistance against abrasion or impact such as falling that could damage the smartphone.

CHAPTER 2

LITERATURE REVIEW

2.1 FUNDAMENTAL OF GAS NITRIDING

Gas nitriding is a heating treatment process used to enhance surface hardness and wears resistant of the materials by creating nitride layer after nitrogen is induced and diffused into the solid surface of ferrous alloy. This reaction requires heating temperature under melting point of metal inside the furnace under nitrogen rich atmosphere [1]. The essence of this process is the nitride layer formed provide impressive case hardening without involving phase change in the molecular structure of the metal. This means that the molecular structure of the ferrite which is body-centered cubic (BCC) lattice does not change its configuration or grow into the face-centered cubic (FCC) lattice characteristic of austenite, as occurs in more conventional methods such as carburizing [2].

The purpose of using low temperatures is to avoid the sensitization which is the formation of chromium nitrides and carbides in alloyed layer. There is two stage for heat treatment which is single-stage nitriding and double stage nitriding. For single stage, the metal to be nitrided is heated to a temperature ranging from 495℃ to 525℃. For double stage, the first stage is the same as single stage. The second stage take place when the temperature is raised to 550℃ to 565℃. This stage is important to lower the case and core hardness as well as the apparent effective depth and increase case depth of the material [1].

The nitride layer has typical thickness approximately between 15 to 30 μm. The greater or lesser thickness of nitride layer can be used depending on particular applications. The nitriding process has several advantages over other coating process. Nitriding is relatively low-temperature process, the process not required quenching for hard case, relatively easy to control in term of process parameters and typically less part distortion [3].

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2.2 ALUMINIUM ALLOY 6061

Aluminium 6061 is a well-known aluminium alloy which has been used in manufacturing industry since 1935. It is one of the most popular used alloys in 6000 series for medium to high strength requirements. Table 1 shows the composition compound of Aluminium 6061 while its mechanical properties is shown in Table 2. It has excellent corrosion resistance to atmospheric environment and good corrosion resistance to saline environment. It can be anodized for stronger and more durable physical appearance by coating the surface electrolytically when aluminium is oxidized to aluminium oxide [4].

The interesting feature that made aluminium alloy to be developed into smartphone casing regardless costly is because of its low density or low weight which make it suitable to be carried effortlessly. Its high stiffness and toughness provide stronger protection structural to the internal components of smartphones preventing them from damage upon impact [5].

TABLE 1. Composition of aluminium alloy 6061

Component	Symbol	Amount (wt. %)
Aluminium	Al	Balance
Magnesium	Mg	0.80 – 1.20
Silicon	Si	0.40 – 0.80
Iron	Fe	Maximum 0.70
Copper	Cu	0.15 – 0.40
Zinc	Zn	Maximum 0.25
Titanium	Ti	Maximum 0.15
Manganese	Mn	Maximum 0.15
Chromium	Cr	0.04 – 0.35
Other	-	0.05

TABLE 2. Mechanical properties of aluminium alloy 6061

Properties	Value
Density	2.70 g/cm^3
Melting Point	650 ℃
Thermal Expansion	23.5×10^{-6} m/m.℃
Modulus of Elasticity	70 GPa
Thermal Conductivity	166 -173 W/m.K
Electrical Resistivity	3.7- 4.0 ×10^{-6} Ω.m
Tensile Strength	Minimum 260 MPa

2.3 GAS NITRIDING PROCESS ON ALUMINIUM ALLOY 6061

2.3.1 Conventional Nitriding Using Ammonia Gas as Nitrogen Donor

In this method, the Aluminium is heated in the furnace with ammonia gas being flown into the furnace at temperature ranging from 500℃ to 590℃. This result with thermal chemical reaction of ammonia as its molecule decomposed into nascent nitrogen and hydrogen when it contact with hot surface metal as shown in the equation below [1].

$$2\,NH_3 \rightarrow 2N + 3H_2 \ldots\ldots\ldots(Eq.1)$$
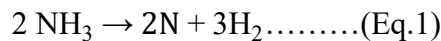
Part of the nascent nitrogen created diffuses into the interior of the aluminium substrate and subsequently forming a nitrided case. The process is controlled by adjusting the flow rate of ammonia where dissociation rate for ammonia is between 15% to 30% for single stage nitriding and 75% to 80% dissociation rate for double stage nitriding [1].
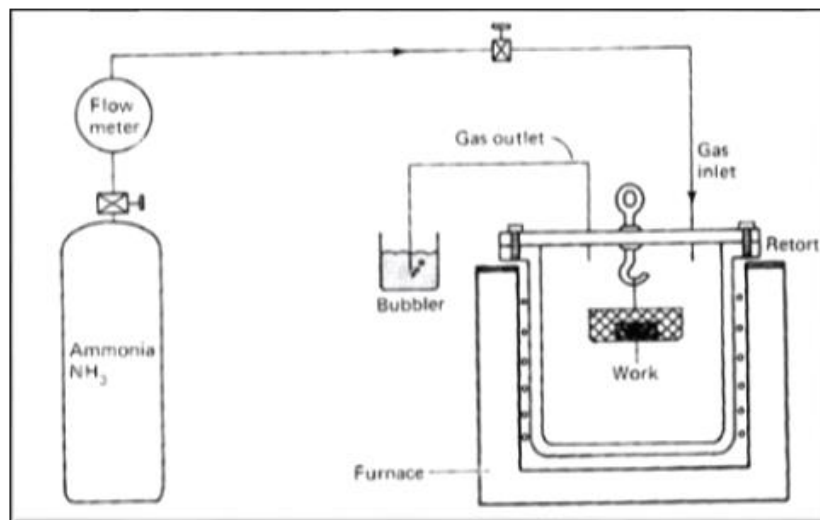
FIGURE 1. Gas nitriding process using ammonia

2.3.2 Barrel Nitriding and Standard Nitriding Using Pure Nitrogen

As the name implied, this method used a barrel which is normally used for plating or as polishing is equipped where Alumina Al_2O_3 powders and Al-Mg alloy powder are made as the filling inside the barrel. The metal is heated and nitrided consecutively during the oscillating movement of the barrel tank. As the barrel swing, these 100 μm grain size powders flow will like a liquid while at the same time nitrogen gas is introduced within the flow rate ranging 0.2 L/min to 3.0 L/min under the

temperature 500℃ to 800℃. Whereas for the standard nitriding, there is no additional powders to react with the substrate and the metals only being exposed to the flowing nitrogen inside the furnace [6].
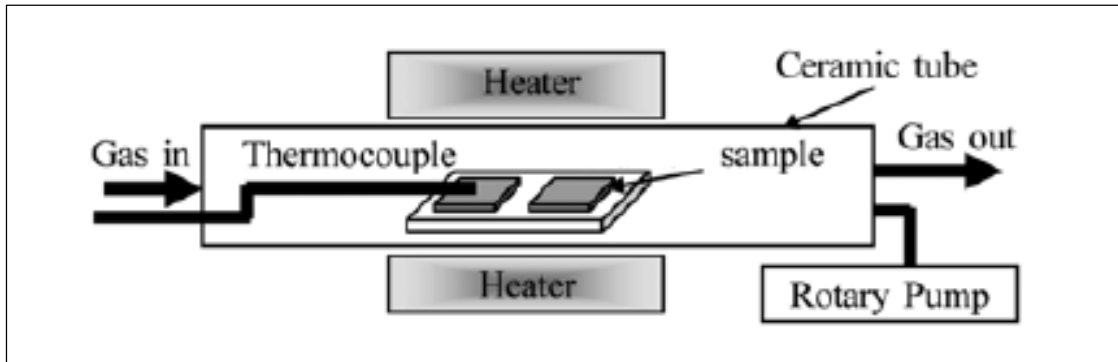
FIGURE 2. Gas nitriding with pure nitrogen
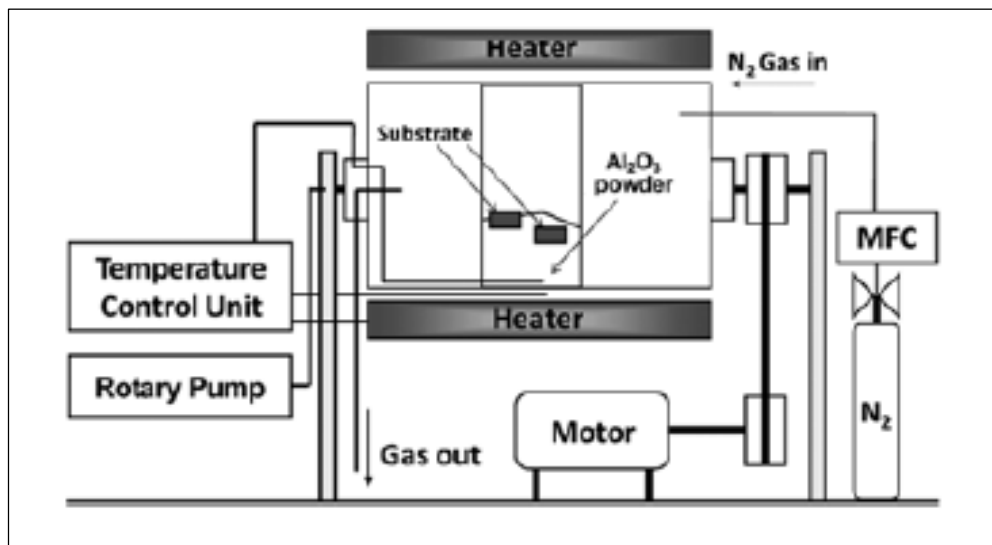
FIGURE 3. Barrel nitriding

