AN EVALUATION OF NI-ALUMINA COMPOSITE ELECTROPLATING ON MILD STEEL

NORLIDA BINTI JAMIL

BACHELOR OF ENGINEERING
UNIVERSITI MALAYSIA PAHANG

2010

NORLIDA BACHELOR OF MECHANICAL ENGINEERING 2010 UMP

UNIVERSITI MALAYSIA PAHANG
FACULTY OF MECHANICAL ENGINEERING

I certify that the project entitled *“An Evaluation Of Ni – Alumina Composite Electroplating On Mild Steel”* is written by *Norlida Binti Jamil*. I have examined the final copy of this project and in our opinion; it is fully adequate in terms of scope and quality for the award of the degree of Bachelor of Engineering. I herewith recommend that it be accepted in partial fulfilment of the requirements for the degree of Bachelor of Mechanical Engineering

(DR. MD. MUSTAFIZUR RAHMAN)
Examiner Signature

EVALUATION OF Ni – ALUMINA COMPOSITE ELECTROPLATING ON MILD STEEL

NORLIDA BINTI JAMIL

Report submitted in partial fulfillment of the requirements
For the award of the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering
UNIVERSITI MALAYSIA PAHANG

NOVEMBER 2010

SUPERVISOR’S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature
Name of Supervisor: DAYANGKU NOORFAZIDAH AWANG SHRI
Position: LECTURER FACULTY OF MECHANICAL ENGINEERING
Date:

STUDENT'S DECLARATION

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

Signature
Name: NORLIDA BINTI JAMIL
ID Number: MA07065
Date:

Dedicated to my beloved parents

ACKNOWLEDGEMENTS

First I would like to express my grateful to ALLAH S.W.T. as for the blessing given that I can finish my project.

First, I have engaged with many people in helping me completing this project. I wish to express my sincere appreciation to my main thesis supervisor Mdm Dayangku Noorfazidah Awng Shri, for his germinal ideas, continuous encouragement, invaluable guidance, advices and motivation. Without his continued support and interest, this thesis would not have been the same as presented here.

The next category people who help me to grow further and influence my project are my previous panels, Dr Sugeng Ariyono, Mr. Mohd Firdaus Hassan, Mr. Nasrul Azuan Alang that advice me on how to complete this project greatly. I would like to acknowledge their comments and suggestions, which was crucial for the successful completion of this study. Also to my colleagues who always help me in order to finish this project. I would like to express my gratitude especially to all FKM laboratory instructors and for them help and advices. I appreciate very much to them because of their excellent co-operation, inspirations, idea and support information given during this study.

Last but not least I acknowledge without endless love and relentless support from my family, I would not have been here. My father, Jamil bin Ajang, my mother, Zalinah Bte Mohd Zin, all my sisters and brother that always support, sacrifice, patience, understanding that were inevitable to make this work possible, motivation and encourage me to success. I cannot find the appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals.

ABSTRACT

This thesis deals with an evaluation of Ni- Alumina composite electroplating on mild steel. Electroplating process has been carried out by utilizing the Watt’s Bath with Alumina oxides powder. Weight changes in both electrode shows that weight loss of anode and weight of deposit on cathode are proportional to plating time and voltage supplied. There are three parameters involve in this project that are Alumina concentration, plating time, and voltage supplied. Mild steel will be the anode and connected to positive terminal. Meanwhile, nickel plate as anode will be connected to negative terminal. Dimension of sample in this project is 80 mm x 30 mm x 1.5 mm. Nickel ion is reduced to form or plate a surface on mild steel. The electroplating bath contains the metal ion to be reduce as well as Alumina oxides to support the flow of electrons. While reduction of the metal occurs at cathode, oxidation is simultaneously occurring at anode. After that, microstructural analysis was done to observe the thickness of coating and its surface structure. Lastly, all the samples were undergoes Vickers hardness test. In this project, it can be conclude that result shows increase of Alumina concentration, plating time, and voltage supplied also increase the thickness of coating.

ABSTRAK

Tesis ini berkaitan dengan penilaian penyaduran Ni-Alumina komposit pada besi tulen. Proses elektroplating dilakukan dengan mengaplikasikan Mandi Watt dengan serbuk oksida Alumina. pPrubahan berat dalam kedua-dua elektrod menunjukkan bahawa kehilangan berat di anod dan berat bertambah di katod setanding dengan tempoh penyaduran dan voltan disediakan. Ada tiga parameter yang terlibat dalam projek iaitu kepekatan Alumina, tempoh sadur, dan voltan disediakan. baja ringan akan menjadi anod dan dihubungkan ke terminal positif. Sementara itu, plat nikel sebagai anoda akan disambungkan ke terminal negatif. Dimensi sampel dalam projek ini adalah 80 mm x 30 mm x 1.5 mm. Ion nikel berkurang untuk membentuk atau plat permukaan pada besi tulen. Cecair penyaduran mengandungi ion logam dapat mengurangkan serta Alumina oksida untuk menyokong aliran elektron. Sementara pengurangan logam terjadi pada katod, pengoksidaan secara serentak terjadi pada anod. Setelah itu, analisa mikrostruktur dilakukan untuk melihat ketebalan lapisan dan struktur permukaannya. Akhir sekali, semua sampel mengalami uji kekerasan Vickers. Dalam projek ini, dapat disimpulkan hasil tersebut menunjukkan peningkatan kepekatan Alumina, tempoh sadur, dan voltan yang disediakan juga meningkatkan ketebalan lapisan.

TABLE OF CONTENTS

CHAPTER 1 INTRODUCTION

CHAPTER 2 LITERATURE REVIEW

CHAPTER 3 METHODOLOGY

CHAPTER 4 RESULTS AND DISCUSSION

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

-	Negative
+	Positive
μ	Micron
%	Percentage
°	Angle / Degree
θ	Degree

LIST OF ABBREVIATIONS

AISI	American Iron and Steel Institute
ASTM	American Society for Testing And Material
SEM	Scanning Electron Microscope
UNS	Unified Numbering System
PSM	Projek Sarjana Muda
PVD	Physical Vapor Deposition
SHS	Self-Propagating High Temperature Synthesis
CED	Conventional Electro Deposition
et al	And Others

APPENDIX A1

Table 6.1: Weight Loss of Anode at Different Parameter

SET	Al_20_3 concentration (g/l)	Weight loss of anode (g)
1	5	0.021
2	10	0.026
3	15	0.027

SET	Times (mins)	Weight loss of anode (g)
1	60	0.989
2	90	0.593
3	120	0.031

SET	Voltage (V)	Weight loss of anode (g)
1	1.5	0.413
2	2.5	1.011
3	3.0	1.335

APPENDIX A2

Table 6.2: Weight Loss of Cathode at Different Parameter

SET	Al_20_3 concentration (g/l)	Deposit weight on cathode (g)
1	5	0.014
2	10	0.095
3	15	0.121

SET	Times (mins)	Deposit weight on cathode (g)
1	60	1.119
2	90	1.140
3	120	0.003

SET	Voltage (V)	Deposit weight on cathode (g)
1	1.5	0.665
2	2.5	0.727
3	3.0	0.925

APPENDIX B

Table 6.3: Set of Parameters

Table 6.3(a): At Parameter of Alumina Concentration

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	2.5
2	10	60	2.5
3	15	60	2.5

Table 6.3(b): At Parameter of Plating Time

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	2.5
2	10	90	2.5
3	15	120	2.5

Table 6.3(c): At Parameter of Alumina Concentration

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	1.5
2	10	60	2.5
3	15	60	3.0

CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Electroplating is a method of covering objects with a thin layer of metal. Discovered by Micheal Faraday in the 1830's it has contributes to development and application in many areas of industry besides our daily lives in many ways.

In this project, the electroplating process has been carried out to observe the dissolution of cast nickel anode together with alumina powder and the deposition of metallic nickel alumina on the mild steel cathode. Besides, this experiment will establish the effect of various electroplating parameters of nickel alumina deposition.

During electroplating, the Nickel Sulphate deposited into Ni^{2+} ion and SO_4^{2-} ion according to this chemical reaction:

$$NiSO_4^{2+} \rightarrow Ni^{2+} + SO_4^{2-} \tag{1.1}$$

While the water will also dissociates into:

$$H_2\mathrm{O} \rightarrow H^{+} + OH^{-} \tag{1.2}$$

The numbers of positive and negative ion charges exists equally in the solution. The object to be plated (cathode) is connected to the negative (-) side of the battery, giving it the negative charge, and the plating metal (anode) is connected to the positive (+) side of the battery, giving it a positive charge. Positive ions flow the anode toward the object being plated, through the plating solution (electrolyte), and finally deposited onto the surface of the object.

In this experiment, since the anode is the same metal of the solution, the anode will deposited to form Ni^{2+} ions and form a positively charged outer under applied condition. On the other hand, at the cathode, its outer layer performs a negatively charged layer under the same applied condition after receiving electron from the current supply. Furthermore, this will attracts Ni^{2+} ions in the solution then replaced by the Ni^{2+} ions from the Nickel anode. So, the concentration of Ni^{2+} ions in the solution is remains constant till the anode fully deposited.

1.2 PROJECT OBJECTIVES

In order to accomplish the objectives of performing the experiment, four electroplating parameters: voltage, plating time and Alumina concentration, had been vary-related into experiments.

a) To determine the effect of various relations of voltage and plating time to the weight of nickel deposit in a constant temperature condition.
b) To determine the effect of different concentration of Alumina to the weight of nickel deposit in constant voltage and plating time.
c) To determine the coating properties such as the hardness and its microstructure by undergoes coating analysis.

1.3 SCOPE OF PROJECT

a. In order to accomplish the set objectives, the following scope of works has been drawn:
b. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference voltage.
c. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference plating time.

d. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference concentration of Alumina.
e. Microstructure analysis on coating.
f. To perform Vickers Hardness Test on coating.

1.4 REPORT ORGANISATION

Based on the project process flow chart, the experiments were started with identify the problems and objectives which related to my title given: Evaluation of Ni-Alumina Composite Electroplating on Mild Steel. Besides, all information was gathered from journals, books, magazines, and websites to finish literature review's part.

When it comes to methodology part, journal has been used as main reference. All information from journal was gathered and understands by discussing with supervisor and technical staff to complete it one by one step procedure. So, analysis parameters were confirmed. They are alumina concentration, time of plating, and plating voltage. The parameters will be various at every different experiment. After that, the analysis of coating test was confirmed, which are its thickness, microstructure observation, and Vickers test. The microstructure observation was done by using inverted microscope and optical microscope and last but not least, hardness Vickers tester was used to undergoes hardness test on coating.

Then, all equipment was prepared. This includes all hardware such as beaker, mild steel and nickel plate besides chemicals to make electroplating solution. Experiment was setting up after done with its preparation. Each of material was confirmed available to be use. The final analysis of experiment was done to make sure everything under good conditions. It will be proceed if the analysis result were okay and if it's not, then it will go back again to start level and re-do from the beginning.

The data from the experiment were collected. This includes the result of plating at before and after conditions. The collected data were structured in a table. Discussions were made after experiment and analysis on coating done successfully. After that, conclusion will be made together with some recommendations.

Last but not least, it will be project presentation. All the material and data were gathered to do slide presentation and finish the report. It follows by submission of report.

1.5 PROJECT FLOW DIAGRAM

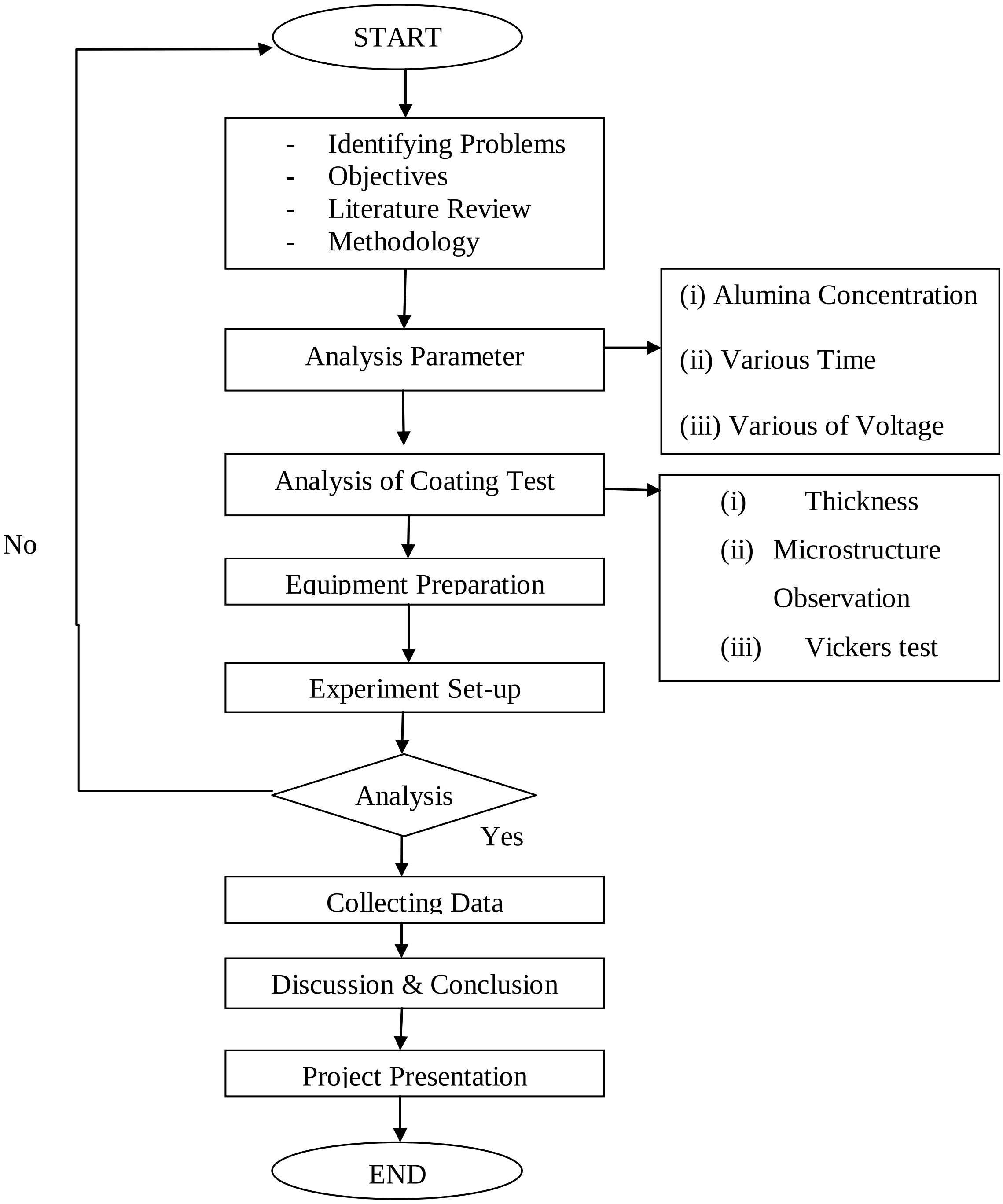


Figure 1.1: Project process flow chart

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Electroplating is a plating process that uses electrical current to reduce cations of a desired material from a solution and coat a conductive object with a thin layer of a material, such as a metal. It is a multidiscipline of engineering, mechanical, and electrical, in co-ordination with applied chemistry. Present day electroplating has become a well-established branch of metal finishing.

Adding ceramics and intermetallics into regular metal coatings is becoming a common method to improve surface properties. Electro-deposition is well established method for fabrication of such materials because of advantages in uniform depositions on complexly shaped substrates, low cost, good reproducibility and the reduction of waste.

