

**OPTIMISATION OF ELECTROPLATING PARAMETERS
AND SURFACE DEFECT ANALYSIS**

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**FACULTY OF ENGINEERING
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OPTIMISATION OF ELECTROPLATING PARAMETERS AND SURFACE DEFECT ANALYSIS

ABSTRACT

Silver is a well-established metal used for coatings in various industries mainly in surface finishing of jewelries and decorative items. The coatings deposited on surface of substrate has to be smooth and even to ensure the coating deposited serves its purpose. There are various electroplating parameters that affect the surface roughness and surface defects of electroplated substrates. Therefore, this research is aimed to obtain the optimum parameters namely current density and plating time of silver cyanide electroplating process for bonded porcelain items produced in Royal Selangor. The optimisation was done by varying the current density and plating time using the Taguchi model to obtain the range of parameters for this study. The plated substrates were evaluated for surface roughness, coating thickness and adhesiveness accordingly. From the analysis done, as the current density and plating time increases, the surface roughness of the substrate surface increases due to increasing nucleation rate. This can be correlated with the surface roughness result and coating thickness images obtained from SEM whereby the coating thickness was also found to increase along with the increasing current density and plating time. Based on the results obtained from this study, it was found that the optimum current density was at 4A and plating time of 3 hours for the silver cyanide electroplating bath in Royal Selangor International.

Keywords: Silver plating, current density, plating time, surface roughness

PENGOPTIMUMAN PARAMETER PENYADURAN DAN ANALISIS

KECACATAN PERMUKAAN

ABSTRAK

Perak adalah logam yang mantap yang digunakan untuk pelapis dalam pelbagai industri terutamanya di permukaan penamat perhiasan dan barangan hiasan. Lapisan yang didepositkan pada permukaan substrat hendaklah lancar dan juga untuk memastikan salutan tersebut memainkan peranannya. Terdapat pelbagai parameter penyaduran yang menjejaskan kekasaran permukaan dan kecacatan permukaan bahan substrat. Oleh itu, penyelidikan ini bertujuan untuk mendapatkan parameter optimum iaitu kepadatan arus dan masa penyaduran proses penyaduran sianida perak untuk barang-barang porselin terikat yang dihasilkan di Royal Selangor. Pengoptimuman dilakukan dengan mengubah kepadatan arus dan masa penyaduran menggunakan model Taguchi untuk mendapatkan pelbagai parameter untuk kajian ini. Substrat bersalut dinilai untuk kekasaran permukaan, ketebalan salutan dan kekuatan pekat yang sesuai. Dari analisis yang dilakukan, apabila ketumpatan arus dan masa penyaduran bertambah, kekasaran permukaan substrat meningkat disebabkan peningkatan kadar nukleasi. Ini boleh dikaitkan dengan hasil kekasaran permukaan dan imej ketebalan salutan yang diperolehi dari SEM dimana ketebalan lapisan juga didapati meningkat seiring dengan peningkatan ketumpatan arus dan penyaduran semasa. Berdasarkan keputusan yang diperolehi daripada kajian ini, didapati ketumpatan arus optimum berada di 4A dan masa penyaduran selama 3 jam untuk penyaduran perak sianida di Royal Selangor International.

Kata kunci: Penyaduran perak, ketumpatan arus, masa penyaduran, kekasaran permukaan

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TABLE OF CONTENTS

Abstract	iii
Abstrak	iv
Acknowledgements	v
Table of Contents	vi
List of Figures	ix
List of Tables	x
List of Symbols and Abbreviations	xi
CHAPTER 1: INTRODUCTION	1
1.1 Introduction	1
1.2 Scope and objective of the research:	2
1.3 Objective of this study	2
1.4 Outline of the thesis	2
CHAPTER 2: LITERATURE REVIEW	4
2.1 Introduction	4
2.2 History of Electroplating	4
2.3 Electroplating Processes	5
2.3.1 Rack plating	7
2.3.2 Continuous Plating	8
2.3.3 Mass Plating	9
2.3.4 In-Line Plating Process	10
2.4 Faraday's Law	11
2.5 Electroplating Parameters	13
2.5.1 Current density	13