2.3.3 Nitrocarburizing Using Carbon and Nitrogen Gas

This process is a modified form of gas nitriding which is known as gas ferritic nitrocarburizing. In the process, nitrogen and carbon are simultaneously introduced into the steel while it is in the ferritic condition, that is at a temperature below which austenite begins to form during heating [10]. Nitrocarburizing atmospheres which typically performed at high temperature for about 570℃ exhibit more complex composition containing NH_3, H_2, CO, CO_2 and H_2O. This atmosphere containing NH_3 and H_2 gas components to initiate nitriding reaction as in equation 2 whereas CO and CO_2 to establish carburizing reaction as in equation 3 [11].

$$CO_2 + H_2 \leftrightarrow CO + H_2O\ldots\ldots..(Eq.2)$$

$$CO + H_2 \rightarrow C + H_2O\ldots\ldots\ldots(Eq.3)$$
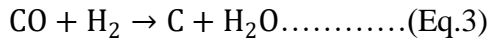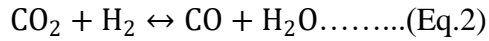
Hydrogen needed for this reaction originates from dissociated ammonia as in equation 1 or by separate addition of hydrogen. Carbon monoxide will formed as the product which then react with hydrogen to form carbon. Table 3 show the typical percent of gas mixture used in nitrocarburising process. Apart from CO_2-H_2-H_2O reaction atmosphere or better known as heterogeneous water gas reaction, carburizing also can be achieved by exposing the specimen in a CH_4-H_2 atmosphere according to equation 4 below [12].

$$CH_4 \leftrightarrow C + 2H_2\ldots\ldots\ldots(Eq.4)$$
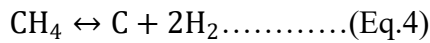
TABLE 3. Composition of gas mixture

Added Gas Mixture	Equilibrium Composition (vol%)					
	N_2	H_2	CO	H_2O	CO_2	NH_3
50% Endogas, 50% NH_3	24.1	25.9	9.0	0.76	0.68	38.4
40% Endogas, 10% Air, 50% NH_3	29.2	24.3	6.7	2.46	1.77	35.4
5% CO_2, 60% N_2, 35% NH_3	58.8	15.5	2.9	2.98	1.45	18.4
5% CO_2, 5% CO, 55% N_2, 35% NH_3	54.4	15.9	6.4	2.22	2.31	18.6
5% CO_2, 20% H_2, 45% CO, 30% NH_3	45.4	26.6	3.4	3.5	1.16	19.9
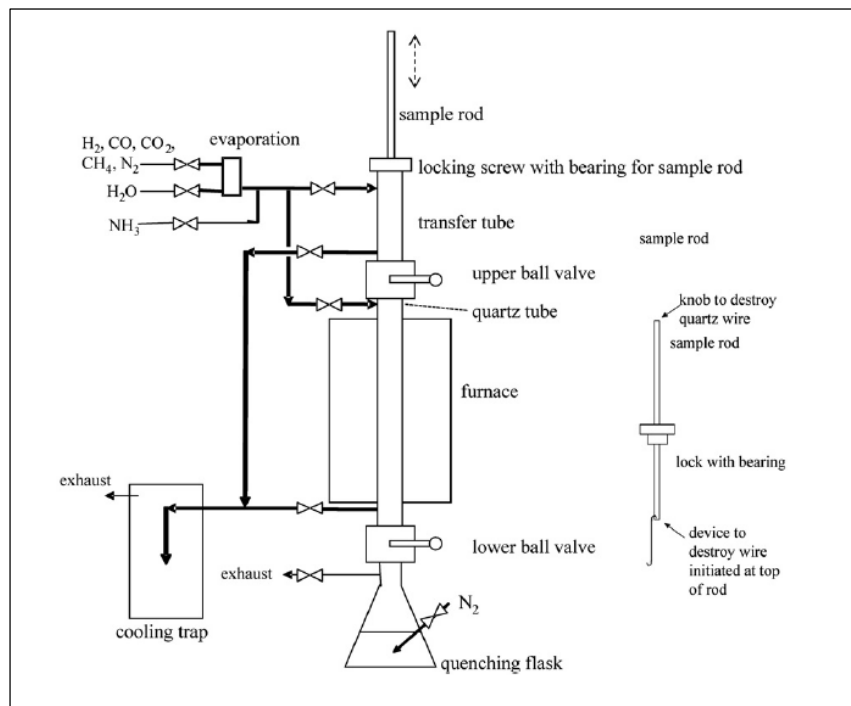
FIGURE 4. Nitrocarburising process

2.3.3 Effects of Gas Nitriding on Aluminium

Nitriding the Aluminium to form nitride layer by nitrogen diffusion is very difficult as it has low solubility of nitrogen due to the formation of dense oxide film that exists on the surface. This layer keeps the dispersion of nitrogen into the aluminium substrate. Furthermore the formation of Al oxide occurs more readily than Aluminium Nitrate because of better affinity between Aluminium and oxygen in comparison with nitrogen. Therefore, the development rate of the aluminium nitride layer is extremely low [7].

Aluminium nitride is known for its high hardness about of HV1400 while normal aluminium only has hardness value around HV96 TO HV98 [8]. After the gas nitriding process was done, the aluminium surface will changed from metallic silver to dark grey or shiny black. The changing on the surface's colour confirmed the formation of aluminium nitride layer on the vicinity of the surface after nitriding. It also exhibits good corrosion resistance, wear resistance, high electrical resistivity and high thermal conductivity which is 320 W/mK. Aluminium nitride has a hexagonal crystal structure and is a covalent bonded material and required lot of energy to break it [7].

CHAPTER 3

METHODOLOGY

3.1 SAMPLE PREPARATION

The sample 4" × 4" × 0.4" aluminium 6061 plate was cut using Electrical Discharge Machine (EDM) into 12 small 2 cm × 2 cm × 2 cm block, two for base reading and another 10 are for after nitriding reading. 5 pieces for 4 hours and the rest for 8 hours gas nitriding process.

FIGURE 5. Sample cut into 12 small pieces

Wire-Cut EDM was used which applied Computer Numerical Control (CNC) to produce specific shape and size on the material. The principle involves a continuous-traveling vertical wire with diameter as small as needle under tension where the path is controlled by the CNC to produce the shape required.
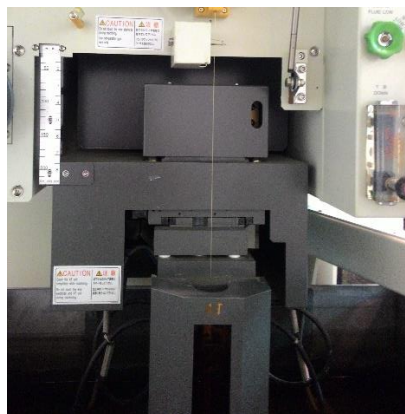
FIGURE 6. Cutting with EDM

All the samples then were grinded and polished to produce clean and smooth surface for even and strong coating. Grinding started with sand paper grit 180, 400, 600, 800 and lastly 1200. Then, the samples were polished using 6 micron diamond paste. All of these process use 300 rpm speed grinder and polisher. The final must be smooth, even and mirror-finish [9].

FIGURE 7. Grinding Process

FIGURE 8. Samples after polishing

3.2 GAS NITRIDING PROCESS

The samples were washed by immersing into 0.5M nitric acid for 3 minutes to remove oxide layer and other contaminants. Then, the samples were place on a tray and put inside the quartz tube furnace. The tube was closed, sealed and the furnace was turned on. The samples were pre-heat for about one and half hour with $10 \text{ cm}^3/\text{min}$ nitrogen gas flow inside the tube until the furnace reach desired temperature. The temperature will remain at 550℃ and samples were heated for 4 hours. The pressure inside the furnace is same as the atmosphere pressure. Once the process was done, the nitrogen gas was turned off when the furnace reaches room temperature. Then, the tube was opened and samples were taken out. All of these steps were same for 8 hours gas nitriding [9].
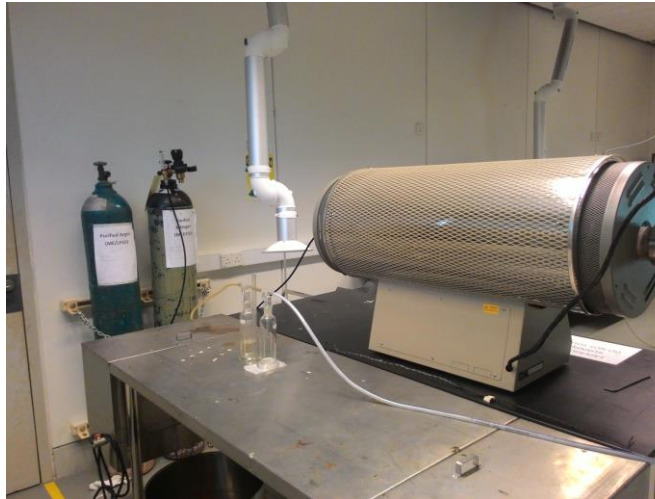
FIGURE 9. Gas nitriding equipment

FIGURE 10. Nitrogen tank

3.3 SECOND SAMPLE PREPARATION

A nitrided sample from each batch was cut into half using abrasive cutter. The half cut was mounted using hot mounting with the inner cross section as the top surface. Then, it was grinded using sand paper grit 180, 400, 600, 800 and 1200 followed by polishing using 6 micron diamond paste until the surface turn into mirror finished.