2.2 HISTORY Of ELECTROPLATING

2.2.1 Samples of Previous Electroplating Research

The development of electroplating has been described in detail by Hunt (1973), Kramer Weiner and Fett (1959) and Pavlova (1963). An aqueous solution of metal salts and a source of electricity are the chief requirements for electroplating, so the early efforts were concentrated on the search for a good electrical source (Wiley,1963).

The commonly accepted opinion is that the first man to deposit metal from its chemical compounds was Professor G.B. Beccaria in 1722, back in the days of the phlogiston theory (phlogiston being something which was thought to be needed to generate metals). The energy, or electricity, was produced by a Layden jar, which acted as a capacitor, and provided a means of storing electricity, but could only supply weak, short pulses of current (Arthur Kenneth Graham, 1971)

In 1971, Luigi Galvani of the old and famous university of Bologna discovered that muscle tissue of frog reacts to pulses of electric current. He observed that a frog's muscle suspended on a copper ring convulses on contact with iron. The matter generating these convulsions was named fluidum and mistakenly thought to be a property of the animal tissue itself, but identical with electricity. The effect was named galvanismus after Galvani. The story of the Elkington cousins, George Richards and Henry Elkington, is very complicated and even L.B. Hunt had problems unraveling it. G.R Elkington referred to himself as a 'Gilt Toy Maker'; he and Henry made small articles like military badges, buttons, and snuff boxes. He was interested in replacing the dangerous amalgamation process for gilding with something less poisonous and easier to handle. The earlier patents of the Elkington mostly covered immersion gilding processes but on March 25th, 1840 the cousins filed a patent (B.P. 8447) 'Improvements in Metals', detailing silver and gold solutions in connection with the application of current. The source of current was very important:' …a solution of chloride of sodium…into this a cylinder of zinc is immersed, with a wire of copper…', clearly it is an electrochemical battery. This patent claim gave Werner von Siemens an opening. He sent his younger brother Wilhelm to England to negotiate his own patent claims with the Elkingtons. This ultimately led to Wilhelm becoming famous as Sir William Siemens, industrialist and scientist, and honored with a memorial window in Westminster Cathedral (Nasser Kanani, 2004).

The work of the scientist Michael Faraday in the 1830s, in generating electricity by elelctromagnetic dynamo was also initially unappreciated commercially. At a meeting in 1850 of the British association, the Elkington cousins still expressed a preference for batteries over the dynamo, even though they had the patents for dynamo since 1846. However, their statements were possibly mere polemic as large Magneto macj=hines were installed at that time in their Newhall Street, Birmingham works, with a capacity to deposit up to 50 ounces of silver in an hour (Mogerman,1974).

Alloy plating where two (or more) metals are simultaneously deposited is increasingly used for decorative finishes. For instance steel can be plated with brass by using an electrolyte of copper and zinc salts in the ratio 60:40 dissolved in a solution of sodium cyanide. Similarly other alloys such as bronze, speculum, lead-tin, etc, can be produced as platings (H.Silman, 1978).

2.2.2 Process Of Ni-Alumina Electroplating

Electroplating is an electrochemical process by which metal is deposited on a substrate by passing a current through the bath. In the Ni- Alumina electroplating process, the anode, nickel plate is connected to the positive terminal of the power supply. On the other hand, the cathode, which mild steel is connected to the negative terminal. The anode or nickel plate will be the source of the material to be deposited and the cathode which is the substrate with negatively charged electrode is to be coated. Besides, the dissolution of cast nickel anode together with alumina powder will result the deposition of metallic nickel alumina on the mild steel cathode.

2.2.3 Nano-Composite Coating

Recent years, nano-composite coating has got much attention for its excellent mechanical properties such as wear resistance, corrosion resistance and lubricant. However, it has been not easy to elevate the content of nano-particles by traditional way up to now. It is well known that the properties of the composite coatings are heavily dependent upon the degree of particle incorporation in the deposit. Higher incorporation percentages and more uniform distribution of inert particles in the metal matrix lead to the improvement of the mechanical, tribological, anti-corrosion and anti-oxidation properties of coatings. In the last three decades, micro and nano-sized inorganic inert particles with metal or alloy such as SiC,ZrO_2, Al_2O_3, Si_3N_4, TiO_2, CeO_2, nano-diamond, carbon nanotubes (CTN), etc.

Previous experiment, effect of micro and nano sized particles such as SiC and Al_2O_3, on different material properties were investigated. Other ceramics like WC, CeO_2 and TiO_2 were successfully electrodeposited and examined too. Electro-deposition of nickel aluminum composite coatings were accomplished in Watts bath based of following chemistry: 300g/dm^3 $NiSO_4.7H_2O$, 45g/dm^3 $NiCl_2.6H_2O$, 40g/dm^3 H_3BO_3, with addition of 15 g/l $Al_2(SO4)_3$, 10 g/l $(NH_4)2SO_4$ and 40 g/l aluminum powder (mean diameter 12 µm). The pH of all solutions which measured set to 3.5 at room temperature. Deposition time was fixed at 30 min. Before plating, the substrate surface was mechanically polished with abrasive paper. The experiment were performed in a 5kW pulsed plasma processing cold wall unit. Three measurements were conducted on each sample and the results were averaged.

- Effect of current density on aluminum particle co-deposition

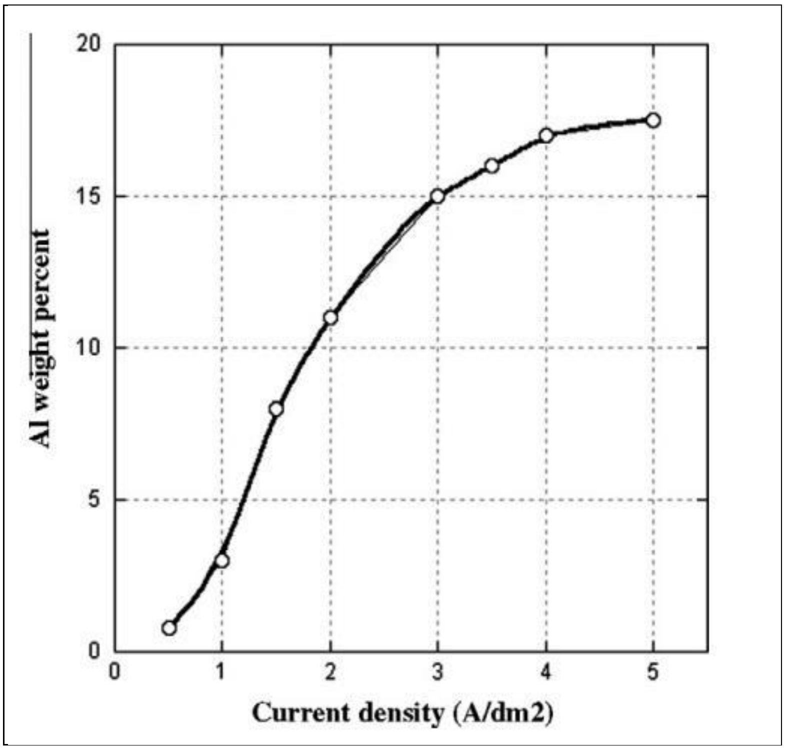


Figure2.1: The Al particle weight percent as a function of current density.

Source: Daemi et al (2010)

The structure and properties of composite coatings depend not only on the concentration, size, distribution, and nature of the reinforced particles, but also on the type of using solution and electroplating parameters such as current density, temperature, and pH value. Chemical composition of different electrodeposited Ni-Al composites, produced by different current densities was analyzed by use of X-ray fluorescence spectroscopy and the result shown in Figure 2.1.

Figure 2.1 shows that by increase of current density the incorporation of aluminum increase in the coating. It can be seen that the slope of the curve is high at current densities below $2A/dm^2$ and as the current density exceeds $3A/dm^2$, the slope decreases noticeably. Increase in Al particulates co-deposition can be attributed to the increasing tendency for adsorbed particles to arrive in the cathode surface

- Surface topography

SEM micrographs from the surface of the samples are illustrated in Figure 2.2.

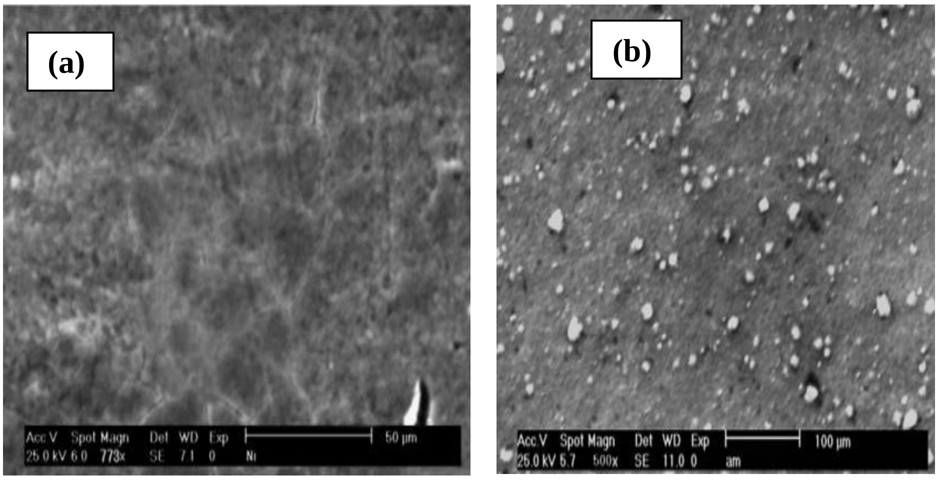


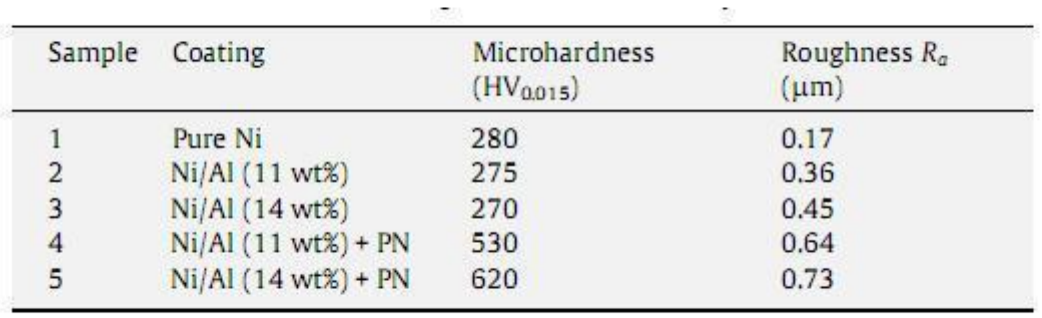
Figure 2.2: Surface morphology of composite coating

Source: Daemi et al (2010)

Figure 2.2 (a) shows pure Ni coating and figure 2.2 (b) shows Ni/Al composite coating. Comparison of Figure 2.2(a) with Figure 2.2(b) shows that the Al particles appear as the light spots in the darker nickel matrix. Besides, the size of particles is bigger than the matrix roughness and they stick out of nickel matrix. Furthermore, as can be seen, pure nickel coating has smoother surface than Ni-Al coating. By increasing embedded particles, the surface become rougher and the roughness of coating are about two and three times more than pure nickel coating.

- Microhardness measurement

Vickers microhardness (HV) values of the selected coatings are tabulated in Figure 2.3.



Sample	Coating	Microhardness ($HV_{0.015}$)	Roughness R_a (μm)
1	Pure Ni	280	0.17
2	Ni/Al (11 wt%)	275	0.36
3	Ni/Al (14 wt%)	270	0.45
4	Ni/Al (11 wt%) + PN	530	0.64
5	Ni/Al (14 wt%) + PN	620	0.73

Figure 2.3: The microhardness and surface roughness of different samples

Source: Daemi et al (2010)

Figure 2.3 shows clearly that incorporation of aluminum in nickel matrix decreases the microhardness of the coating to some extent. The mean value of Vickers microhardness of pure nickel coating has been found of about 280HV while that of composite coating is less than it. According to the rule of mixture the composite hardness can be estimated by Eq 2.1.

$$H = \frac{1}{\frac{fh}{Hh}+\frac{fs}{Hs}} \tag{2.1}$$

Which;

Hh = Hardness values of hard phases

Hs = Hardness values of soft phases

fh = volume fraction of hard phases

fs = volume fraction of soft phases

Also, other literature regarding Ni-SiC, Ni-Si_3N_4, Ni-TiO_2, etc., have reported that the presence of nano-particles caused changes in the grain size and texture of matrix. The presence of nano-particles provides more nucleation sites by increasing the surface area of the cathode and thus perturbs the nickel growth.

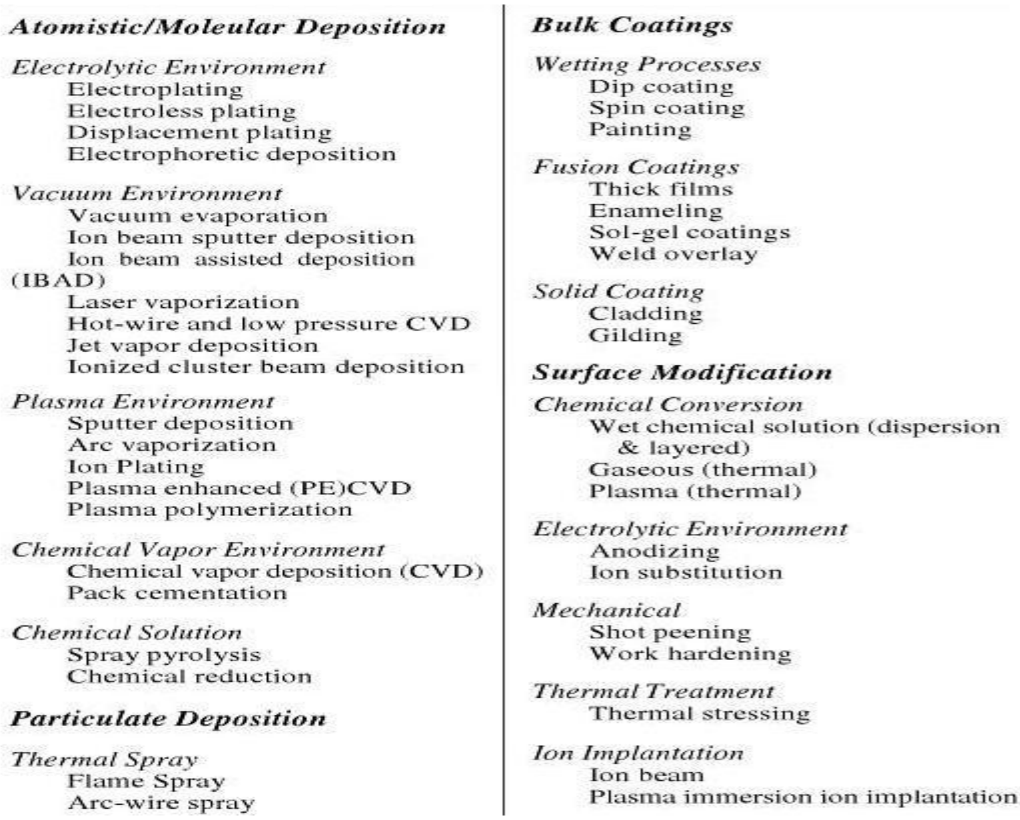
Composite coatings consisting of nickel matrix and aluminum particles can be successfully fabricated by means of electro-deposition. Al particles in the coatings increase with increase of current density (voltage). Consequently, with increasing of Al particle in coating, the roughness of coating increases.