2.5.2	Plating Time	15
2.6	Surface Roughness.....	16
2.6.1	Arithmetic Average Height (R_a)	16
2.6.2	Root Mean Square roughness (R_q).....	17
2.7	Deposition Process.....	18

CHAPTER 3: METHODS AND MATERIAL 21

3.1	Introduction	21
3.2	Substrate Preparation (Cathode).....	21
3.3	Taguchi Approach	23
3.4	Silver bar (Anode) Preparation/Casting.....	24
3.5	Electrolyte (Cyanide bath) Preparation.....	25
3.6	Electroplating Process.....	26
3.7	Coating Characterization.....	26
3.7.1	Visual Observations	27
3.7.2	Tape Test	27
3.7.3	Surface Roughness Test	28
3.7.4	Scanning Electron Microscope (SEM).....	29

CHAPTER 4: RESULT AND DISCUSSION 30

4.1	Result and discussion.....	30
4.2	Visual Observation	30
4.3	Tape Test.....	32
4.4	Surface Roughness Analysis	36
4.5	Scanning Electron Microscope (SEM) Analysis.....	39

CHAPTER 5: CONCLUSION 43

5.1 Conclusion.....	43
REFERENCE	45

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LIST OF FIGURES

Figure 2.1: Silver-plated jewelries.....	7
Figure 2.2: Four basic types of plating rack.....	8
Figure 2.3: Cost reduction achieved with in-line plating.....	10
Figure 2.4: Variables affecting the rate of an electrode reaction.	12
Figure 2.5: SEM Imaging of Copper deposition at varied current density and time.....	15
Figure 2.6: Cu thickness at different plating times.....	15
Figure 2.7: Definition of arithmetic average height	17
Figure 2.8: Surface morphology of solid substance	18
Figure 2.9: Processes during bright plating.....	20
Figure 3.1: Process flow chart.....	21
Figure 3.2: Substrate preparation (phase 1)	22
Figure 3.3: Substrate preparation (phase 2)	22
Figure 3.4: Substrate preparation (phase 3)	23
Figure 3.5: Silver anode preparation.....	25
Figure 3.6: Electroplating process	26
Figure 3.7: Tape test method.....	28
Figure 3.8: Surface roughness test.....	29
Figure 3.9: SEM sample preparation	29
Figure 4.1: Peel Profile	33
Figure 4.2: Graph of surface roughness (average) against time	37
Figure 4.3: Graph of surface roughness (mid) against current density.....	37
Figure 4.4: Graph of surface roughness (mid) against time.....	38

LIST OF TABLES

Table 3.1: Array selector.....	24
Table 3.2: Process parameters and their levels.....	24
Table 3.3: Electrolyte composition.....	25
Table 4.1: Sample weight increment and visual observations	30
Table 4.2: Visual observations of samples.....	30
Table 4.3: Classification of Tape Test (Adhesion) Test Result	33
Table 4.4: Surface roughness test result.....	36
Table 4.5: SEM Images.....	40

LIST OF SYMBOLS AND ABBREVIATIONS

SEM	:	Scanning Electron Microscope
ASTM	:	American Society for Testing and Materials
CLA	:	Centre Line Average
BP	:	Bonded porcelain
Cu	:	Copper
Ag	:	Argentum (silver)
R _a	:	Arithmetic Average Height
R _q	:	Root Mean Square roughness
μm	:	micrometer
cm	:	centimeter

CHAPTER 1: INTRODUCTION

1.1 Introduction

Silver plating is a metal deposition coating process that is meant to increase the durability of substrates or to change the appearance of substrates. Silver plating is one of the oldest electroplating processes and widely utilized in multiple industries such as in aerospace, microelectronics, automotive, and jewelries (Liu et al., 2015). This is mainly due to silver's good corrosion and abrasion resistance, excellent physiochemical and magnetic properties, high bulk conductivity, and beautiful features for decorative purposes. Furthermore, silver plating process has been considered as one of the inexpensive and economically feasible coating techniques in industries (Rashidi & Amadeh, 2010). In this case, the plating bath consists of the electroplating metal, silver bar as the anode and a substrate, bonded porcelain as the cathode along with an external electrical supply to the bath to allow coating on the substrate. Silver anode dissolves readily in electrolytes and forms the silver deposit on the substrate. The thickness of the silver deposit is effected by the current density, pH of the bath along with the electrolyte concentration (Kumar, Pande, & Verma, 2015).