FIGURE 11. Samples after mounting

After completely cool, the mounted sample was washed using tap water to remove any debris followed by ethanol wash to remove excess water. The samples were left to dry inside the dryer. Then the sample is etched using Keller's etchant by immersing the sample into it for 10 to 30 seconds. This is to make grain boundaries more visible under the microscope. Keller's Etchant is a mixture of 190ml of distilled water, 5ml of nitric acid, 5ml of hydrochloric acid and 5ml of hydrofluoric acid. Finally, the mounted sample was washed again using tap water followed by ethanol and left dried inside the dryer. The etching is successful when the mirror finish turns into matte once it dry.

3.4 MICROSTRUCTURE TEST

Two microstructure test were conducted using Optical Microscope (OM) and Scanning Electron Microscope (SEM). The etched sample was place inside the SEM. Magnification started from 50X until 3000X. Measurement was done using the provided software tools and all images were saved in viewer software. The composition of the sample can be determined using the built in Energy Dispersive Spectroscopy (EDS) in the SEM. After the image can be seen clearly, the thickness of the nitrided layer was measured. The desired area was marked as inspection point for the EDS to determine the element present on it. The EDS software was ran and the result will come out in graph form. Highest peak in graph showed the highest percentage and the dominancy of the element in the marked area. Line scanning also was carried out to access the diffusion profile of nitrogen into the aluminium. The test also was viewed using OM to see the changes in the grain boundary between nitrided and unnitrided samples.
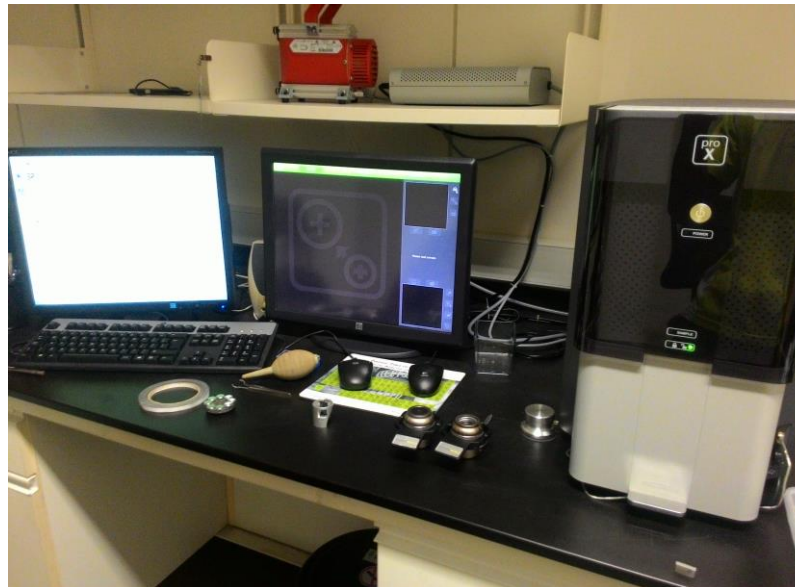
FIGURE 12. SEM equipment

3.5 HARDNESS TEST

The hardness test is conducted using the Rockwell Hardness machine with scale B following the ASTM E 18-2000 Standard. The load is 100kgf load with 1/16" ball indenter. The dial measured the depth of the impression. From the value obtained, HRB reading measured can determine the hardness of the sample.
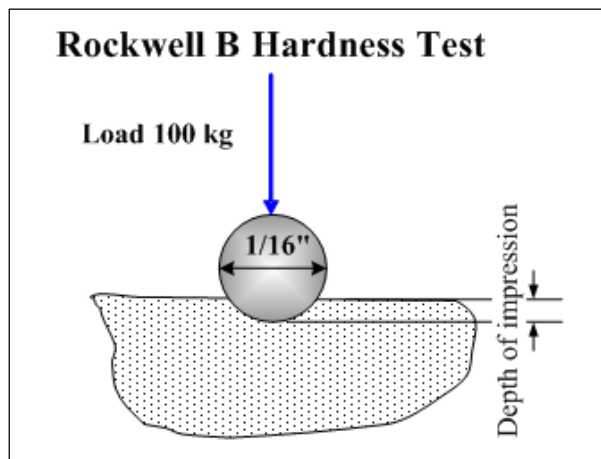
FIGURE 13. Rockwell hardness test scale B

3.6 SCRATCH TEST

Scratch Test was done using the Revetest Scratch Test Machine. The test following the standard ASTM-G171 for scratch hardness of materials using a diamond stylus. The settings are 5N constant load, 5mm track length and 5mm/min speed. The scratcher is 200µm, 120° spherical diamond stylus. Each sample was scratched and the width track was measured at three points [9].

FIGURE 14. CSM Revetest Scratch Test

3.7 PROCESS FLOW CHART

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                    ┌─────────────┐
                    │    START    │
                    └──────┬──────┘
                           ▼
        ┌──────────────────────────────────────────┐
        │   Comprehensive study on the literature review │
        └──────────────────────┬───────────────────┘
                               ▼
        ┌──────────────────────────────────────────┐
        │     Developing the experimental procedure │
        │          1. Gas Nitriding Process         │
        │          2. Microstructure                │
        │          3. Hardness Test                 │
        │          4. Scratch Test                  │
        └──────────────────────┬───────────────────┘
                               ▼
        ┌──────────────────────────────────────────┐
        │          Preparing the sample             │
        │    1. Cutting - according to the standard │
        │    2. Cleaning - grinding and polishing   │
        │    3. Mounting - hot & cold mounting      │
        │    4. Etching - Keller's etchant          │
        └──────────────────────┬───────────────────┘
                               ▼
        ┌──────────────────────────────────────────┐
        │         Carry out the experiment          │
        └──────────────────────┬───────────────────┘
                               ▼
                         ◇ Validation? ◇ ──── NO
                               │
                              YES
                               ▼
        ┌──────────────────────────────────────────┐
        │         Performing sample testing         │
        │          1. Microstructure                │
        │          2. Hardness Test                 │
        │          3. Scratch Test                  │
        └──────────────────────┬───────────────────┘
                               ▼
        ┌──────────────────────────────────────────┐
        │   Final report and technical paper progress │
        └──────────────────────┬───────────────────┘
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                        │     END     │
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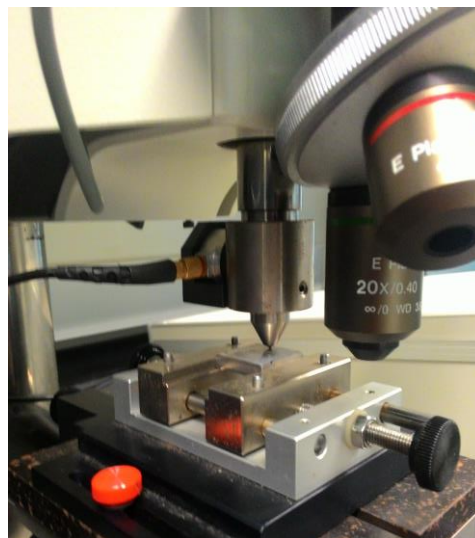
3.8 KEY MILESTONE

TABLE 4. Key milestone of the project

TASK	COMPLETION DATE
Topic selection	1st October 2015
Submission of Extended Proposal	7th November 2015
Proposal Defense	3rd December 2015
Submission of Interim Report	31st December 2015
Material Shopping	31st January 2016
Sample Preparation	7th February 2016
Submission of Progress Report	6th March 2016
Gas Nitriding Process	3rd April 2016
Experimental Testing	10th April 2016
Submission of Interim Report and Technical Paper	17th April 2016
Final Year Project Presentation	24th April 2016

3.9 GANTT CHART

Semester 1

TABLE 5. Gantt chart for first semester

Detail / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Topic	■	■												
Preliminary Research Work			■	■	■	■								
Extended Proposal							■							
Experimental method study								■	■					
Proposal Defense										■	■			
Interim Report Writing											■	■	■	
Interim Report Submission														■

Semester 2

TABLE 6. Gantt chart for second semester

Detail / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Material Shopping	■	■												
Sample Preparation	■	■	■											
Progress Report		■	■	■	■	■	■							
Gas Nitriding Process				■	■	■	■	■	■	■	■			
Experimental Testing								■	■	■	■	■		
Final Report writing											■	■		
Project Presentation													■	■

CHAPTER 4

RESULTS AND DISCUSSION

4.1 RESULTS & DATA GATHERING

4.1.1 Gas Nitriding Results

Diffusing nitrogen gas into the surface of aluminium sample changes the appearance from shiny silvery to dark grey. The change of colour is due to the formation of black layer along the surface. As nitrogen gas was induced during heat treatment on aluminium, the nitrogen gas diffused into the aluminium surface and form aluminium nitride layer. As time increase, more nitrogen gas diffused and form aluminium nitride. This compound deposited on the aluminium surface became thicker and covered the entire surface. Layer become thicker and more visible. Aluminium has low solubility of nitrogen gas due to its FCC structure.