2.2.4 Current Coating Technologies

Coating technologies has played a big role in surface engineering. This is because it involves changing the properties of the surface and near-surface region in a desirable way. Surface engineering can involve an overlay process or a surface modification process. A surface modification process changes the properties of the surface but the substrate material is still present on the surface. For example, in aluminum anodization, oxygen reacts with the anodic aluminum electrode of electrolysis cell to produce a thick oxide layer on the aluminum surface.

Nowadays, many coating techniques have been employed to produce Ni_3Al intermetallic coating on various substrates. They are such as physical vapor deposition (PVD), thermal spraying and conventional electrodeposition. Physical vapour deposition (PVD) is fundamentally a vaporisation coating technique, involving transfer of material on an atomic level. It is an alternative process to electroplating. PVD processes are carried out under vacuum conditions. The process involved four steps: evaporation, transportation, reaction, and deposition.

Figure 2.4 shows a number of overlay and surface modification processes that can be used for surface engineering. Each process has its advantages, disadvantages and applications.



Atomistic/Moleular Deposition

Electrolytic Environment
- Electroplating
- Electroless plating
- Displacement plating
- Electrophoretic deposition

Vacuum Environment
- Vacuum evaporation
- Ion beam sputter deposition
- Ion beam assisted deposition (IBAD)
- Laser vaporization
- Hot-wire and low pressure CVD
- Jet vapor deposition
- Ionized cluster beam deposition

Plasma Environment
- Sputter deposition
- Arc vaporization
- Ion Plating
- Plasma enhanced (PE)CVD
- Plasma polymerization

Chemical Vapor Environment
- Chemical vapor deposition (CVD)
- Pack cementation

Chemical Solution
- Spray pyrolysis
- Chemical reduction

Particulate Deposition

Thermal Spray
- Flame Spray
- Arc-wire spray

Bulk Coatings

Wetting Processes
- Dip coating
- Spin coating
- Painting

Fusion Coatings
- Thick films
- Enameling
- Sol-gel coatings
- Weld overlay

Solid Coating
- Cladding
- Gilding

Surface Modification

Chemical Conversion
- Wet chemical solution (dispersion & layered)
- Gaseous (thermal)
- Plasma (thermal)

Electrolytic Environment
- Anodizing
- Ion substitution

Mechanical
- Shot peening
- Work hardening

Thermal Treatment
- Thermal stressing

Ion Implantation
- Ion beam
- Plasma immersion ion implantation

Figure 2.4: Processes for surface engineering

Source: D.M.Mattox (1998)

During evaporation stage, a target, consisting of the material to be deposited is bombarded by a high ebergy source suchg as a beam of electrons or ions. This dislodges atoms from the surface of the target, ‘vaporising’ them. During transport session, the process simply consists of the movement of ‘vaporised’ atoms from the target to the substrate to be coated and will generally be a straight line affair. In some cases coatings will consist of metal oxides, nitrides, carbides and other such materials. In these cases, the target will consist of the metal. The atoms of metal will then react with the appropriate gas during the transport stage. For the above examples, the reactive gases may be oxygen, nitrogen and methane.In instances where the coating consists of the target material alone; this step would not be part of the process. Lastly, during deposition process, this is the process of coating build up on the substrate surface. Depending on the actual process, some reactions between target materials and the reactive gases may also take place at the substrate surface simultaneously with the deposition process.

The advantages of PVD process are the materials can be deposited with improved properties compared to the substrate material. Besides, almost any type of inorganic material can be used as well as some kinds of organic materials. For environmental advantages, the process is more environmentally friendly than processes such as electroplating. On the other hand, it is a line of sight technique meaning that it is extremely difficult to coat undercuts and similar surface features besides with high capital cost. Some processes also operate at high vacuums and temperatures requiring skilled operators and the rate of coating deposition is usually quite slow.

Thermal spray coating is a coating by a process in which molten or softened particles are applied by impact onto a substrate. A common feature of all thermal spray coatings is their lenticular or lamellar grain structure resulting from the rapid solidification of small globules, flattened from striking a cold surface at high velocities. They are three stages involves in the thermal spray process. The stages by sequenced are generation of thermal or kinetic energy, interaction of energy with spray material and lastly interaction of spray particles with substrate.

Most metallic coatings suffer oxidation during normal thermal spraying in air. The products of oxidation are usually included in the coating. Oxides are generally much harder than the parent metal. Coatings of high oxide content are usually harder and more wear resistant. Oxides in coatings can be detrimental towards corrosion, strength and machinability properties. Generally the as-sprayed surface is rough and textured. The rough and high bond strength coatings are ideal for bond coats for less strongly bonding coatings. Many coatings have high friction surfaces as-sprayed and this property is made use of in many applications. Some plasma sprayed ceramic coatings produce smooth but textured coatings important in the textile industry. Other applications make use of the abrasive nature of some coating surfaces. Thermally sprayed coatings do not provide bright high finish coatings without finishing like that of electroplated deposits. Coatings generally have poor strength, ductility and impact properties. These properties tend to be dictated by the "weakest link in the chain" which in coatings tends to be the particle or grain boundaries and coating/substrate interface. Coatings are limited to the load they can carry, and thus require a substrate for support; even then, coatings are poor when point loaded. Internal tensile coating stresses generally adversely affect properties. Effective bond strength is reduced and can be destroyed by increasing levels of internal stress. This in turn affects coating thickness limits. Coatings on external diameters can be built up to greater thickness than that on internal diameters. Surface properties such as wear resistance are usually good, but the properties are more specific to the material or materials used in the coating. The properties of a substrate need only to be strength, ease of fabrication and economic (like mild steel). The coating supplies the specific surface properties desired. For example, materials used for applications of thermal barrier and abradable clearance control by nature have poor strength and thus benefit from being applied as a coating onto a substrate which supplies the strength.

2.3 OVERVIEW ON Ni-ALUMINA ELECTROPLATING

Electroplating is often also called "electro deposition", and the two terms are used interchangeably. As a matter of fact, "electroplating" can be considered to occur by the process of electrode position. Electro deposition is the process of producing a coating, usually metallic, on a surface by the action of electric current. The deposition of a metallic coating onto an object is achieved by putting a negative charge on the object to be coated and immersing it into a solution which contains a salt of the metal to be deposited.

In other words, the object to be plated is made the cathode of an electrolytic cell). The metallic ions of the salt carry a positive charge and are thus attracted to the object. When they reach the negatively charged object (that is to be electroplated), it provides electrons to reduce the positively charged ions to metallic form.

Figure 2.5 shows a diagram of the electroplating apparatus used in this experiment and below shows that electrode features in an electrolytic cell.

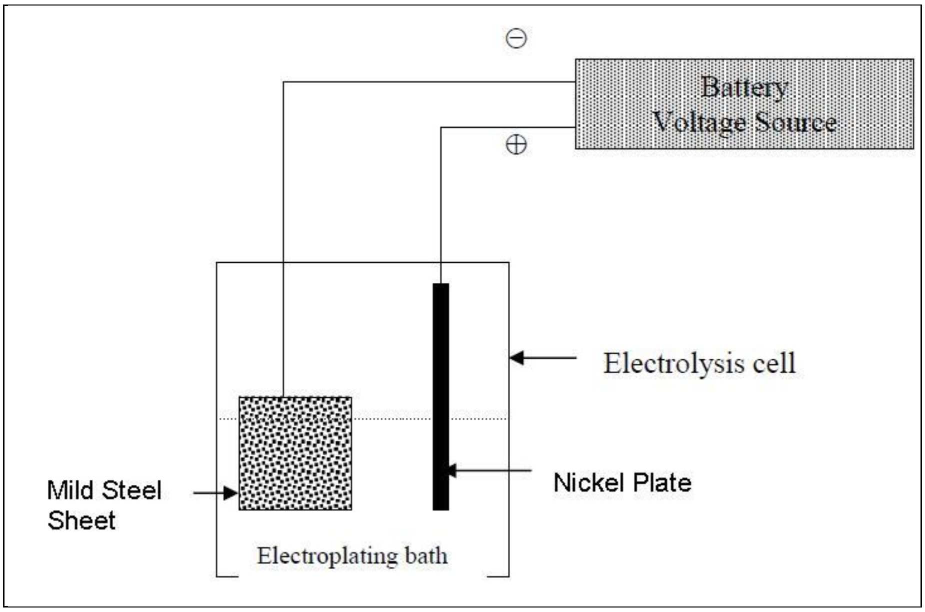


Figure 2.5: An electroplating diagram

During electroplating, a metal ion is reduced to its elemental form to coat, or plate, a surface on the cathode, the electrode connected to the negative terminal of the voltage source. The electroplating bath contains the metal ion to be reduced, as well as other ions to support the flow of electrons during electrolysis. While reduction of the metal occurs at the cathode, oxidation is simultaneously occurring at the other electrode, the anode, which is connected to the positive terminal of the voltage source.

Table 2.1: Process occurring in electroplating

	CHARGE	**PROCESS OCCURING**
Anode	Positive	Oxidation
Cathode	Negative	Reduction

Thus, back to this research, in recent years, Nickel Alumina (Ni_3Al) intermetallic alloys have attracted considerable attention because of their great potential for high-temperature structural applications due to the low density, high strength at the elevated temperature, and excellent resistance to high-temperature corrosion.

During electroplating process, the Nickel Sulfate deposited into Ni^{2+} ion and SO_4^{2-} ion according to this chemical reaction equation shows in Eq 1.1:

$$NiSO_4^{2-} \rightarrow Ni^{2+} + SO_4^{2-} \tag{1.1}$$

While the water will also dissociates shows in Eq 1.2:

$$H_2O \rightarrow H^+ + OH^- \tag{1.2}$$

The numbers of positive and negative ion charges exists equally in the solution. The object to be plated (cathode) is connected to the negative (-) side of the battery, giving it the negative charge, and the plating metal (anode) is connected to the positive (+) side of the battery, giving it a positive charge. Positive ions flow the anode toward the object being plated, through the plating solution (electrolyte), and finally deposited onto the surface of the object.

Currently, many coating techniques such as physical vapor deposition (PVD), self-propagating high-temperature synthesis (SHS), and thermal spraying have been employed to produce Ni_3Al intermetallic coatings on various substrates. Recent interests are to adopt conventional electrodeposition (CED) to prepare nickel aluminide coatings. As mentioned above, this is a two-step process including co-deposition of metal matrix (Ni) / metal particle (Al) composites, and undergoes diffusion to form alloy (Ni – Al) coatings. Nickel, being an engineering material, is the widely used metal matrix. Besides, the availability of nano-size particles in late 1990s and the resulting enhanced properties on the coating have increased the interest in the production of nickel based nano-composite coatings.

Cathode, made of mild steel was positioned in vertical plane with anode. The electrochemical studies were performed under few parameters. Surface morphologies of nano-composite coatings were examined by metallurgical microscope and optical microscope (OM). Finally, the hardness of the nano-composites coatings were measured under Vickers test

2.4 MATERIALS

2.4.1 Electrolyte

2.4.1.1 Nickel Sulfate

Nickel sulfate hexahydrate ($NiSO_4{\cdot}6H_2O$) is a blue-green crystal with odorless character. It is the major source of the nickel ions in solution. A high nickel sulfate concentration is used when high current densities are required. Besides, nickel sulfate also used in electroplating, as a mordant in dyeing, in preparation of other nickel compounds, and in paints, varnishes, and ceramics.

2.4.1.2 Nickel Chloride

Nickel chloride ($NiCl_2$--$6H_2O$), with appearance green in colour is high purity, non caking salt for use in all types of nickel plating processes. Nickel chloride serves primarily to improve anode corrosion, but also increase conductivity and uniformity of coating thickness distribution. Excessive amounts of chloride increase the corrosivity of the solution and the internal stress of the deposits. Internal stress here refers to forces created within the deposit as a result of the electro crystallization process and the co-deposition of impurities such as hydrogen, sulfur and other elements. Internal stress is either tensile or compressive, and may cause plating problems if excessively high.

2.4.1.3 Boric Acid

Boric acid has exists in the form of colorless crystals or a white powder and dissolves in water. It has the chemical formula H_3BO_3, sometimes written $B\ (OH)_3$. It is used in this electroplating solution for buffering purposes. Its concentration may affect the appearance of the deposits.

2.4.1.4 Aluminum Oxides

Aluminum Oxide, Al_2O_3 is a major engineering material. It has good mechanical properties and electrical properties leading to a wide range of applications. Alumina can be produced in range of purities with additive designed to enhance properties. A wide variety of ceramic processing methods can be applied to produce a wide variety of sizes and shapes of component.

Typical Alumina characteristics include:

(i) Good strength and stiffness
(ii) Good hardness and wear resistance
(iii) Good corrosion resistance
(iv) Good thermal stability
(v) Excellent dielectric properties
(vi) Low dielectric constant
(vii) Low loss tangent

Typical Alumina applications include:

(i) Seal rings
(ii) Medical prostheses
(iii) Laser tubes
(iv) Electrical insulators
(v) Grinding media
(vi) Wear components

2.4.2 ELECTRODES

2.4.2.1 Mild Steel

Mild Steel is sometimes referred to as carbon steel or plain carbon steel. The American Iron and Steel Institute (AISI) defines a carbon steel as having no more than 2

% carbon and no other appreciable alloying element. Carbon steel makes up the largest part of steel production and is used in a vast range of applications.

Typically carbon steels are stiff and strong. They also exhibit ferromagnetism (i.e. they are magnetic). This means they are extensively used in motors and electrical appliances. Welding carbon steels with carbon content greater than 0.3 % requires that special precautions be taken. However, welding carbon steel presents far fewer problems than welding stainless steels. The corrosion resistance of carbon steels is poor (i.e. they rust) and so they should not be used in a corrosive environment unless some form of protective coating is used.

Some of the advantages of using mild steel are they are cheap beside wide variety available with different properties. Mild steel also has high stiffness and good magnet permeability. This is because all common steel including mild steel are ferromagnetic. Besides, it also easy machine and weld. Last but not least, mild steel also has its advantages, which poor corrosion resistance such as rusts.

2.4.2.2 Nickel

Nickel is a silvery white metal which takes on a high polish. It belongs to iron group and is hard, ductile and malleable and somewhat ferromagnetic. It is a fair conductor of heat and electricity. Nickel is a member of the iron-cobalt group of metals.

Typical use of Nickel:

(i) making of austenitic stainless steel
(ii) As superalloys
(iii) In alloy steel, rechargeable batteries, foundry products, and plating.
(iv) In crucibles for chemical laboratories
(v) Smart wire, or shape memory alloys are use in robotics
(vi) High resistant to corrosion which very used for propellers, kitchen supplies, and chemical industry plumbing.

Electroplating is the most widely use method of nickel plating. For surface preparation, prior to plating operation the cathode surface should be cleaned from mineral oils, rust protection oils, cutting fluid such as coolant, greases, paints, animal lubricant and vegetable lubricants, fingerprints, miscellaneous solid particles, oxides, scale, smut, and rust.

They are many of properties of nickel coatings. One of the properties are decorative appearance. Lustrous bright, satin semi-bright or black nickel coating may be obtained b different plating methods. Besides, it also has good corrosion protection and low coefficient of friction. Nickel deposited on a part made of a softer metal protects the part from wear, too. Hardness of nickel plating may be controlled by the plating process parameters. Furthermore, nickel coating has controllable internal mechanical stresses. Low stress coatings are important in electroforming and applications, in which Fatigue strength is critical.