Accurate control of current distribution at a macroscopic level results in uniform surface coating. Other factors like proper agitation during plating along with uniform current distribution would result in a smooth coating finishing (Gupta et al., 2018). Also according to (Gupta et al., 2018), combination of a group of parameters that includes offset voltage, bath agitation, bath concentration and temperature and along with appropriate cathode-anode separation plays an important role in obtaining uniform current distribution. There are several types of electroplating baths that has been used in the past to obtain a bright silver deposit on the substrate. Some examples of sources of silver that has been used are provided by silver salts such as silver nitrate, silver halides,

silver sulfates, silver alkane sulfonates and silver alkanol sulfonates. The amount of silver salts that are being added in the bath depends on the desired silver to be deposited solutions as well as the parameters of the electroplating baths.

1.2 Scope and objective of the research:

Surface finishing after electroplating has become a major concern in industry due to inconsistent quality as the silver/crystal/dendrites formation on the sample surface is rough and uneven. Suitable electroplating conditions are required in order to form an even surface of silver deposits on the substrate being electroplated. The scope of this research is to obtain suitable operating conditions of silver electroplating on substrate by alternating the current density and plating time of the electroplating bath accordingly. The thickness of the electrodeposited coating would be analyzed with SEM.

1.3 Objective of this study

- 1) To identify suitable current density and plating time to obtain smooth surface finishing after electroplating.
- 2) To study the effect of current density on the thickness of deposited silver on substrate during plating.

1.4 Outline of the thesis

This thesis consists of five chapters. Chapter 1 presents of a brief overview about silver plating, identifying the gaps in the literature and establishing the objective of this research. The literature review is presented in Chapter 2. This chapter deliberates on the findings in the available literatures on the concept of electroplating, parameters affecting the process and strategies that have been employed for it's used as a presented in Chapter 3. The results gained in this research and discussions on the results are presented in

Chapter 4. In this chapter, the effect of electroplating current density on the surface roughness on the plated samples are studied and discussed. Along with that, scratch test and tape test are conducted in order to identify the adhesion strength of the coated silver metal on experimented substrate for varying current density and plating time. The results of the microstructure formed and surface roughness as a function of plating temperature is also deliberated. Finally, the conclusion draw from the present research is presented in Chapter 5.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

In this chapter, a summary of all the other pervious researches done on electroplating are discussed and elaborated deliberately.

2.2 History of Electroplating

The 18th century was the beginning of the science and art of new era electroplating. The first documented gold metal electrodeposition experiment was done from a saturated gold solution onto the surface of two large silver medals by Professor, Luigi V. Brugnatelli, an Italian University (Hunt, 2011). This set up was connected to the voltaic Zn/AgO pile invented in 1776 by Alessandro Volta. Three decades later, his work was revised to a similar process by scientists from Russia and England. They introduced the electroplating process of copper onto metal plates. A year later, Prof. J.F. Daniell rediscovered electroplating and deposition of uniform thin film coating. This has led to the discovery of electroforming when B.S. Jacobi has performed electrodeposition of metal onto engraved copper plate. In 1840, electroplating industry came to a limelight when Henry and George Elkington from Birmingham, England, were granted the primary patent for adjusting this innovation to gold and silver testimony utilizing potassium cyanide as an electrolyte. This prompted the foundation of Birmingham as the mechanical place for electroplating, where the principal enormous scale plant was developed on 1876. Afterward, Jacobi announced another noteworthy modern improvement on the utilization of progressively stable ferrocyanides in gold plating, consequently giving the ability of keeping gold compounds containing silver and copper. From that point on, electroplating rapidly spread all through the remainder of the world and turned into a typical procedure for keeping high value and non-high valuable metals, including silver, gold, nickel, copper, tin, and zinc.