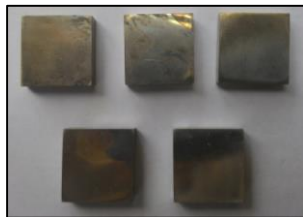FIGURE 15. Unnitrided samples

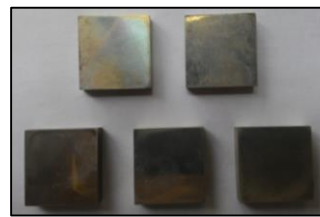FIGURE 16. 4 hours nitrided Samples   FIGURE 17. 8 hours nitrided Samples

FIGURE 15 shows all the unnitrided samples after being grinded and polished. It can be seen that the process produced mirror-like silvery surface on the samples. Whereas on FIGURE 16 and FIGURE 17, the surfaces became dull grey proving nitriding had taken place.

4.1.2 Microstructure Results

From the microstructure test, results obtained from the SEM can be used to investigate the thickness of nitrided layer formed, element composition of the sample and diffusion pattern of the nitrogen from the gas nitriding process conducted.
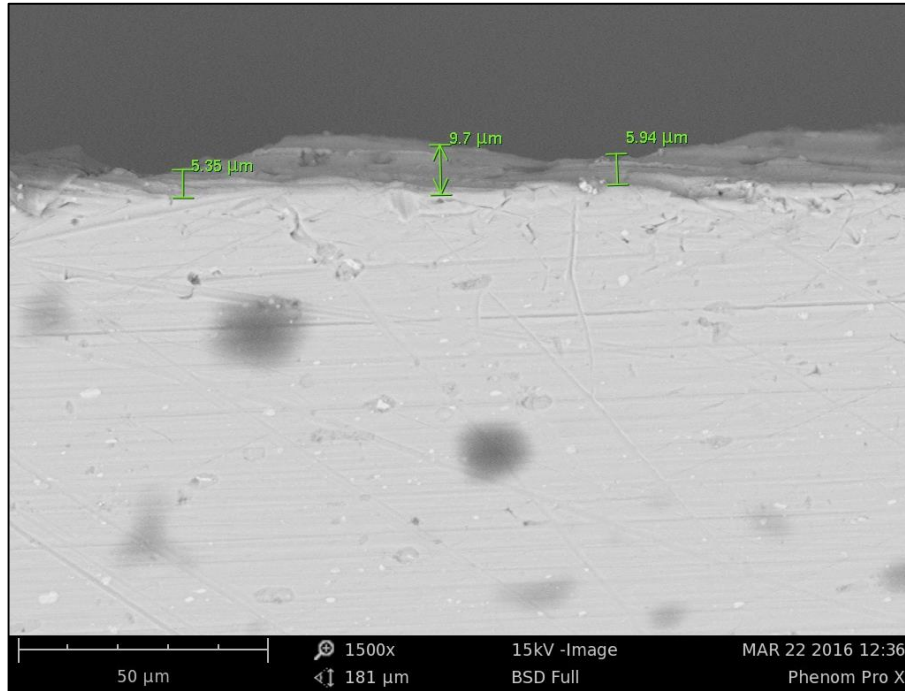

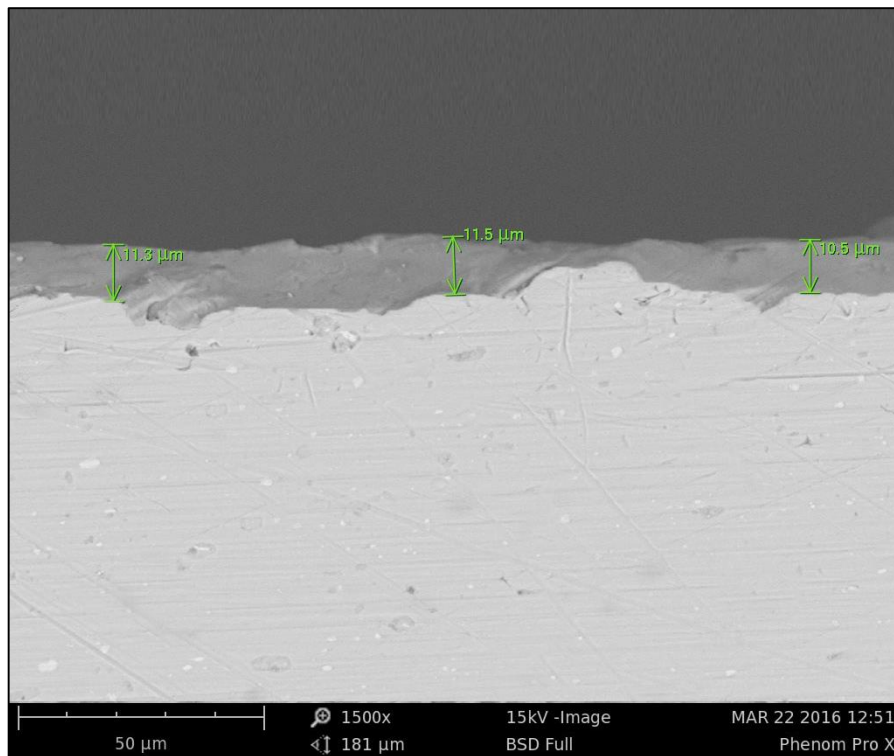FIGURE 18. SEM image of nitrided layer for 4 hours

FIGURE 19. SEM image of nitrided layer for 8 hours

Based on FIGURE 18 and FIGURE 19 above, it clearly can be seen that nitrided layer have formed on the sample surface. Light grey is the aluminium 6061 sample while thin, darker grey indicates the nitrided layer. Three points of measurement for thickness were taken on the layer with the magnification 1500x. Table below shows the thickness readings of the nitrided layer.

TABLE 7.   Thickness of nitrided layer

Thicknes	4 Hours Sample (µm)	8 Hours Sample (µm)
Reading 1	5.4	11.3
Reading 2	9.7	11.5
Reading 3	5.9	10.5
Average Reading	7.0	11.1

Line Scanning was conducted to identify the aluminium and nitrogen diffusion profile on the nitrided layer. Zebra dotted line in FIGURE 20 and FIGURE 21 below show the cross sectional line where the profiles were taken to see the diffusion pattern of nitrogen into aluminium sample surface.
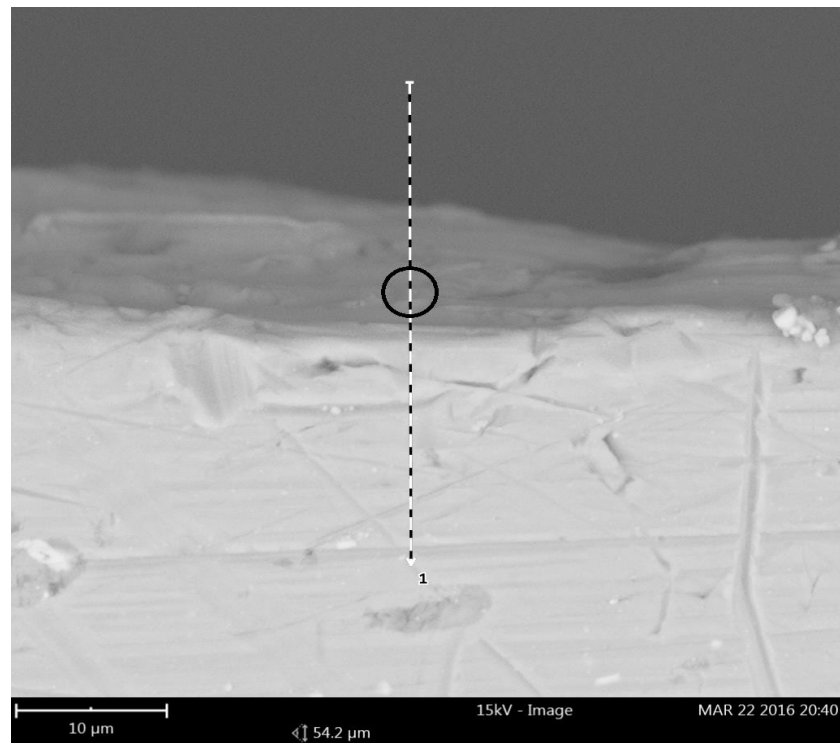