2.5 HARDNESS TEST

Vickers test are one of hardness test of a material. The Vickers hardness test involves a diamond indenter, in the form of a square-based pyramid with an apex angle of 136°, being pressed under load for 10 to 15 s into the surface of the material under test. The result is a square-shaped impression. After the load and indenter are removed the diagonals d of the indentation are measured. The Vickers hardness number (HV) is obtained by dividing the size of the force F, in units of N, applied by the surface area, in mm^2, of the indentation like in Eq 2.2:

$$\text{VickersHardness} = \frac{Applied\ Force}{Surface\ Area\ of\ Identation} \tag{2.2}$$

The surface area can be calculated like Eq 2.3 from the mean diagonal value, the indentation being assumed to be a right pyramid with a square base and apex angle Θ of 136°.

$$\text{Area}=\frac{d^2}{2\sin\frac{\theta}{2}}=\frac{d^2}{1.854} \tag{2.3}$$

Thus the Vickers hardness HV is given by Eq 2.4:

$$\text{HV}=\frac{1.854F}{d^2} \tag{2.4}$$

In this project, Vickers hardness will be applied on Ni-Alumina coating. Using Vickers hardness tester, 500N load will be applied and three readings will be get. Then, the values will be averages to get an accurate hardness value. The graph will be plot later to see more clearly relation between coating hardness and given parameter.

2.6 PURPOSES OF Ni-ALUMINA ELECTROPLATING

Some of the purposes of electroplating are:

(i) Appearance
(ii) Protection
(iii) Special surface properties
(iv) Mechanical properties

Electroplated coatings are applied to metals for many reasons. Not only must the metal to be electroplated be specified, but also its thickness and often other properties specific to the use of article. For example, gold can be applied as a coating so thin that it serves, as customer appeal, or its thickness can be sufficient to survive many years of use. Since most metals are expensive, and since the cost of an electroplated coating rises with its thickness, it is important to specify sufficient coating to serve the purpose but no more.

When the function of the coating is to protect the basis metal and appearance is of minor importance. The meaning of the word protection here means against environmental corrosion as is undergone by many metals such as steel in seacoast, industrial, and rural environments. In other word: rusting, corrosion, and their equivalents.

Besides, electroplated finishes may be used to improve the appearance. This appearance must be maintained for a reasonable time, so in most cases some protective function is involved too. For example, automobile bumper would probably fulfill their function even if rusty, but the appearance of the car would be unsatisfactory to the customer. Thus, the plated finish must protect the basis metal. There are a few examples of purely decorative finishes, with no protective function, but most so-called decorative finishes might better be called decorative/ protective.

Electroplated finishes may be used to modify the chemical or physical properties of the surface, or to change its dimensions. These includes improvement of bearing properties, solderability, wearibility, and abrasion resistance, building-up dimensions of worn or mismachined parts, modification of hardness and reflectivity, and much besides.

CHAPTER 3

METHODOLOGY

3.1 PROJECT PROCESS FLOW

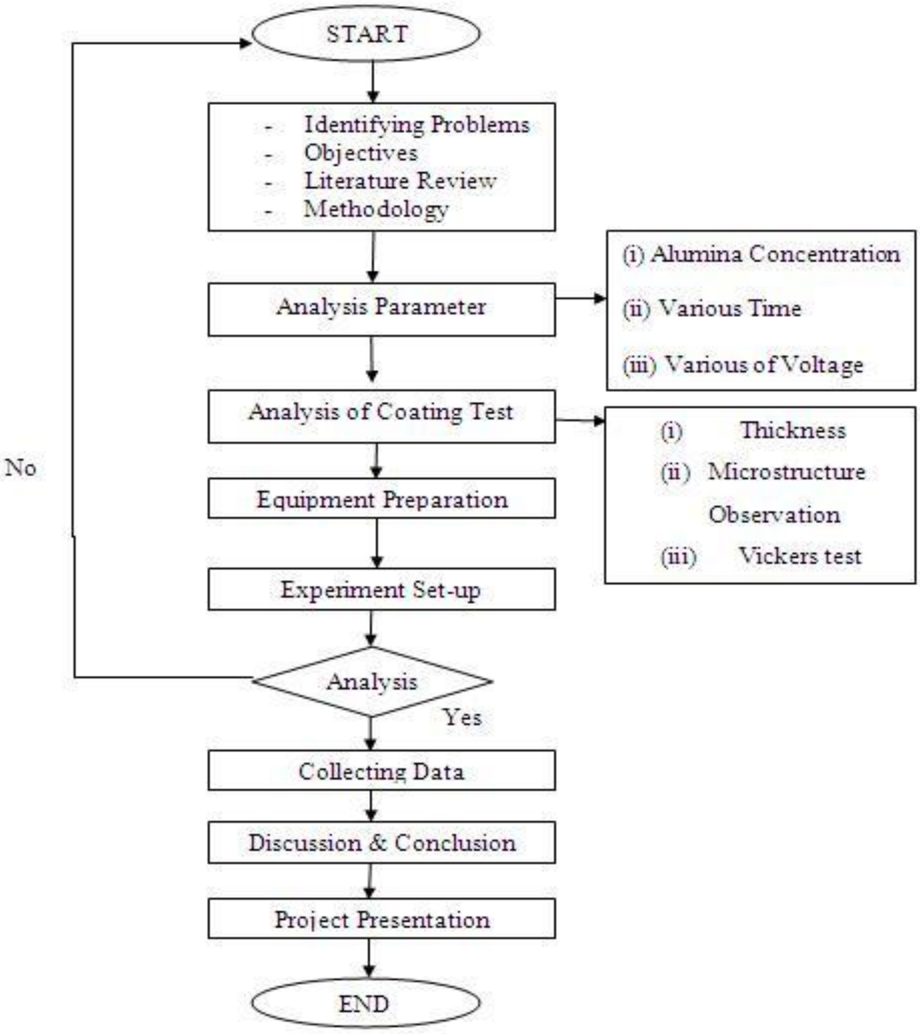


Figure 1.1: Project process flow chart.

3.2 PREPARATION OF ELECTRODE

3.2.1 Type Of Metal Use

The metals use for both electrodes on this project is mild steel and nickel plate. At cathode, it will be mild steel sheet. Nowadays, mild steel is the common form of steel because its price relatively low while it provides material properties that are acceptable for many applications. In this project, Hot Rolled Mild Steel Sheet A569/ASTM A1011 was used. From a large sheet of mild steel were cut into smaller pieces using machine show in

Figure 3.1: The Metal Cutting Guillotine Machine

Mild steel differs from stainless steel in its chromium content. Stainless steel contains a lot more chromium than mild steel. Amount of carbon makes mild steel different from other types of steel. It’s make mild steel stronger and stiffer than others. However, the hardness comes at the price of a decrease in ductility. In addition, carbon atoms get affixed in the interstitial sites of the iron lattice and make it stronger.

On the other hand, at anode side, it will be nickel plate. In this project, Nickel 201 Sheet/ Plate (UNS N02201 ASTM B162) will be use. Same with mild steel, it will be cut into smaller and specific size. However, the process to cut nickel plate didn’t need to use metal cutting guillotine machine. This is because the thickness and the hardness of nickel plate were much lower. Nickel has an advantages because of it contains alloy. Generally, it will have good corrosion resistance and thus has an ability of a metal to avoid surface damage as well as erosion and abrasion. This property makes nickel alloy useful in industries. Besides, nickel alloy has high temperature performance. They are known for the retention of important mechanical properties, such as impact strength, yield strength, and hardness.

3.2.2 Size Of Specimen

Specifically, the size of mild steel will be 80mm x 30mm x 1.5mm, length, width, and thickness respectively. Figure 3.2 (a) and Figure 3.2(b) shows the dimension of specimen of mild steel and all specimens will be in the same size.

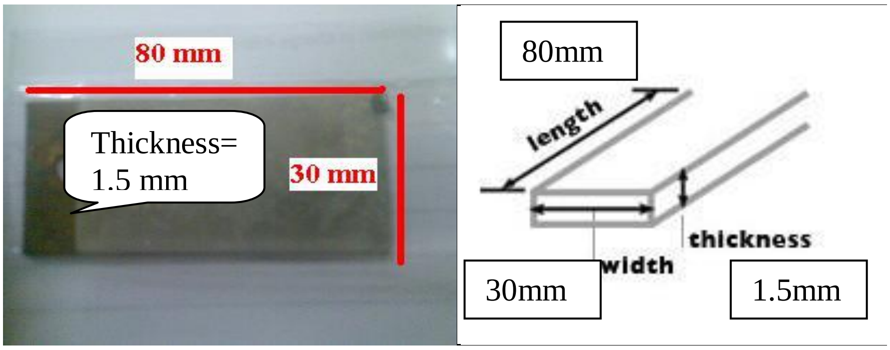


Figure 3.2: Dimension of mild steel

For nickel plate, all specimens will be cut into smaller size with 80mm x 30mm length and width dimension respectively. Figure 3.3 shows one of specimen of nickel plate that been cut into smaller and ready to be use in this project.

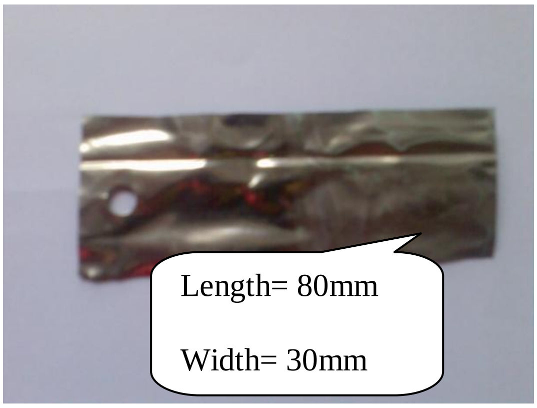


Figure 3.3: Dimension of Nickel Plate

3.2.3 Total Quantity Specimen In Experiment

The quantity specimen in experiment were depends on experiment parameter. In this project, they are three parameters that will experiments. They are concentration of Alumina, plating time, and voltages supplied. Furthermore, in each parameter, they will be three samples. Thus, total quantity specimen needed in this experiment can be define by Eq 3.1.

$$\text{Quantity of specimen} = 3 \times N \tag{3.1}$$

Where;
N = total of parameters

Since they are three parameters on this experiment, the value of n will be 3. Thus, total specimens needed are:

$$3 \times N = 3 \times 3$$
$$= 9 \text{ (specimens)}$$

3.3 PREPARATION OF ELECTROLYTE

3.3.1 Electroplating Bath

The content of this project's electroplating bath will be shows in Table 3.1. However, Alumina powder will not be included in this table since it will be one of parameter. Thus, the quantity use will be different by experiment. Overall diagram sample with parameter can be seen in Appendices. This electroplating bath is based Watts bath.

Table 3.1: The Content of Electroplating Bath

ELECTROPLATING BATH	
300g/l	Nickel Sulfate Hexahydrate ($NiSO_4.6H_2O$)
45g/l	Nickel Chloride Hexahydrate ($NiCl_4.6H_2O$)
35g/l	Boric acid (H_3BO_3)

3.3.2 Preparation of Solution

So, the content of electroplating bath will be Nickel Sulfate Hexahydrate ($NiSO_4.6H_2O$), Nickel Chloride Hexahydrate ($NiCl_4.6H_2O$), Boric acid (H_3BO_3), and Alumina powder. This chemical will be mix to make the electroplating solution.

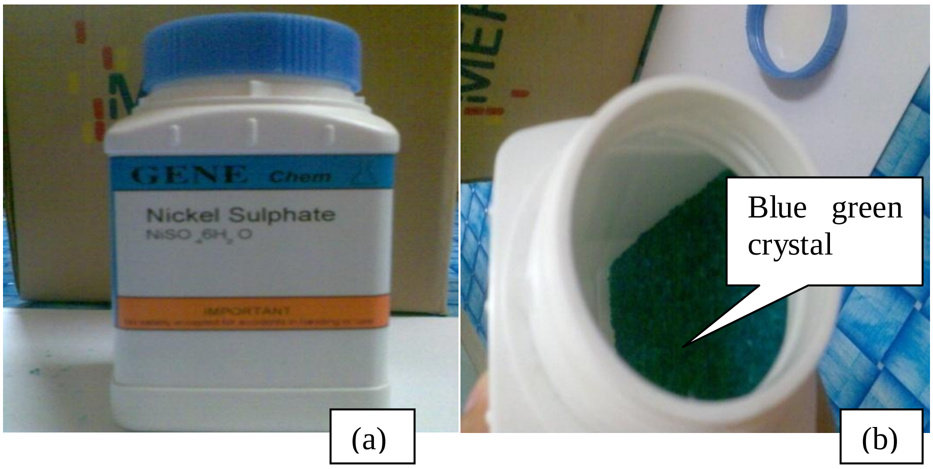


Figure 3.4: Nickel Sulfate Hexahydrate ($NiSO_4.6H_2O$)

Figure 3.4(a) shows Nickel Sulfate and meanwhile Figure 3.4(b) shows its appearance. Nickel sulfate were use in this electroplating as a mordant in dyeing, in preparation of other nickel compounds, and in paints, varnishes, and ceramics.

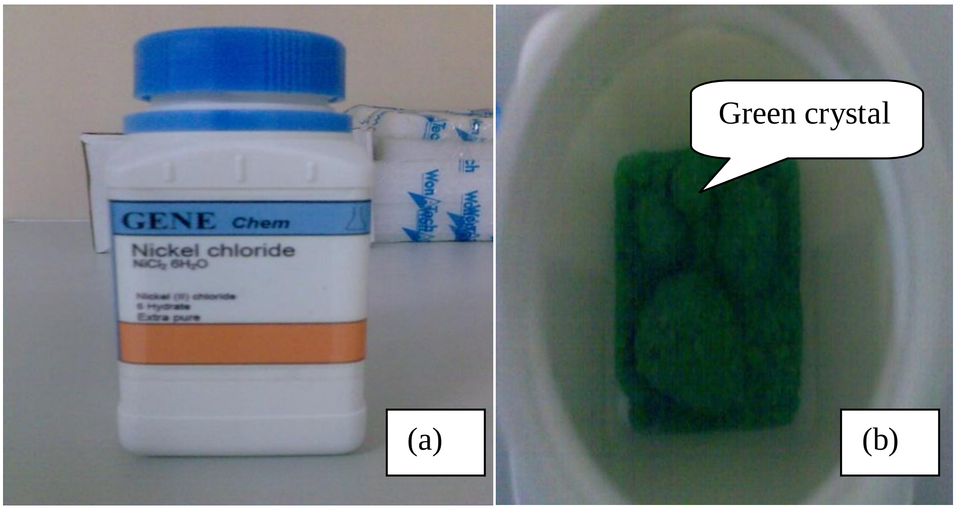


Figure 3.5: Nickel Chloride Hexahydrate ($NiCl_4.6H_2O$)

Figure 3.5 (a) shows Nickel Chloride Hexahydrate and Figure 3.5 (b) shows it is green in color. It will be use in every plating process due to its properties. It helps to increase the corrosion and deposits activities at anode. Thus, increase the coating thickness.

Figure 3.6 : Boric Acid

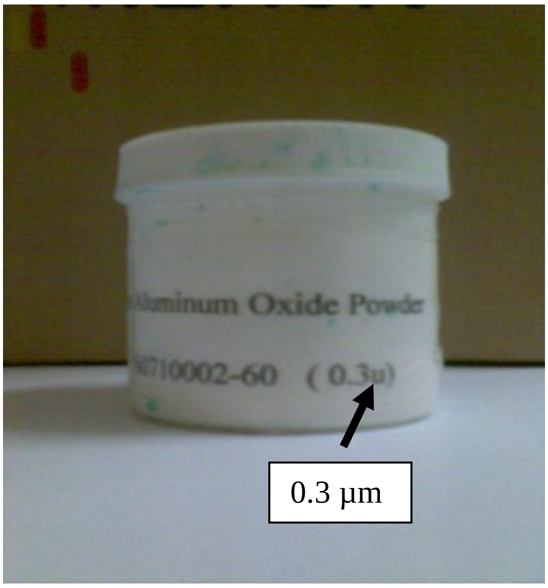


Figure 3.7: Alumina Oxide Powder

In addition, boric acid as shown in Figure 3.6 has been used as buffer agent in this electroplating. Appear in the form of colorless; this chemical has been widely use in varities of industries. Figure 3.7 shows alumina powder. The size of alumina oxide powder use in this electroplating is 0.3 µm. The size gives different result of electroplating. However, the affect of alumina powder size will be neglects in this project. The ceramic properties of alumina powder also give it advantages.