Aside from the approach of electrical power age and hard chromium plating, couple of huge logical enhancements were made inside the late nineteenth and mid twentieth century. Rediscovery of electroplating happened after the Second World War with the revelation of transistors and the development of the hardware's business. In 1946 Abner Brenner and Grace E. Riddell found the primary autocatalytic metal affidavit (otherwise called "electroless" testimony) by adding sodium hypophosphite to a nickel shower. Later research on electroless affidavit profited a huge scope of modern divisions, specifically the metallization of printed wiring sheets. Parallel to the revelation of electroless testimony was the generation of multi-layered stores from a solitary arrangement. Noteworthy enhancements for alloyed and unadulterated multi-layers were sketched out by Tench and Yahalomin in the 1980s, along these lines delivering stores with one of a kind mechanical, electrical, optical, and attractive properties. Notwithstanding progresses in plating, more "easy to understand" and practical showers were actualized since the mid-1950s by supplanting cyanide showers with acidic arrangements. Today, a more profound information of the electrochemical perspective fundamental electrodeposition just as research on new materials and rising innovations are driving the customary assembling process towards an increasingly solid, adaptable and interconnected creations (Giurlani et al., 2018).

2.3 Electroplating Processes

With the advancement of research and development in Industrial Revolution Four, there is a high demand in metal finishing sectors around the world. Electroplating process has been one of the widely used surface finishing method in various industries extending from automotive industry till decorative applications (Giurlani et al., 2018). According to (Yli-Pentti, 2014), electroplating is a method of coating in which the material to be coated is set as the cathode. Cathode end is dip into an aqueous electrolyte containing metal salts.

On the other end, metal that is to be used for plating is set as the anode. In some cases, inert anode will be used as a substitute when the metal used for plating has been mixed into the aqueous electrolyte. The substrate to be coated has to be electrically conductive by nature or has to be treated to be conductive prior to the electroplating process. This process takes place with the presence of electric current passing through electrolyte containing the metal ion which is in contact with the substrate to be coated and metal used for coating.

Generally, metals substrates electroplated on an industrial scale are precious metals such as gold and silver, platinum, copper, iron, lead, chromium, cobalt, tin, zinc and also amalgams such as zinc-cobalt, lead-tin, nickel-iron and numerous gold amalgams (Horner,1994). The first reason for electroplating is to upgrade the estimation of metal articles by improving their appearance. Be that as it may, the significance of metal finishing for simply embellishing purposes has diminished. As of now, the trend is toward surface treatments which will bestow erosion resistance or specific physical or mechanical properties to the surface (for example conductivity, heat or wear resistance, soldering capability and lubricative properties), and henceforth to utilize less expensive substrate metals or plastics secured to give fundamental metallic surface properties or embellishing for covering purposes. In current industry, there are a few methods of electroplating that are still in practice depending on the size, structure and geometry of the samples to be plated. To name a few are rack plating, continuous plating, mass plating and in-line plating.



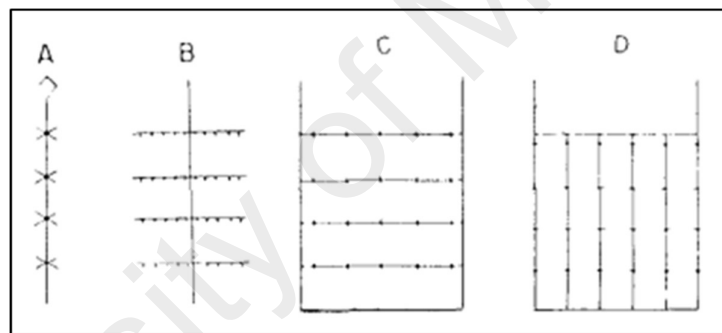
Figure 2.1: Silver-plated jewelries

(Source: Yli-Pentti, 2014)