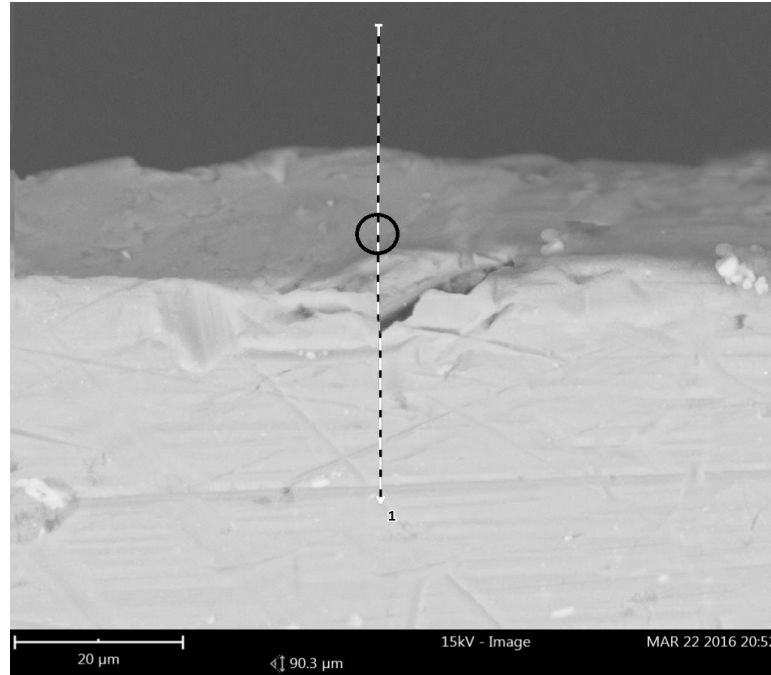FIGURE 20: Cross sectional line of nitrided layer for 4 hours

FIGURE 21. Cross sectional line of nitrided layer for 8 hours

FIGURE 22 and FIGURE 23 below display aluminium and nitrogen diffusion profile from the line scanning where orange line is the aluminium pattern and the blue line is the nitrogen diffusion pattern. On the region near the surface, nitrogen portion is high. However the pattern decreases as it goes to the core showing the amount of diffused nitrogen is lesser on the region near the sample core.
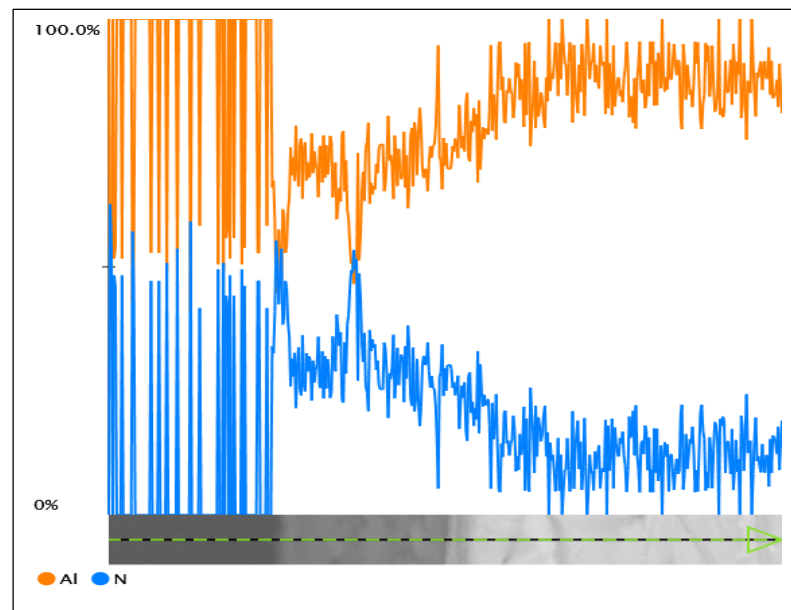

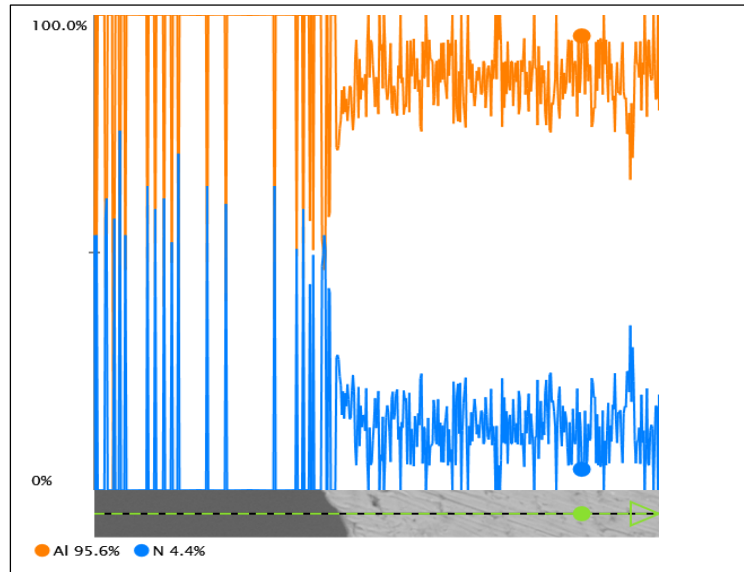FIGURE 22. Line Scanning Profile of nitrided layer for 4 hours

FIGURE 23. Line Scanning Profile of nitrided layer for 8 hours

As for the FIGURE 24 and FIGURE 25 below, the element composition of aluminium and nitrogen were shown from the region marked with black circle in FIGURE 19 and FIGURE 20. The marked region indicates the inspection point for the composition inspection. From the graph, it can be seen that the aluminium shows the highest peak since this element made up the alloy while the nitrogen is present although the amount is small. The presence of this two elements proved that the dark grey layer is aluminium nitride layer.
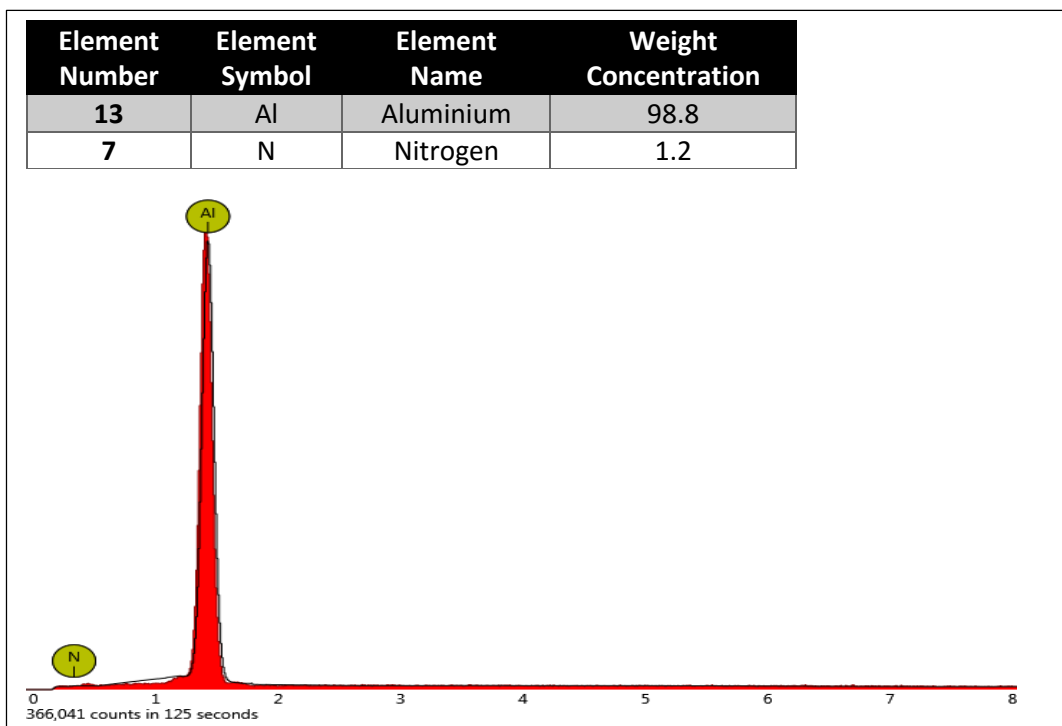
Element Number	Element Symbol	Element Name	Weight Concentration
13	Al	Aluminium	98.8
7	N	Nitrogen	1.2