3.4 EXPERIMENT APPARATUS & INSTRUMENTATION PREPARATION

3.4.1 Apparatus Applied

There are a lot of apparatus involves in this electroplating. Fig 3.8 shows a DC power supply that used to be voltage supplier in this electroplating process. The current supplied will be set 1.5A. Meanwhile the voltage will be supplied according to set of parameter. Besides, two electroplating can be work out in a same time using this power supply. From figure, we can see that red wires are connected to negative terminal and black wires are connected to positive terminal.

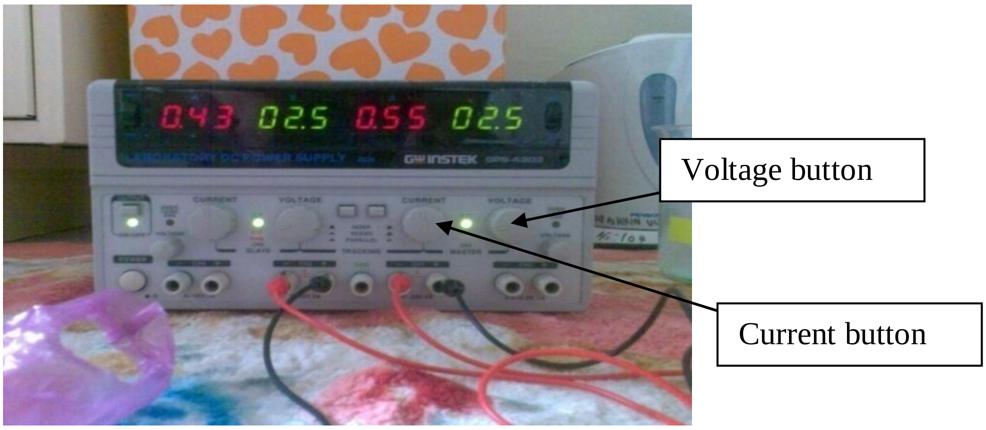


Figure 3.8: DC power supply

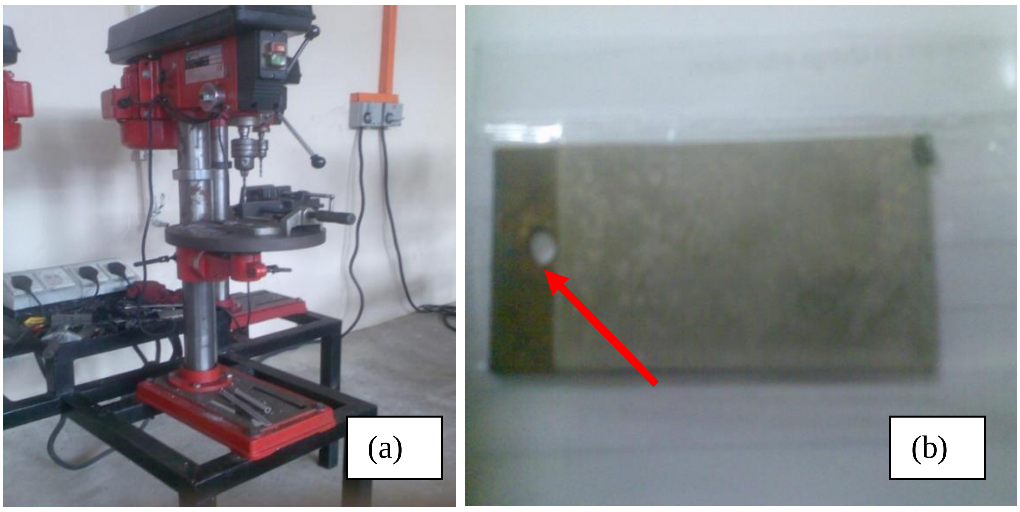


Figure 3.9: Bench Drill

Figure 3.9(a) shows a bench drill. This mechanical drill is use to drill a small hole on mild steel plate. This is also one of preparation for specimen. In addition, Figure 3.9 (b) shows an arrow that show a hole drilled by bench drill.

Figure 3.10: Gsm Weighing Machine

Figure 3.10 shows a gsm weighing machine. Samples of mild steel and nickel plate will be scales before and after electroplating. These scales give an accurate and precision weight measurement. Besides, preparation for solution also will be using this machine. Meanwhile, Figure 3.11 shows fume hood, where all the solution were prepared and etching process done.

Figure 3.11: Fume hood

Figure 3.12(a) is a linear precision saw machine that used to cut workpieces into smaller samples. There are nine work pieces that been cut using this machine. After electroplating, mild steel were grind into size of 20mm x 30 mm, as shown in Figure 3.12 (b). The samples were grind into a suitable size to undergo mounting process.

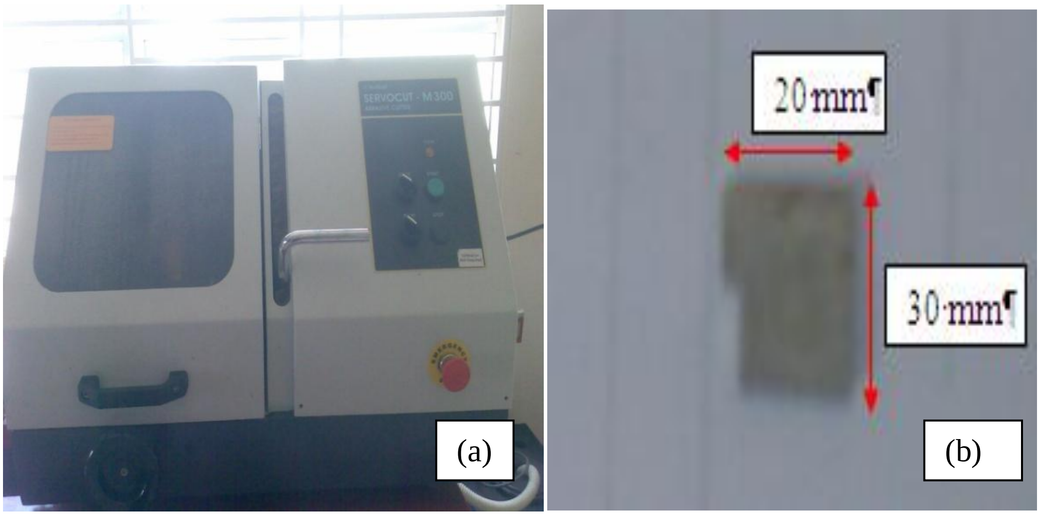


Figure 3.12: Linear Precision Saw Machine

Figure 3.13(a) shows the metal mounting machine that use to mount all the samples. Mounting is a process to watch properly the material structure and measure the hardness of the material by hardness tester later. Besides, Figure 3.13(b) shows some of samples that had done mounting successfully using the metal mounting machine.

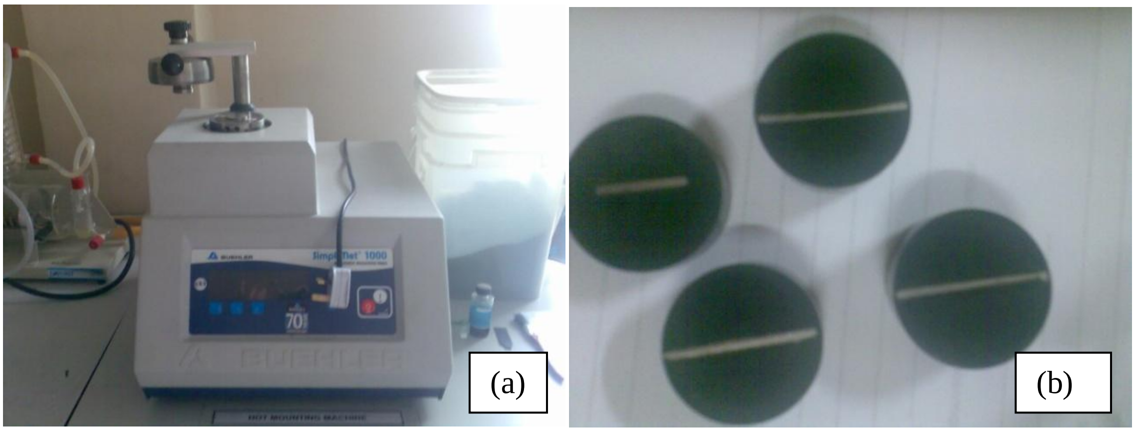


Figure 3.13: Metal Mounting Machine

Figure 3.14 shows the polishing machine that use to grind and polish samples surface. Figure 3.15 shows the roll grinding machine. The function is same as Figure 3.14., which used to grind samples to have a good surface.

Figure 3.14: Polishing Machine **Figure 3.15:** Roll Grinding Machine

Figure 3.16(a) is a metallurgical microscope. It's used to observe the thickness of coating beside to observe the surface microstructure on coating. In addition, this microscope come with USB digital imaging to capture the microscopic image that are later on adapted to metallographic microscope. There are four magnifications on this microscope as shown in Figure 3.16 (b).

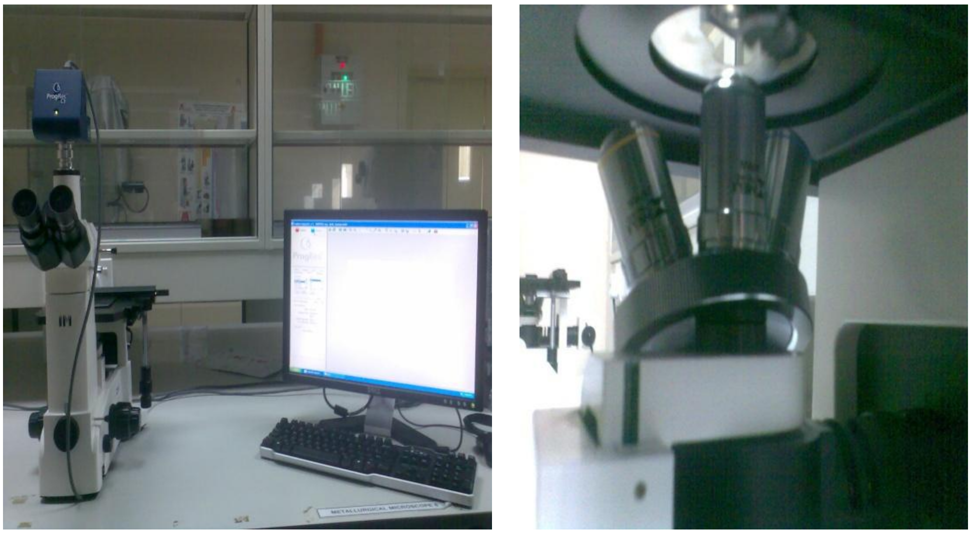


Figure 3.16: Metallurgical Microscope

Figure 3.17 shows the Vickers Hardness Tester that use to perform the hardness test on coating. Load 500g were applied on specimen and five reading will be taken to observe the hardness.

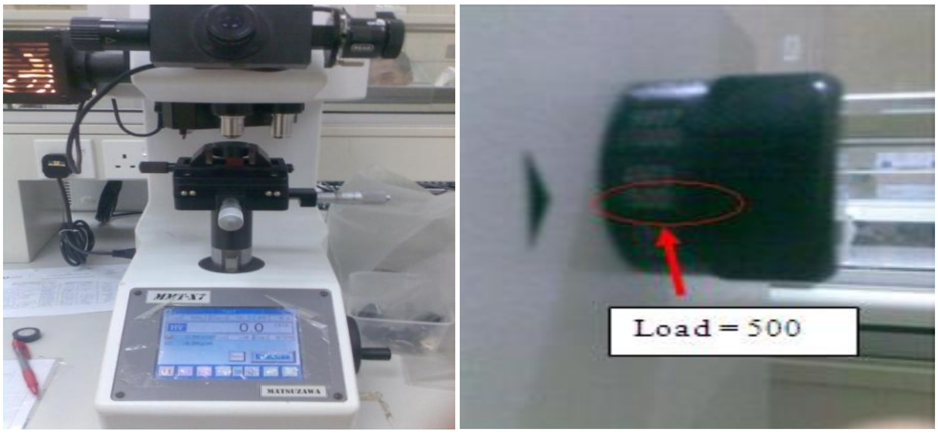
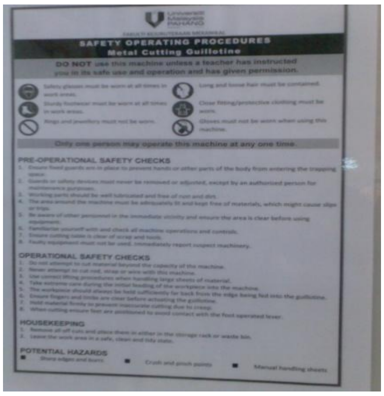


Figure 3.17: Vickers Hardness Tester

3.4.2 Caution and Disposal of Instrumentation

They are some safety precautions that have to be aware during lab area. Especially when handling with many kind of machine there. Figure 3.18 shows that safety operating procedures note of metal cutting guillotine machine were stick on the machine. Only one person may operate this machine at any one time. Pre-operational and operational safety check is highlighted to make sure the safety guaranteed. Furthermore, housekeeping is important, too. Leave the work area in a safe, clean, and tidy state. The potential hazards are such as sharp edges, crush and pinch point.



SAFETY OPERATING PROCEDURES
Metal Cutting Guillotine

DO NOT use this machine unless a teacher has instructed you in its safe use and operation and has given permission.

Only one person may operate this machine at any one time.

PRE-OPERATIONAL SAFETY CHECKS

OPERATIONAL SAFETY CHECKS

HOUSEKEEPING

POTENTIAL HAZARDS

Figure 3.18: Safety Operating Procedure Note

Figure 3.19: Caution Note on Linear Precision Saw Machine

In Figure 3.19, an arrow shows there is caution note on linear precision saw machine, too. Do not force to open the hood before switching the instrument on. Furthermore, do not force to open the hood during cutting. Otherwise, the interlocking safety switch will be defect.

3.5 OVERALL EXPERIMENT CONFIGURATION

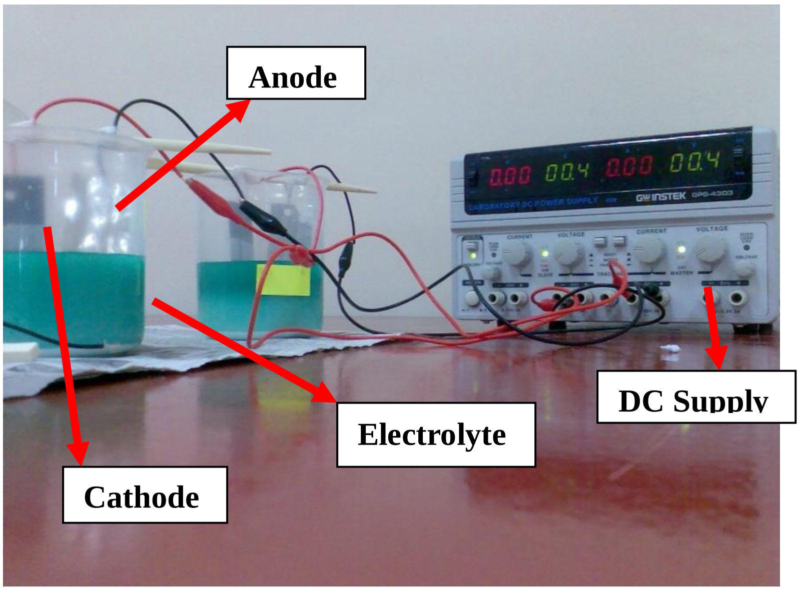


Figure 3.20: Experiment Set-up Configuration

Figure 3.20 shows the set-up configuration of Ni-Alumina electroplating. Mild steel will at cathode and nickel plate is at anode terminal. The solution color is in green color due to presence of nickel sulfate and nickel chloride. DC supply as power supply in the process.

3.6 DESIGN OF EXPERIMENT (D.O.E)

3.6.1 General Flow of Experiment

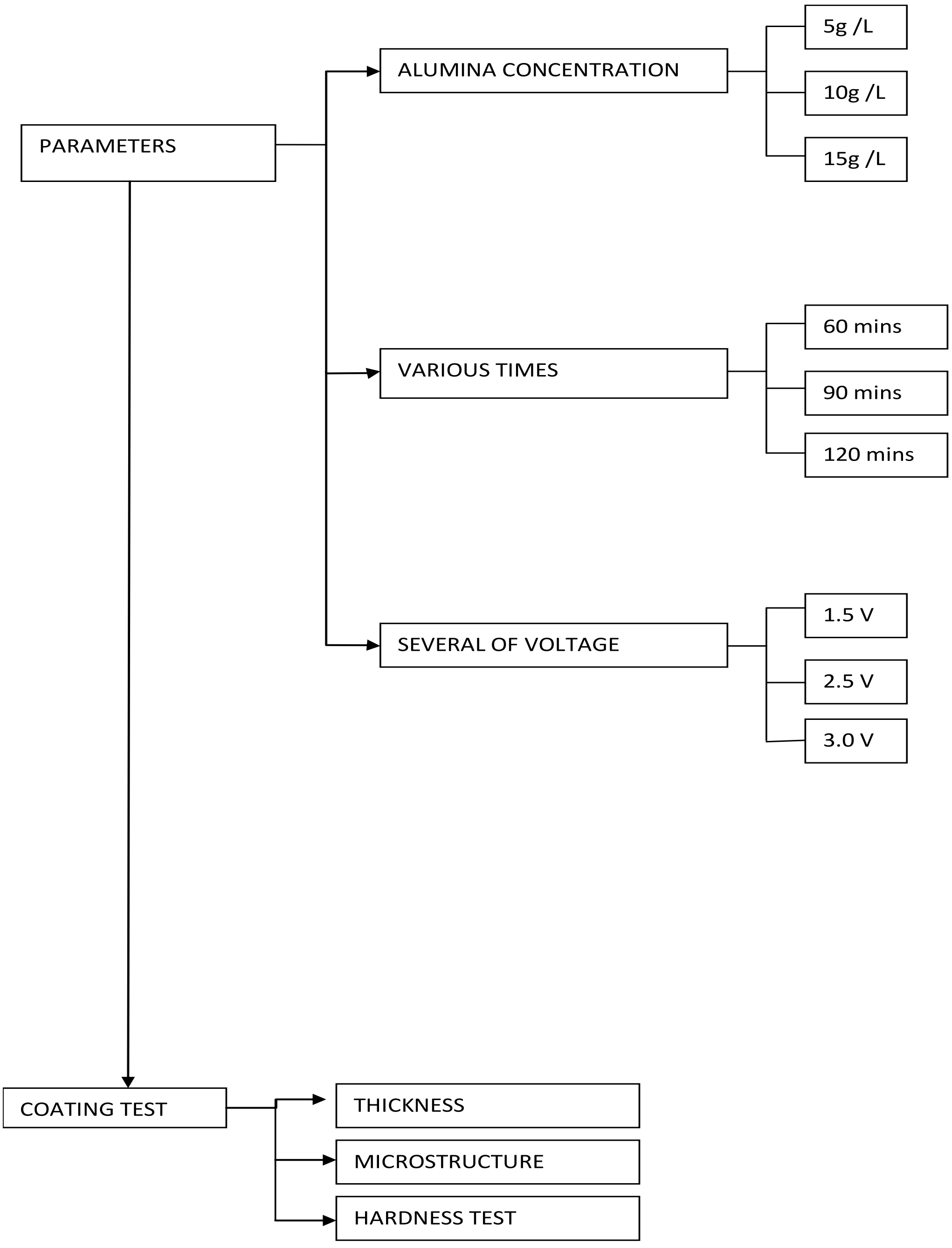


Figure 3.21: Experiment Process Flow

Figure 3.21 shows the electroplating experiment process flow together with its parameters.

3.6.2 Step By Step Procedure

i. Prepare all the equipment and apparatus and make sure all the apparatus can function well.

ii. Firstly, cut both mild steel and nickel plate into 80mm x 30mm dimension.

iii. For sample preparation, clean the mild steels using sand paper to remove scale, oxide and rust. Figure 3.22 shows a sample of mild steel with scale, oxide and rust surface.

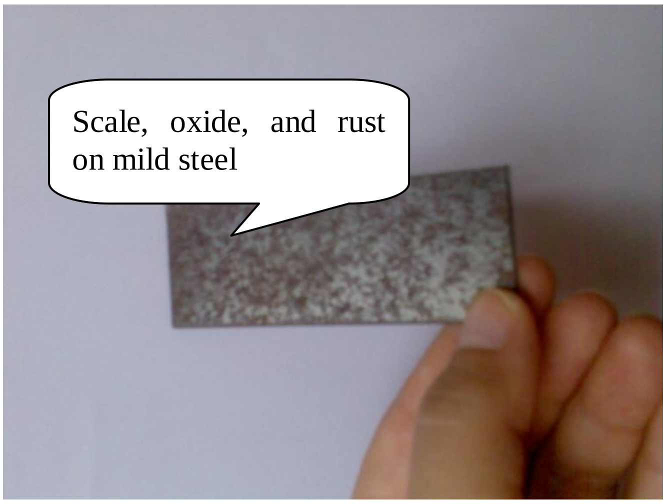


Figure 3.22: Sample of Mild Steel before Cleaning Process

iv. Then, wash all specimens with distilled water, wipe and dry.

v. Table 3.1 shows the content of the electroplating bath.

vi. Pour Alumina Powder separately into electrolyte due to different parameter.

vii. The current supply will be constant with value of 1.5 A in room temperature conditions.

viii. In order to accomplish the objectives of performing the experiment, there will be 3 electroplating parameters: Alumina concentration, plating time, and voltage supplied which vary-related into three main sub-experiments.

Table 3.2 shows the parameter of alumina concentration was observed on experiment I.

Table 3.2: Parameter experiment I

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	2.5
2	10	60	2.5
3	15	60	2.5

Table 3.3 shows the parameter of plating time was observed on experiment II.

Table 3.3: Parameter experiment II

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	2.5
2	10	90	2.5
3	15	120	2.5

Table 3.4 shows the parameter of voltage supplied was observed on experiment III.

Table 3.4: Parameter experiment III

SET	Al_20_3 CONCENTRATION (g/l)	TIMES (MINS)	VOLTAGE (V)
1	5	60	1.5
2	10	60	2.5
3	15	60	3.0

ix. Observe the differences of the electrode. .
x. Then, remove and dry each electrode.
xi. Calculate and determine the weight loss of Nickel plate.
xii. After that, grind mild steels with coating into 20 mm x 30 mm dimension.
xiii. Mount all of the specimens using metal mounting machine with green resin, which shown in Figure 3.23.

Figure 3.23: Green Resin

xiv. Next is grinding and polishing session.

xv. Grind specimens started with 180 grade silicon carbide paper and water.

xvi. Continue grinding until the sample surface is flat, and all the scratches are in a single orientation.

xvii. Wash the sample in water and then move to the next level until 1200 grade.

xviii. Then, finish the polish process by starting with 6 micron grade disc covered on polisher surface.

xix. Continue polish until the grinding scratches remove.

xx. Next, investigate the coating thickness and microstructure.

xxi. Finally, apply hardness test on specimen using Vickers Hardness Tester.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter will be present the experiment analysis data taken from the beginning until the end of the experiment.

4.2 RESULTS

4.2.1 Coating Thickness (µm)

Table 4.1 Thickness of Ni-Alumina coating

	PARAMETER		
SET	**Al_2O_3 CONCENTRATION**	**PLATING TIME**	**VOLTAGE SUPPLIED**
1	6.786	15.23	26.53
2	7.403	20.98	26.58
3	8.659	-	83.90

Table 4.1 shows the experimental results for various different parameters (Al_2O_3 concentration, plating time, and voltage supplied) to coating thickness in units of micro meter. For Alumina Oxides concentration, 5, 10, and 15 g/l of Alumina powder sized of 3µm were dividing into three set respectively. The experiment shows the increment use of alumina powder in the electrolyte directly proportional to the coating thickness. Besides, experiments were done on plating time parameter. Theoretically, the coating thickness will increase when plating time take longer. To prove this, the experiment was

done in 60 minutes at first, and then the experiment will be repeated with 90 minutes. The result on experiment also shows the increment of coating by plating time.

The last parameter were used are voltage that supplied to the electroplating process. At first, 1.5 voltages were supplied to the process and resulted of 26.53 µm of coating thickness. Then, the voltages were set different higher on the other two experiments. Finally, the result shows that the higher of voltage supplied the thicker of coating thickness that will be got. Three graphs with different parameter can be compared in order to achieve the objective which is defining relationship between the parameters to the coating thickness.

4.2.2 Weight loss At Anode

Table 4.2 Weight loss of Anode

	PARAMETER		
SET	**Al_2O_3 CONCENTRATION**	**PLATING TIME**	**VOLTAGE SUPPLIED**
1	0.021	0.593	0.413
2	0.026	0.989	1.011
3	0.027	0.031	1.335

Table 4.2 shows that the parameters (Al_2O_3 Concentration, plating time, and voltage supplied) will affect the weight loss of anode. As designed, the anode will be the nickel plate, which the one that will be deposited and thus, will lose its weight. For first parameter, Al_2O_3 Concentration, there are three sets. Alumina powder will be increase in three different set respectively. So, as a result, it shows that increase of alumina powder will increase the weight loss of nickel plate on anode.

On second parameter- plating time, the result also shows that the weight loss of anode was directly proportional to the plating time taken. It means that, when the electroplating time taken longer, more nickel will be deposited and dissolved in the electrolyte and thus produces thicker coating at cathode.

Voltage supplied was used to activate the electron inside the electrolyte to react to each other. On third parameter, the voltages supplied are increasing in three sets order respectively. As a result, the higher the voltage supplied, more nickel plate on anode will dissolve in electrolyte and thus the higher weight of loss will be got. Three graphs with different parameter can be compared in order to achieve the objective which is defining relationship between the parameters to the coating thickness.

4.2.3 Microstructural Analysis

Microstructural analysis had done at all the samples to observe the surface due to Al_2O_3 concentration, plating time, and voltage supplied effect respectively.

4.2.3.1 Coating Thickness due to Al_2O_3 Concentration

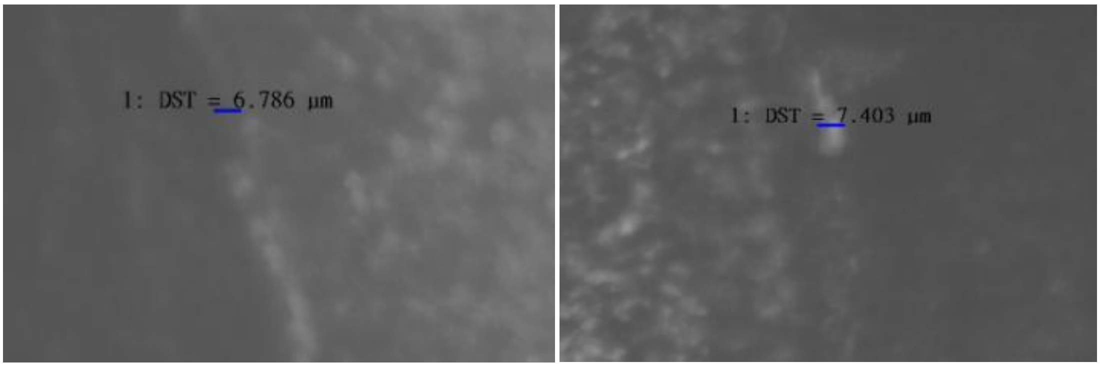


Figure 4.1(a): at magnification 400x **Figure 4.1(b):** at magnification 400x

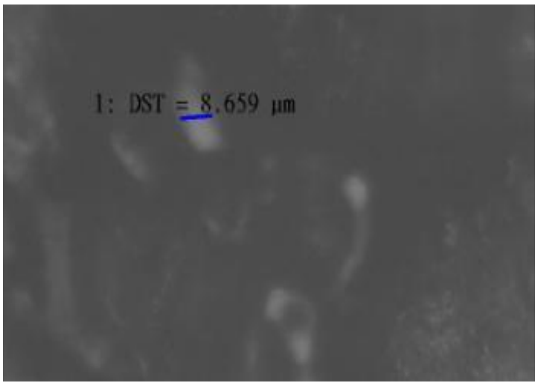


Figure 4.1(c): at magnification 400x

Figure 4.1: Coating Thickness Due to Al_2O_3 Concentration

Figure 4.1(a), Figure 4.1(b), and Figure 4.1(c) was taken by Inverted Microscope with magnification 400x. These are sample for experiment I with different alumina concentration, same plating time, and same voltage in standard room temperature condition. Figure 4.1(a), is for electrolyte contains of 5g/l Alumina powder, Figure 4.1(b) is for 10g/l Alumina powder, and Figure 4.1(c) is for 15g/l Alumina powder. The microstructures of these samples have different structure and thickness. From figure 4.1, we can see the thickness is approximately the same but increasing. This is because of Alumina powder does not affect its thickness much, but give the coating its ceramic properties. However, alumina powder helps to improve the coating thickness.

4.2.3.2 Coating Thickness due to Plating Time

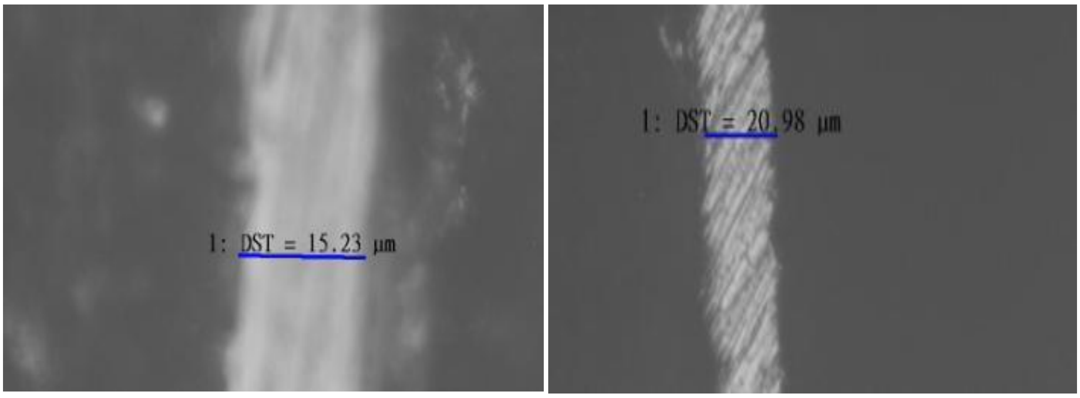


Figure 4.2(a) at magnification 400x **Figure 4.2(b)** at magnification 400x

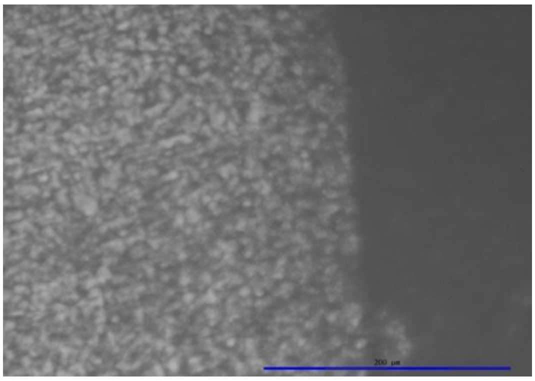


Figure 4.2(c) at magnification 400x

Figure 4.2: Coating Thickness Due to Plating Time.