FIGURE 24. Element composition on 4 hours nitrided

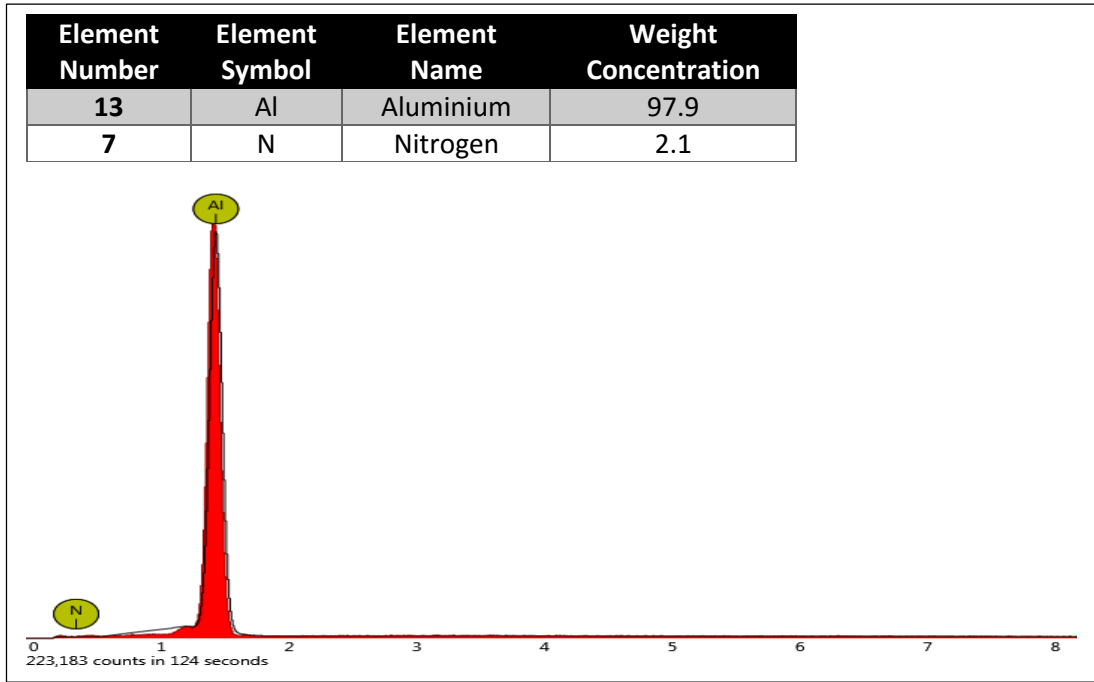| Element Number	Element Symbol	Element Name	Weight Concentration
13	Al	Aluminium	97.9
7	N	Nitrogen	2.1

223,183 counts in 124 seconds

FIGURE 25. Element composition on 8 hours nitrided

4.1.3 Hardness Results

Hardness is defined as resistance of material to plastic deformation caused by indentation. Hardness Rockwell scale B is used to determine the hardness of unnitrided and nitrided aluminium. The load in this scale is 100kgf with 1/16" ball indenter. Table below shows the hardness value for each samples.

TABLE 8. HRB Reading

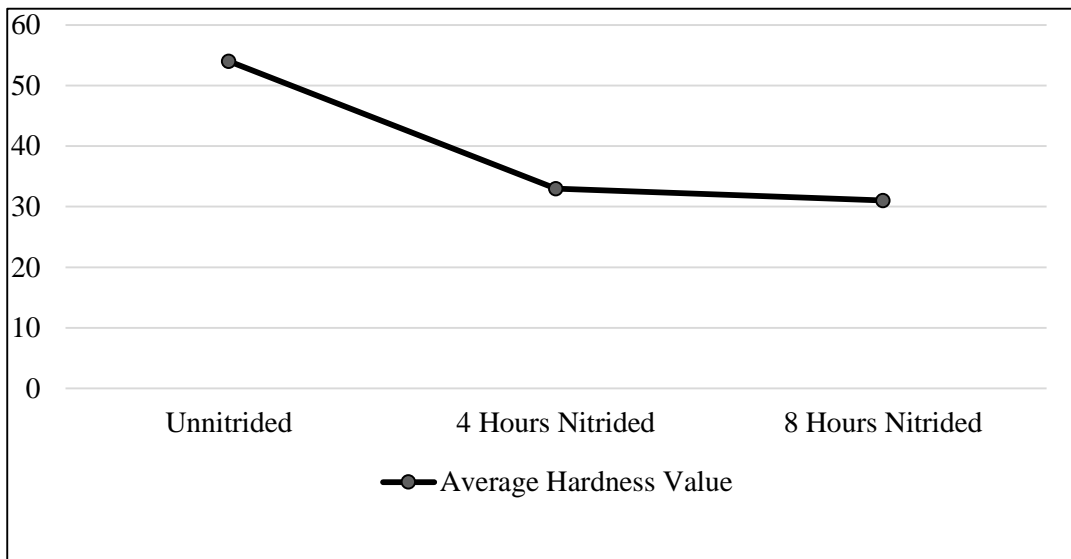| Aluminium Sample	Value 1	Value 2	Value 3	Average Value
Unnitrided	54	54	53	54
4 Hours Nitrided	34	33	32	33
8 Hours Nitrided	32	31	30	31

FIGURE 26. Average HRB Value with respect to exposure time of nitriding

From Table 8 and FIGURE 26 above, hardness value from Rockwell test of both 4 hours nitrided and 8 hours nitrided samples are lower than the unnitrided sample. Hardness obtained for 4 hours nitriding is HRB33 whereas 8 hours nitriding is HRB31. These values showing decrement of 38% for 4 hours nitriding and 42% for 8 hours nitriding compared to the unnitrided hardness which has the value of HRB54.

4.1.4 Scratch Test Results

The test was conducted to measure the resistance of material surface to permanent deformation under the straight line motion of 200μm diamond stylus spherical scratcher with 5N constant load, 5mm track length and 5mm/min speed.
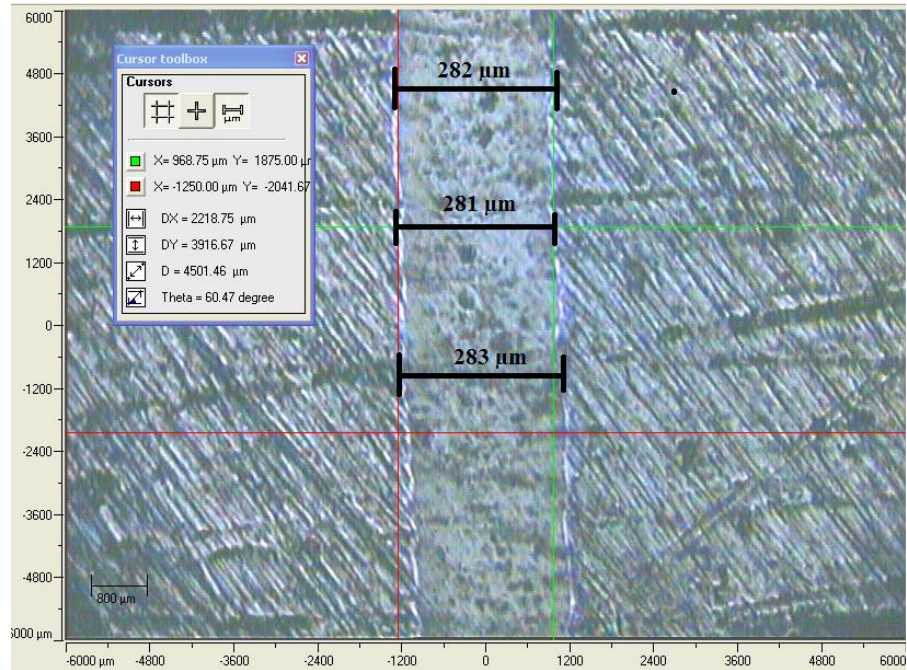

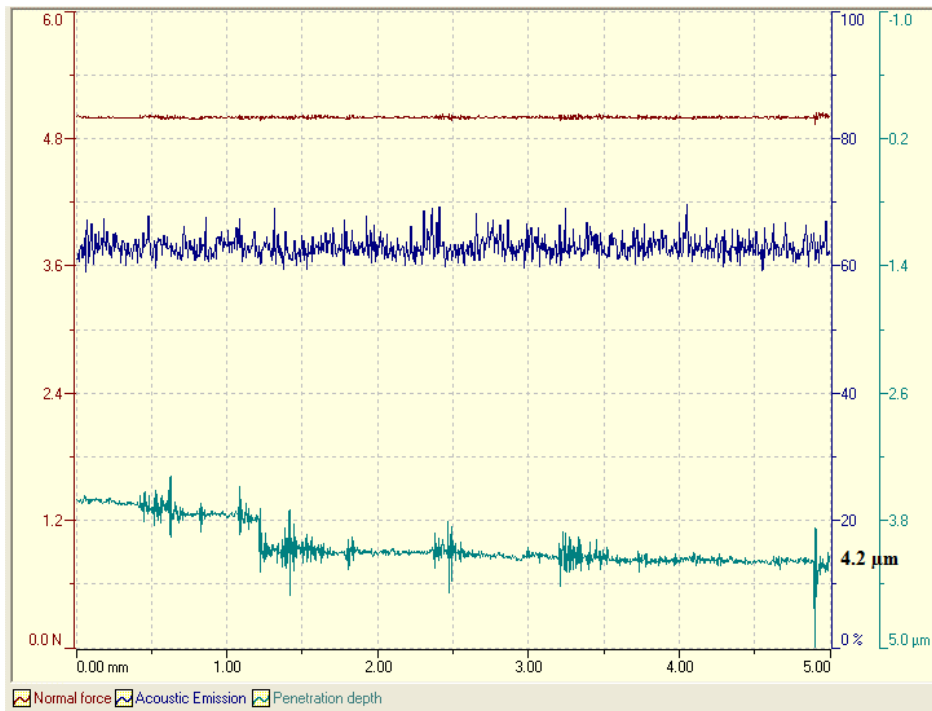FIGURE 27. Scratch track on unnitrided sample

FIGURE 28. Penetration depth on unnitrided sample

FIGURE 27 shows the scratch track on unnitrided sample. The width of the track was measured at three points and the average width track for this sample is 282 µm. FIGURE 28 shows the graph for penetration depth on unitrided sample which is 4.2 µm. The red line represent the constant load of 5N that was applied along the process while the blue line is the acoustic emission indicating the sound of surface fractured.
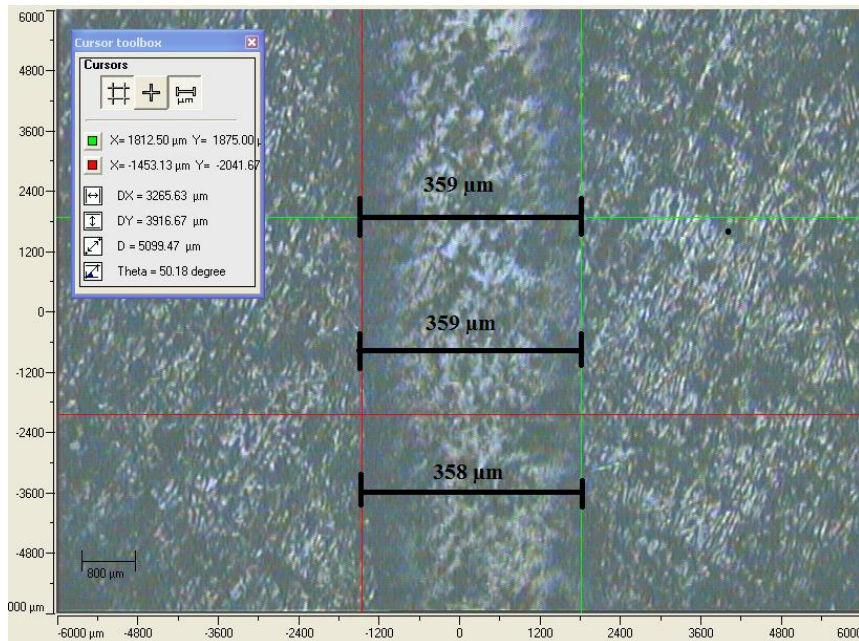

FIGURE 29. Scratch track on 4 hours nitrided sample

FIGURE 30. Penetration depth on 4 hours nitrided sample

FIGURE 29 shows the scratch track on 4 hours nitrided sample. It can be observed that the width track is bigger compared to unnitrided sample with the average of 359 μm. FIGURE 30 shows the graph for penetration depth for the 4 hours sample's scratch track. The penetration depth on this sample is 9.5 μm which is 55% deeper than the unnitrided sample.
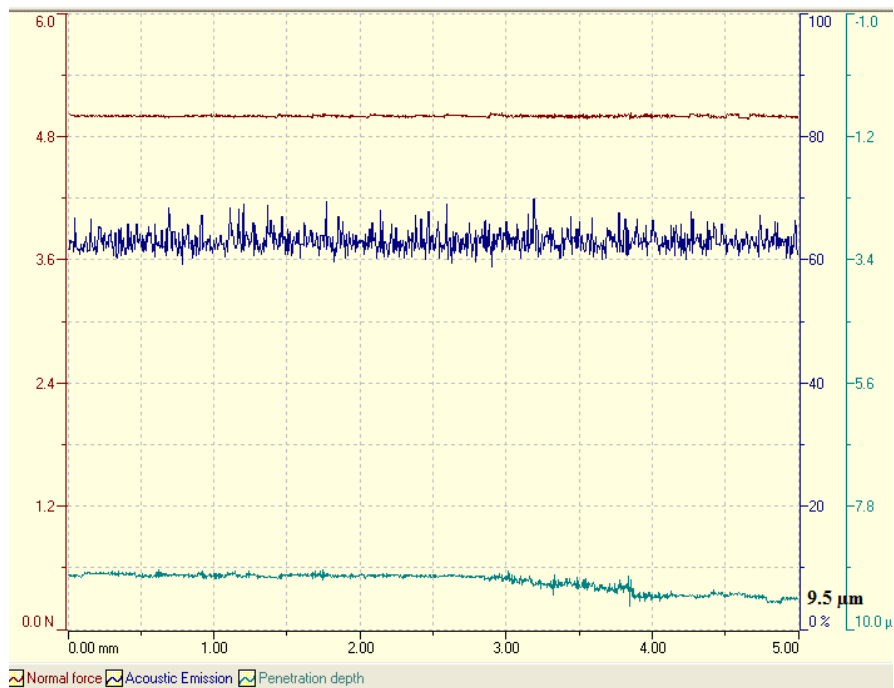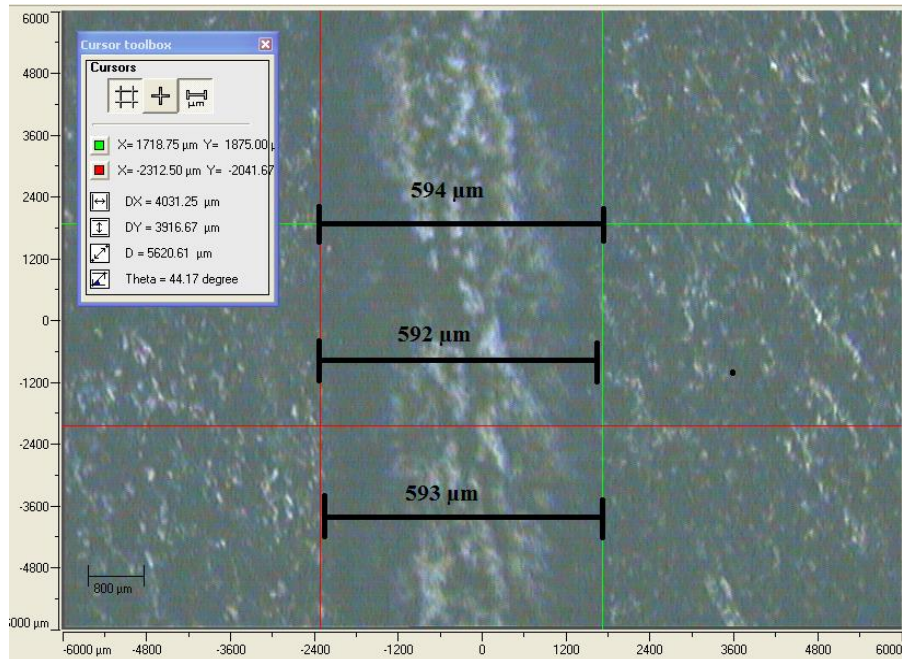

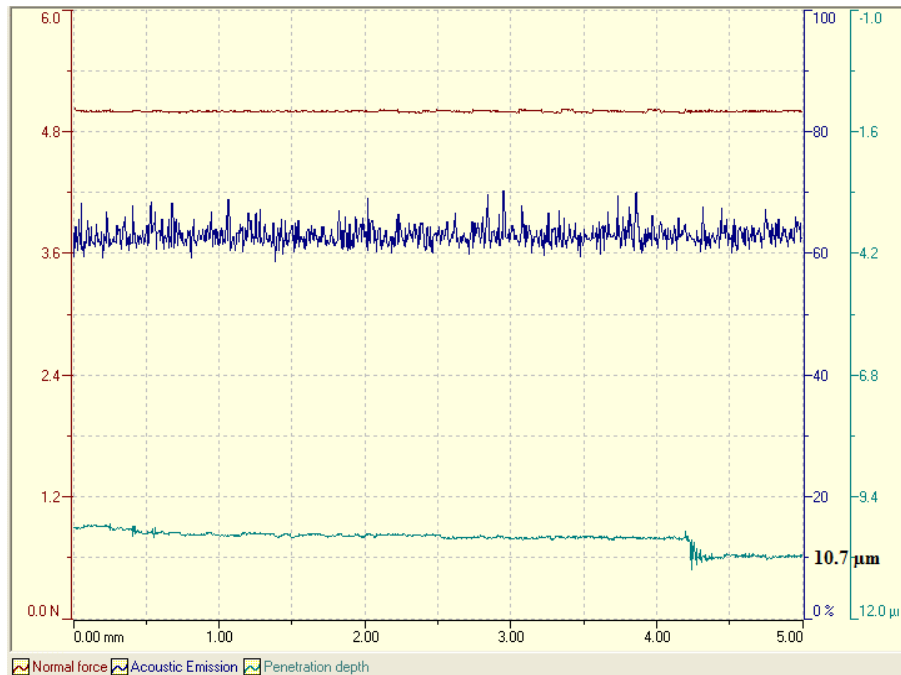FIGURE 31. Scratch track on 8 hours nitrided sample

FIGURE 32. Penetration depth on 8 hours nitrided sample

FIGURE 31 shows the scratch track on 8 hours nitrided sample. The width track is the biggest compared to unnitrided and 4 hours nitrided samples with the average of 593 μm. FIGURE 32 shows the graph for penetration depth for the 8 hours nitrided sample. The penetration depth is 10.7 μm which is 61% deeper than the unnitrided sample. Table 9 below shows the summary for the scratch tracks of all the three samples.