Figure 4.2(a), Figure 4.2. (b), Figure 4.2(c) are samples for experiment II. The experiments were done with different of plating time, same Al_2O_3 concentration and voltage supplied in standard room temperature condition. Figure 4.2(a) is for 60 minutes plating time, Figure 4.2(b) is for 90 minutes plating time, and Figure 4.2(c) is for 120 minutes plating time. From Figure 4.2(a), we can observe there are bright Ni-Alumina plating which is 15.23 µm. Meanwhile, the coating for Figure 4.2(b).12 is thicker than Figure 4.2(a). This is because the time plating influences to get better structure and thickness. Figure 4.2(c) can be assumed as an exception after the experiment fail to electroplate successfully. More details about this will be discussed on Limitation part later.

4.2.3.3 Coating Thickness due to Voltage Supplied

All three Figures 4.3 are samples for experiment III. Figure 4.3 (a), Figure 4.3 (b), and Figure 4.3 (c) are samples after undergoes experiment with different of voltage supplied, same Al_2O_3 concentration and plating time in standard room temperature condition.

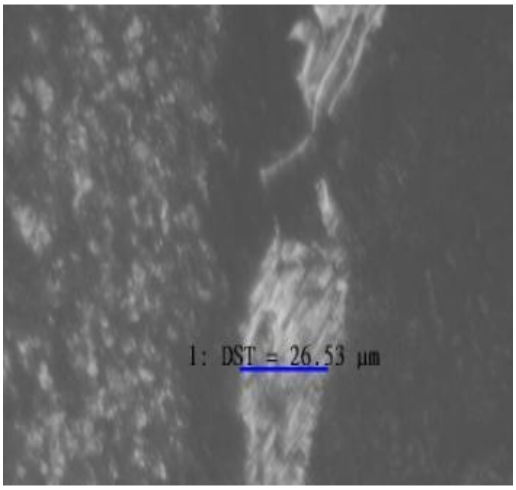


Figure 4.3(a) at magnification 400x

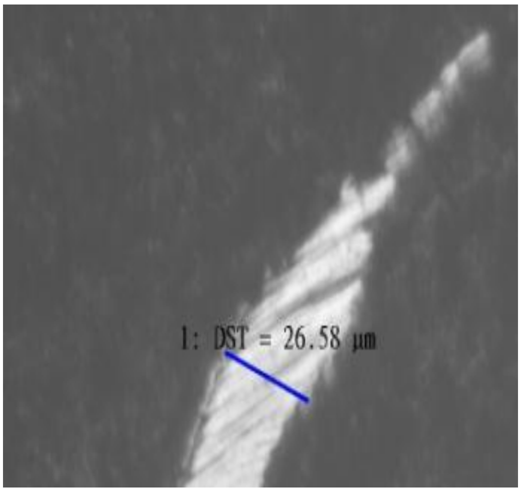


Figure 4.3 (b) at magnification 400x

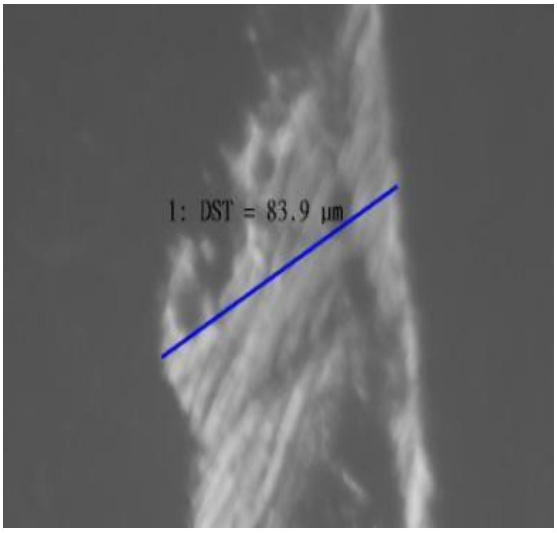


Figure 4.3(c) at magnification 400x

Figure 4.3: Coating Thickness Due to Voltages Supplied.

These experiment proves that the voltage supplied were directly proportional to coating thickness. From Figure 4.3 (a), the Ni-Alumina coatings obtained were 26.53 µm. The thickness is increase to 26.58 µm for Figure 4.3(b) with 2.5V supplied. From Figure 4.3 (c), we can see that the Ni-Alumina coating was coated perfectly with 3.0V of voltages supplied. This shows that voltage have its influence on electroplating process.

4.2.4 Microstructure of Coating Surface

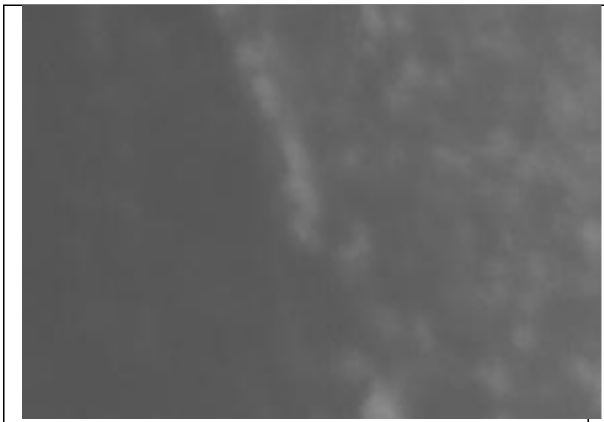
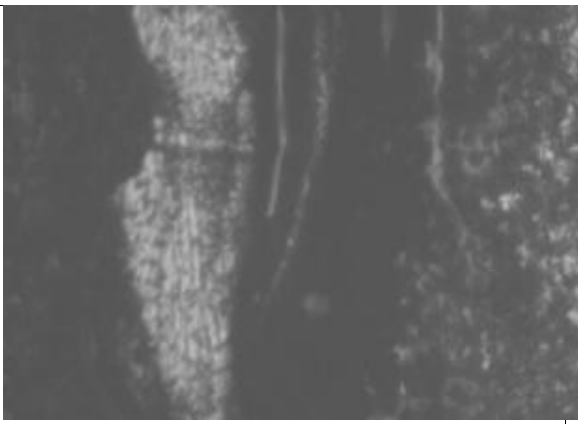
	

Figure 4.4(a) at magnification 400x	Figure 4.4(b) at magnification 400x

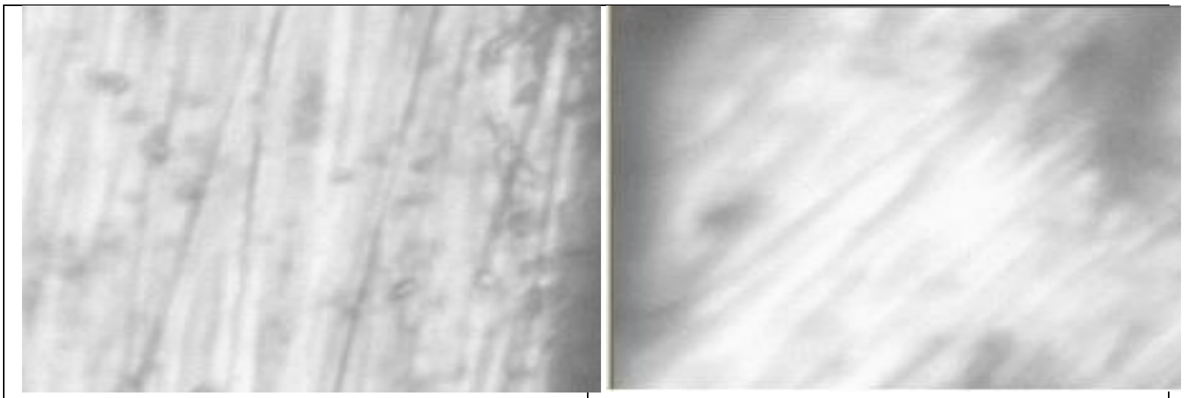


Figure 4.4(c) at magnification 400x

Figure 4.4(d) at magnification 400x

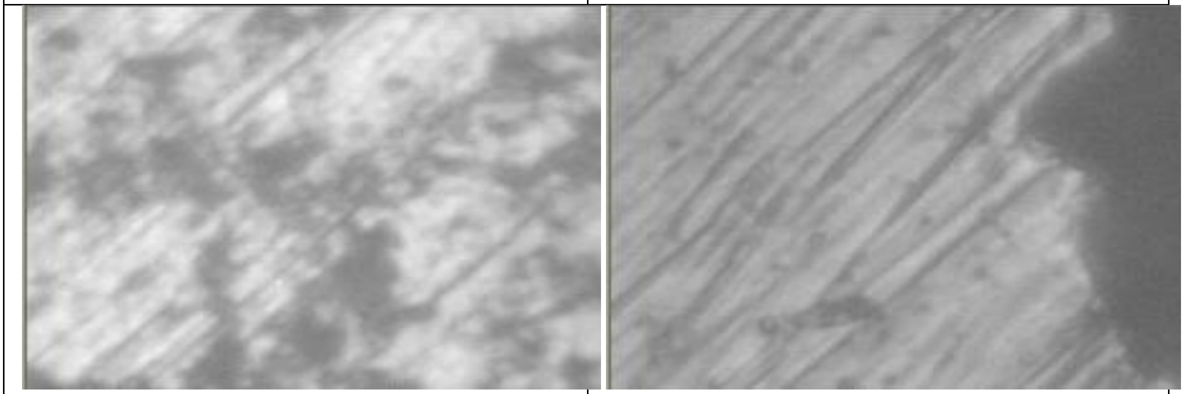


Figure 4.4(e) at magnification 400x

Figure 4.4(f) at magnification 400x

Figure 4.4: Microstructural Surface Observes By Metallurgical Microscope

Figure 4.4 illustrates the surface microstructure of Ni-Alumina plating. We can see that the mild steel cover was coated with bright nickel.

4.3 HARDNESS TEST

The coating specimens were tested under Vickers indentation to evaluate the bonding strengths.. The fracture behaviors of the coatings were investigated through the response of coating cracking to the indentation load. The coating surfaces were polished before the Vickers indentation test in order to remove the loose or porousstructure in the top layer of the coating specimens. The indentation test was performed on the polished surface of each specimen using a standard Vickers machine with a diamond tip of 136 [degrees] angle with 500N load applied. Five indentations were taken on each specimen under each different distance on the plate. There are three graphs that plotted based on three parameters that decided earlier.

- Alumina Concentration Effect

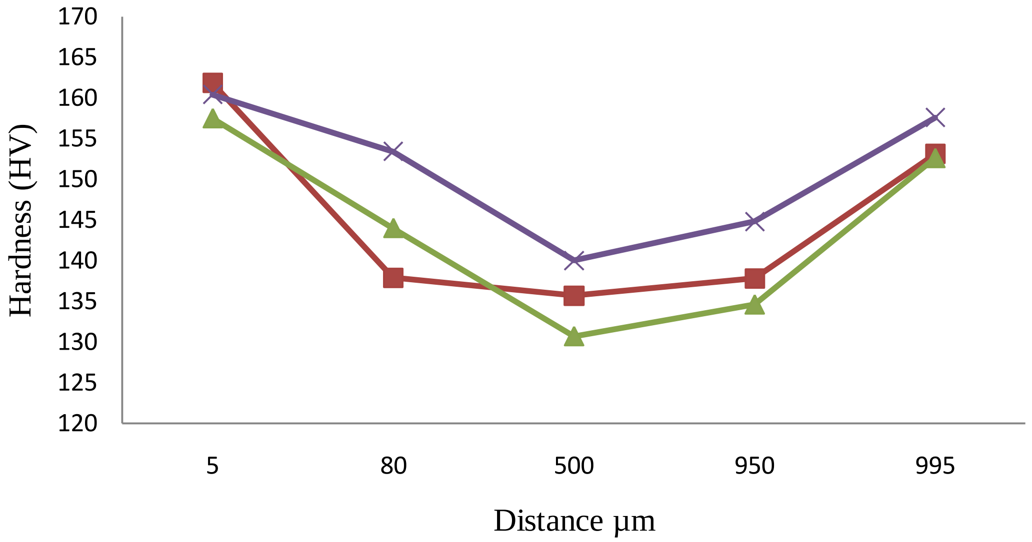


Figure 4.5: Graph effect of Alumina concentration on hardness (HV) vs. distance (µm)

The plotted graph was showing the hardness of Ni-Alumina electroplating specimen versus the distance which across the Ni-Alumina coated part to its original mild steel and back to the Ni-Alumina coated part.

Table 4.3 Vickers Hardness Test Value

	SET (HV)		
DISTANCE (µm)	**SET 1**	**SET 2**	**SET 3**
5	161.8	157.5	160.4
80	137.9	144	153.4
500	135.7	130.7	140
950	137.8	134.6	144.8
995	153.1	152.6	157.6

Figure 4.5 and Table 4.3 shows the result of Alumina concentration effect. It shows that on the Ni-Al_2O_3 electroplating part has higher hardness value than the

original mild steel. It conducted the average hardness value of 159.9HV at the initial position. Then, the hardness value jumped down to an average of 135.47HV at its original mild steel. The hardness value increased again critically to an average of 154.43HV at last. There was a big difference of the hardness value along the tested part which it originated from higher hardness value to lower hardness value and lastly back to its higher hardness value. This condition was because there were two different materials across the tested part of the mounted specimens. As a conclusion, the nickel electroplating part has higher hardness value at both end of the graph whereas the original mild steel has lower hardness value at the middle of the plotted graph.

As we know that Ni- Al_2O_3 is one of nano-composite which has good properties. Based on our result, it shows that the increase of Alumina concentration will also increase the thickness of coating. When the concentration is increased, more H+ ions are converted to hydrogen gas, which evolved from the cathode during the electroplating and the effect will occur. However, the concentration of Alumina is actually more use on its properties rather than the thickness. This is because the microhardness of the nano-composite coating increases with increasing weight percent of the Ni-Alumina nano-particulate.

In previous study, we know that microstructure and surface morphology of Ni-Alumina composite coatings, and it was shown that the Al_2O_3 particle distributed homogeneously in Ni matrix. As we know, the crystallite size of coating decreases remarkably with increasing of Al_2O_3 concentration in plating solution. That is to say, the incorporation of nanometer Al_2O_3 particulates could restrain the growth of Ni crystals, and result in grain refining. The codeposited particles appear to have an effect on the growth of deposited Ni crystallites, which may have resulted from: (i) increased numbers of nucleation of electrodeposits (ii) retarded growth of the Ni grains or (iii) change in the preffered growth orientations for grains.

Moreover, in nano-composite deposits, nano- Al_2O_3 particles embedded at the borders of Ni crystallites, as well as inside the the Ni crystal can help to prevent the corrosive pits from growing up, and the incorporation of nano- Al_2O_3 particles contributes to accelerate the passivation process of the matrix as well. Subsequently, the corrosion resistance of the Ni-Al_2O_3 nano composite coatings was improved.