TABLE 9. Width reading of the scratch tracks

Aluminium Sample	Width 1 (μm)	Width 2 (μm)	Width 3 (μm)	Average Width (μm)
Unnitrided	282	281	283	282
4 Hours Nitrided	359	359	358	359
8 Hours Nitrided	594	592	593	593

The average width of the scratch tracks measured is used to calculate scratch hardness number, HS_p from the following formula:

$$HS_p = \frac{8P}{\pi w^2}$$

Where P is the normal force applied and w is the average width track in meter.

TABLE 10. Scratch Hardness Number

Sample	HS_p (GPa)
Unnitrided	0.16
4 Hours Nitrided	0.09
8 Hours Nitrided	0.03

Scratch hardness number cannot determine the exact wear rate value for the material. However HS_p number can still be used to justify the wear resistance of compared samples. High HS_p number means the sample has high wear resistance while low HS_p number means low wear resistance held by the sample.

4.2 DISCUSSION AND DATA ANALYSIS

From the microstructure results obtained, aluminium nitride, AlN layer formed proved the gas nitriding process was successful. Nitrogen gas, N_2 have diffused into the aluminium surface as seen through SEM. Nevertheless, data gathering from the mechanical testing showed the contrary results. Instead of hardening the surface of aluminium 6061, it seem gas nitriding process conducted is softening the samples. With reference to the literature, the hardness should be increase when nitriding with ammonia gas, NH_3. However, for my project, only nitrogen gas N_2 was applied.

Aluminium alloy has strong oxide layer, Al_2O_3 which prevent the N_2 gas from diffusing into the surface metal. Injecting NH_3 gas during the nitriding process will break down the ammonia molecule into the nitrogen and hydrogen atom when reacted with aluminium under heating. Thus the surface layer of Al with the formality of aluminium oxide Al_2O_3 prevented the diffusion of NH_3 into the aluminium alloy. When Al exposed to the atmosphere, Al_2O_3 was easily formed due to the oxidation. When using NH_3, the decomposition will reduce the oxide layer as the hydrogen will react with oxide to form water, hence diffusion of N_2 will take place as in the equation below:

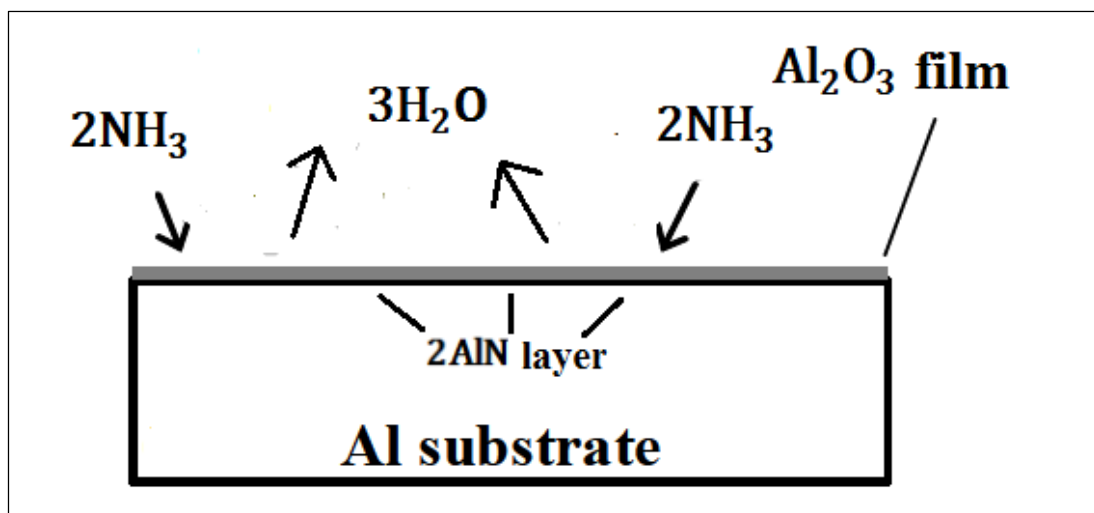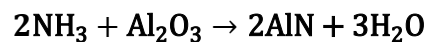$$2NH_3 + Al_2O_3 \rightarrow 2AlN + 3H_2O$$

FIGURE 33. Decomposition of ammonia in gas nitriding

Lower scratch hardness number, HS_p and Rockwell hardness number, HRB signify the lower hardness possess by the aluminium alloy after undergo the gas nitriding process. This is due to ageing process that occurred naturally after prolong heating at 550℃ for 4 hours and 8 hours left to cool in ambient temperature. Therefore from the microstructure analysis of using OM, the formation of grain growth can be analyzed. The grain growth affects the decrease in hardness. Prolong heating increase the diffusion rate of atom, in this case nitrogen atom which created the nitrided layer. Nonetheless, this resulted in coarsening of the grains where the small grains dissolute and the big grains grew larger [13].

FIGURE 34 below shows the microstructures of the grain boundary for unitrided sample, 4 hours nitrided sample and 8 hours nitrided sample viewed using Optical Microscope. It can be observed that grain growth have occurred in nitriding samples as the grains grew larger than the unnitrided sample. Darker grains indicates the nitrogen have diffused into the grains structure.
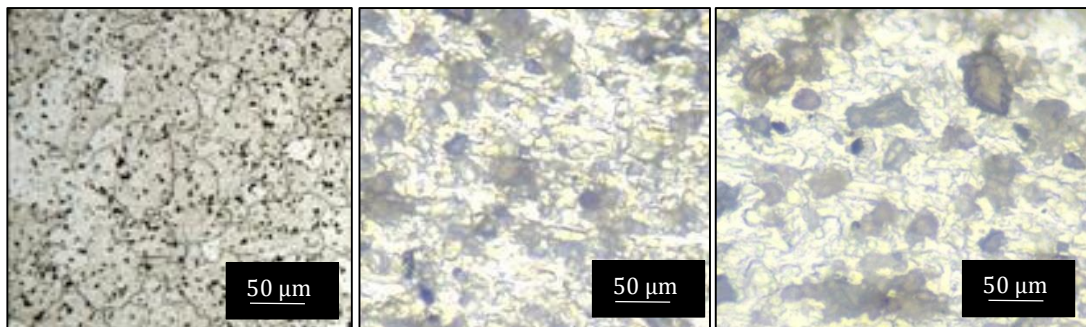
FIGURE 34. Microstructures in all three samples: (a) unnitrided,
(b) 4 hours nitrided, (c) 8 hours nitrided

This unexpected ageing causing the grains growth to occur which consequence to reducing the hardness but increasing ductility properties, making it more workable such as bending. It is found that this process worked similar to the annealing process where there is action of heating and cooling of material to increase the ductility while decreasing the hardness to make it easier to be workable for material fabrication.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

All Objectives were achieved. The gas nitriding was successfully conducted on aluminium 6061 at 550℃ for 4 hours and 8 hours. Observation on the sample surface changes from shiny silvery to dark grey indicates the nitride layer have formed. When viewed using SEM, the microstructure test confirmed the formation of aluminium nitride layer with the thickness of 7 µm in 4 hours nitrided sample and 11.1 µm in 8 hours nitrided sample. EDS will used X-ray emission to sort out the elements in a material and give a result in graph form. Aluminium nitride presence is indicated by high peak of aluminium and nitrogen when inspected using EDS. This graph will resulted in aluminium as the highest peak followed by nitrogen.

The HRB number from the Rockwell hardness test and scratch hardness number, HS_p for both nitrided samples giving unexpected results as the average HRB and HS_p for 4 hours nitrided are 33 and 0.09 GPa whereas HRB and HS_p for 8 hours are 31 and 0.03 GPa respectively. These values are lower than the unnitrided sample which has the hardness value of HRB54 and HS_p of 0.16 GPa. Lower hardness number in both Rockwell test and scratch test signify the change of mechanical properties at which hardness is reduced while ductility has increased. This explained by the occurrence of natural ageing process which resulted in grain growth in both nitrided samples as seen through Optical Microscope. This discovery could be the alternative method for annealing in reducing the hardness with increased ductility for working with material fabrication.

5.2 RECOMMENDATION

Sample preparation is very important before conducting the gas nitridning process and microstructure test. Sample must be ensured to have even, smooth and mirror finish from grinding and polishing process and conduct proper etching before analyse with OM or SEM so as to have clear view on the microstructure.

Ammonia gas, NH_3 is more suitable to be used instead of pure nitrogen, N_2 for gas nitriding on aluminium alloy since Aluminium has strong passive oxide film, Al_2O_3. The decomposition of ammonia to hydrogen and nitrogen will make the diffusion of nitrogen into the surface to take place easier as the hydrogen will react with the oxide on the aluminium and formed water. Yet, ammonia is an explosive gas which need to be handled carefully.

Since the nitrided layer formed is in micron scale, Micro Vickers Hardness is more compatible choice to carry out the hardness testing. This is because Micro Vickers is measured as HV number which indicates values in micron scale. Compared to Rockwell Hardness B-scale, Micro Vickers Testing can determine the hardness of the nitrided layer formed without deforming of damaging the sample surface.

Wear resistance indicates by the wear rate number must be determined using the correct wear test. This can be done by using ASTM standard such as Pin-On-Disk Test (ASTM G99) or conducting Wet Sand/Rubber Wheel Abrasion Tests (G195).

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