Obviously, the nano-Al_2O_3 particles played a major role for improving the corrosion protection in three mechanisms. Firstly, these nano-Al_2O_3 particle act as inert physical barriers to the initial and development of defect corrosion, modifying the microstructure of the nickel layer and hence improving the corrosion resistance of the coating. Secondly, dispersion of nano-Al_2O_3 particle in the nickel layer results information of many corrosion micro cells in which the nano-Al_2O_3 particle act as cathode and nickel metal acts as anode because the standard potential of Al_2O_3 is more positive than nickel. Therefore, in the presence of nano-Al_2O_3 particle, localized corrosion is inhibited, and mainly homogeneous corrosion occurs. Thirdly, because the crystallite size of coating is in range of nanometer, corrosion must proceed along a circuitous path to reach the substrate, which retards the corrosion process, thus improving the corrosion resistance.

- Plating Time Effect

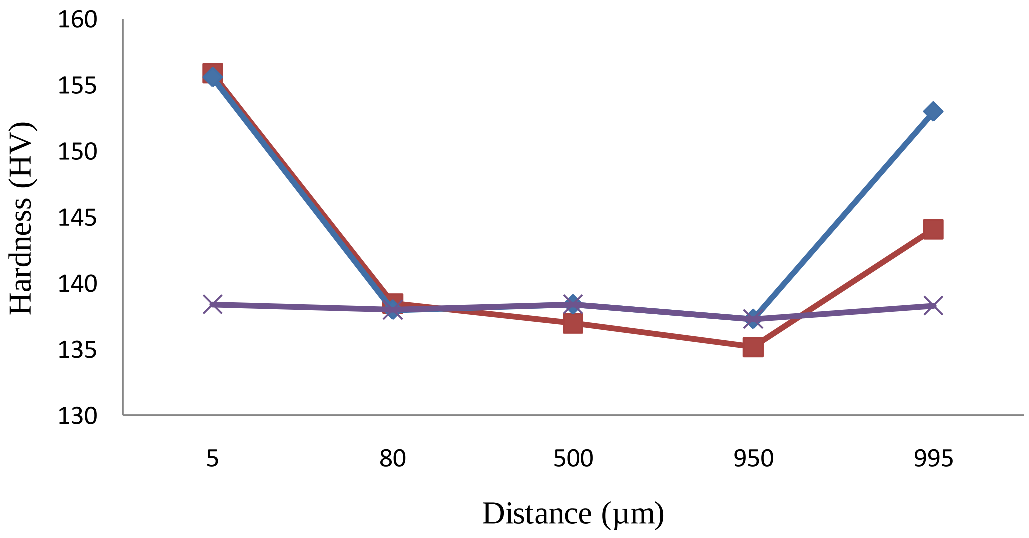


Figure 4.6: Graph Effect of Plating Time on Hardness (HV) vs Distance (µm)

Table 4.4 Vickers Hardness Test Value

	SET (HV)		
DISTANCE (µm)	**SET 1**	**SET 2**	**SET 3**
5	155.9	155.6	138.4
80	138.5	138	138
500	137	138.4	138.4
950	135.2	137.3	137.3
995	144.1	153	138.3

Figure 4.6 and Table 4.4 was showing the hardness of Ni-Alumina electroplating specimen versus the distance which across the Ni-Alumina coated part to its original mild steel and back to the Ni-Alumina coated part. The parameters that take place are plating time. Thus, the graph shows that on the Ni- Al_2O_3 electroplating part has higher hardness value than the original mild steel. It conducted the average hardness value of 149.97HV at the initial position. Then, the hardness value jumped down to an average of 137.9HV at its original mild steel. The hardness value increased again critically to an average of 145.13HV at last. There was a big difference of the hardness value along the tested part which it originated from higher hardness value to lower hardness value and lastly back to its higher hardness value. This condition was because there were two different materials across the tested part of the mounted specimens. As a conclusion, the nickel electroplating part has higher hardness value at both end of the graph whereas the original mild steel has lower hardness value at the middle of the plotted graph.

From our experiment, the thickness of coating will increase by electroplating time. the longer it takes, the thicker of coating that will got. When the electroplating time increase to 90 and 120 minutes, the self dissolution of the anodes were appeared more obviously. Thus, it can be said that for longer electroplating time, the more anodes will dissolute readily. Compare of these two samples for corrosion test it see like the sample with plating time 60 minutes will get corrode first. It is because for this sample it plated Ni-Alumina too thin and faster to get reaction with environment condition. Besides, plating time influence to get the structure better and smooth coating surface.

In addition, during the electroplating process, it was found that a black, poorly conductive and adhesive film, Ni $(OH)_2$ was deposited on the anode by the chemical reactions between Ni^{2+} and the products of oxygen, precipitation of $Ni(OH)_2$ at the anode due to increase in local pH from oxygen reduction, diminishes both nucleation and growth of nickel at cathode.

- Voltage Supplied Effect

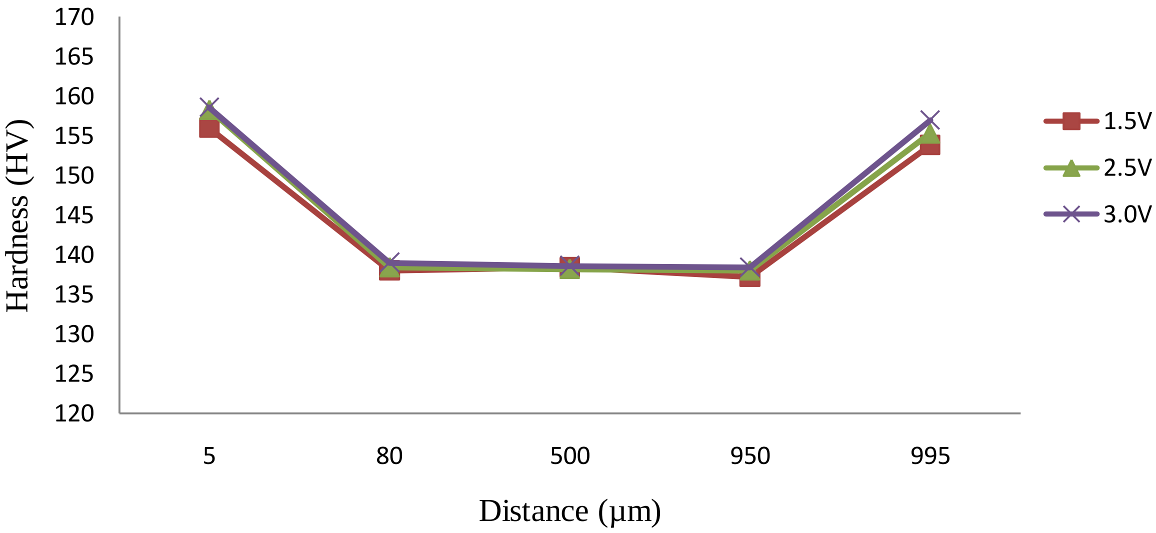


Figure 4.7: Graph Effect of Voltage Supplied on Hardness (HV) vs. Distance (µm)

Table 4.5: Vickers Hardness Test Value

	SET (HV)		
DISTANCE (µm)	**SET 1**	**SET 2**	**SET 3**
5	156	158.3	158.6
80	138	138.4	139
500	138.4	138.2	138.6
950	137.2	138	138.4
995	153.8	155.3	157

Lastly, Figure 4.7 and Table 4.5 was also showing the hardness of Ni-Alumina electroplating specimen versus the distance which across the Ni-Alumina coated part to its original mild steel and back to the Ni-Alumina coated part. However, this graph is study on voltages supplied parameter. The graph shows that on the Ni- Al_2O_3

electroplating part has higher hardness value than the original mild steel. It conducted the average hardness value of 157.63HV at the initial position. Then, the hardness value jumped down to an average of 138.40HV at its original mild steel. The hardness value increased again critically to an average of 155.36HV at last. There was a big difference of the hardness value along the tested part which it originated from higher hardness value to lower hardness value and lastly back to its higher hardness value. This condition was because there were two different materials across the tested part of the mounted specimens. As a conclusion, the nickel electroplating part has higher hardness value at both end of the graph whereas the original mild steel has lower hardness value at the middle of the plotted graph. It proves that increases of voltages supplied will also increase the thickness.

On last parameter, which is voltages supplied, the result shows that increase of voltages will increase the thickness of coating too. Actually, part of the current applied had been wasted during the electroplating operation. The explanation is that a small percentage of the current is consumed at the cathode in discharging hydrogen ions. Hydrogen evolution in the form of hydrogen bubbles, which was occurred during the electroplating process, adhered at the surface of cathode. It shows clearly that the bubbles of hydrogen that adherent at the surface of cathodes during electroplating operation retard the deposition of the productive nickel layer on cathodes.

The small red brownish holes were the rust formed when those parts were not protected by nickel from corrosion. The sample was produced was coated with dark brownish colored layer that was believed an oxide. The adhesive oxide film was adhered relatively stronger than the oxide films formed on anodes. It could be removed by using sand paper. It was further discovered that a bright nickel deposit was adhered strongly at the surface of the iron electrode, under the oxide film. The same observation was also occurring on other dull electroplated cathodes. Notice that those obvious bright scratches were nickel deposit under the oxide film.

4.4 CONCLUSIONS

The electrodeposited nickel depends on voltages supplied and plating time. The voltages supplied and Alumina content in solution influence the cathode efficiency. Increase of Alumina concentration, plating time and voltages supplied, the cathode efficiency increase. Alumina nano-particle can be successfully co-deposited with nickel by electro deposition. The cathodic polarization potential of the Ni-Alumina electrolyte increases with increasing Alumina concentration in the plating bath.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 INTRODUCTION

The amount of dissolution of anodes and deposition on cathodes, involving different electroplating condition in the experiment, was proportional to the electroplating time and voltage. The longer the electroplating, the more weight loss from anodes and the more weight gained on cathodes. Also, the higher voltage that supplied, the more weight loss from anode and the more weight gained on cathodes.

Electroplating produced a sticky and brownish deposit but still process acceptable corrosion resistance ability. During the electroplating process, it was found that a black, poorly conductive and adhesive film, $Ni(OH)_2$ was deposited on the anode by the chemical reactions between Ni^{2+} and the products of oxygen reduction.

From the result obtained it is proven that Alumina concentration, plating time, and voltage supplied increase will increased the thickness of coating and thus, give a better properties of coating on mild steel.

5.2 RECOMMENDATIONS FOR THE FUTURE RESEARCH

Based on Final Year Project that was done, a lot of experiences were gained during this project. About 10 months time was given for this final year project is a good duration for students to complete their projects which relates with the theoretical knowledge to the application in industry.

It is recommended to prepare the Ni-Alumina nano-particulate composite coating of the largest weight percent of alumina nano-particulates by plating in the bath containing higher amount of Alumina concentration. In this project, alumina concentrations don’t show much effect on coating. This is due to amount of Alumina powder used. Thus, increase of Alumina concentration will increase the efficiency. Besides, it is recommended to add on temperature parameter. In this project, parameter is set to be in room temperature. However, room temperature is only at 20-27’C and it will influence the result. The suitable temperature during electroplating will be 30’C. Previous research have proved that nano-composite coating have higher microhrdness and better wear resistance than the nickel coating, which is attribute to the grain fining and dispersive strengthening effects of the deposited hard nano-particulates. Thus, it is recommended to do wear resistance test on coating to prove nano-composite coating has good corrosion resistance.

5.3 CONCLUSION

Composite coatings consisting of nickel matrix and aluminum particles can be successfully fabricated by means of electro-deposition. The weight percent of Al particles in the coating increase with increase of Alumina concentration, plating time and voltages supplied. Consequently, with increasing of Al particle in the coating, microhardness of Ni-Alumina is better.

REFERENCES

Surender M, Balasubramaniam R, Basu B. 2004. Electrochemical behavior of electrodeposited Ni-WC composite coatings, Surf Coat Technol. 187;93-7

C.T.J.Low, R.G.A. Wills, F.C. Walsh. 2006. Surf. Coat. Technol. 201: 371-383

L.B Hunt. 1973. The Early History of Gold Plating.6 (1); 16-27

Leslie B. Hunt. 1984. The Mystery Of The Galvanic Goblet. 17(4); 140-141

Electrochemical Society, Frederick Adolph Lowenheim. 1963. Modern Electroplating Book: Wiley.

Arthur Kenneth Graham. 1971. Electroplating Engineering Handbook: Van Nostrad Reinhold Co.

H.Silman, George Isserlis, A.F.Averill. 1978. Protective and Decorative Coatings for Metals. Finishing Publications

Nasser Kanani. 2004. Electroplating:Basic Principle, Processes, and Practitice: Elsevier

W.D. Mogerman, 1974. Faraday's Lecture On Gold. 7(1); 22-24

Stojak JL, Fransaer J, Tabolt JB. 2002. Review of electrodeposition. In:Alkire RC, Kolb DM, editors, Advances in electrochemical science and engineering, Weinheim: Wiley-VCH Verlag: 193-23

Fontana MG. 1986. Corrosion engineering, 3rd ed, New York: McGraw-Hill Book Company

Guglielmi N. Kinetics of the deposition of inert particles from electrolytic baths, Electrochem soc 1972; 119:1009-12

Xi YT, Liu DX, Han D. 2008. Improvement of corrosion and wear resistance of AISI 420 martensitic stainless steel using plasma nitriding at low temperature, Surf coat Technol.202;2577-83

Bell T, Sun Y, Suhaidi A. 2000. Environmental and technical aspects of plasma nitrocarburising, Vacuum.59;14-23

Susan DF, Misiolek WZ, Marder AR. 2001. Reaction synthesis of Ni-Al based particle composite coatings, Metal Trans A.32A;379-90

Susan DF, Marder AR. 2002. Oxidation of Ni-Al base electrodeposited composite coatings, II: oxidation kinetics and morphology at 100 (degree) celcius, Oxidat Metal;57:159-80.

Sheng-Lung Kuo. 2005. Effect of Nickel Ion Concentration on Ni-Al_20_3 Composite Coatings. Journal of Chinese Institute of Engineering. 28; 1-8

V. Ganesh, D.Vijayaghavan, V. Lakshminarayanan. 2005. Fine Grain Growth of Nickel Electrodeposition: Effect of Applied Magnetic Field During Deposition. Applied Surface Science Journal. 240; 286-295

Denny Thiemig, Andreas Bund. 2008. Characterization of Electrodeposition Ni-TiO2 nanocomposite coatings. Journal of Surface & Coating Technology.202;2976-2984

Haifeng Liu, Weixing Chen. 2005. Electrodeposited Ni-Al Composite Coatings With High Al Content by Sediment Co-deposition. Journal of Surface & Coating Techology. 191; 341-350

M.R. Vaezi, S.K. Sadrnezhaad, L.Nikzad. 2008. Electrodeposition of Ni-SiC Nano-Composite Coatings And Evaluation of Wear and Corrosion Resistance And Electroplating Characteristics. Journal of Colloids and Surface A: Physlcochem Eng Aspects. 315;176-182

Qiuyuan Feng, Tingju Li, Haitao teng, Xiaoli Zhang, Yu Zhang, Changsheng Liu, Junze Jin. 2008. Investigation On The Corrosion And Oxidation Resistance Of Ni-Al2O3 Nono-Composite Coatings Prepared by Sediment Co-deposition. Journal of Surface & Coatings Technology. 202;4137-4144

N.Daemi, F.Mahboubi, H.Alimadadi. 2010. Effect of Plasma Nitriding On Electrodeposited Ni-Al Composite Coating. Journal of Materials and Design.32;971-973