2.3.1 Rack plating

Completed parts or semi-makes, which, in view of their size, shape or special highlights of development, can't be mass electroplated, are joined to racks, that is, installations reasonable for drenching in the plating arrangement. Accordingly mounted, these are exposed to an appropriate pretreatment and cleaning arrangement, at that point plated and in a few cases, exposed to a post-treatment. The procedure is at times known as batch plating. The connecting of segments to the rack are generally done physically, regularly by methods of copper wire. After this, the racks are moved, frequently by hand, to the first of the procedure tanks. After submersion for the recommended time, the rack is pulled back and proceeded onward to the following tank until the succession is finished. Lastly, the plated parts are altogether flushed and dried and taken to the stacking bay. Manual rack plating of this sort (alleged manual plant) is by definition work escalated

and therefore moderately costly. Be that as it may, it makes its mark when managing twisted or cylindrical parts. In such cases, the parts mounted on the racks must be drenched in arrangement and unsettled so that electrolyte buildups are totally depleted. A level of robotization is found in the supposed self-loader electroplating plant. In this, the racks after they have been physically passed from one tank to another tank, in which they are precisely upset, are then naturally returned, in the wake of emptying, to the start of the cycle. Much of the time, the racks are raised and brought down into the succession of tanks utilizing an overhead transport which is physically controlled. The fundamental bit of leeway of this methodology is the decrease in the measure of physical work involved (Kanani, 2007).



A - Spine B - T type C - Box type D - Multiple spine

Figure 2.2: Four basic types of plating rack

(Source: Heimke.S, 1999)

2.3.2 Continuous Plating

The electroplating of metal strip, cylinder and wire is done in continuous plating baths. In these, the things to be electroplated move consistently past either that one column or between two lines of anodes at a considerable rate. It pursues that working conditions in such plants might be totally not the same as those found in the comparable to batch plating processes. Provided the geometry of the substrate being electroplated is basic and even, the procedure can convey amazing outcomes. Provided with the high deposition rate

obtained in such plants, the fundamental stay time is a component of line speed and the length of the plating tank. For multiple reasons, it is important to arrange such plants as a progression of plating tanks through which the work passes, in a steady progression. As a result of the high current densities utilized, electrolytes utilized in such consistent procedures are described by their high metal particle fixation and high electrolytic conductivity. Since no extraordinary measure of tossing force is required, such electrolytes regularly contain no added substances. Being in all respects exceedingly mechanized, such plants regularly require at least supervision and support. A noteworthy bit of leeway of continuous plating lines emerges on account of plated metal strip. For this situation, coatings on either side of the metal plate may not be of a similar thickness, bringing about incredible investment funds in the utilization of the energy and crude materials (Kanani, 2007).

2.3.3 Mass Plating

Among the different sorts of segments which are plated, are little things, for example, screws or nuts. Rack-plating is anything but a possible suggestion in situations where throughputs might be a large number of things every day. They are in this way prepared by one or different strategies referred to on the whole as 'mass finishing'. Contingent upon their geometry, measurements what's more, shape, the plating of such things require special hardware. The most broadly utilized framework utilizes the plating barrel. Additionally, there are plating chimes and vibratory plating units. Mass plating, however it offers numerous preferences, it isn't reasonable for fragile parts where there is a threat of twisting, scratching, or entrapment. Right activity can limit such perils, and maintaining a strategic distance from over-stacking, under-stacking, utilization of right turn speeds and including 'balance' to the remaining task at hand are among the methods for guaranteeing ideal outcomes (Kanani, 2007).

2.3.4 In-Line Plating Process

An improvement as of late has been the combination of the plating and completing procedures into the principle creation line. This can bring numerous advantages incorporating a noteworthy decrease in use of eco-disagreeable materials. In-line plating likewise permits some pretreatment stages to be overlooked, on account of nearer control of the plating and generation lines and this again can decrease the utilization of synthetic substances. The principle profits by utilizing this methodology, both financial and ecological can be outlined as pursues:

- Investment funds in synthetic substances utilized
- Diminished effluent release
- Complete reusing of synthetic compounds utilized in the process arrangements
- Reduction in energy utilization

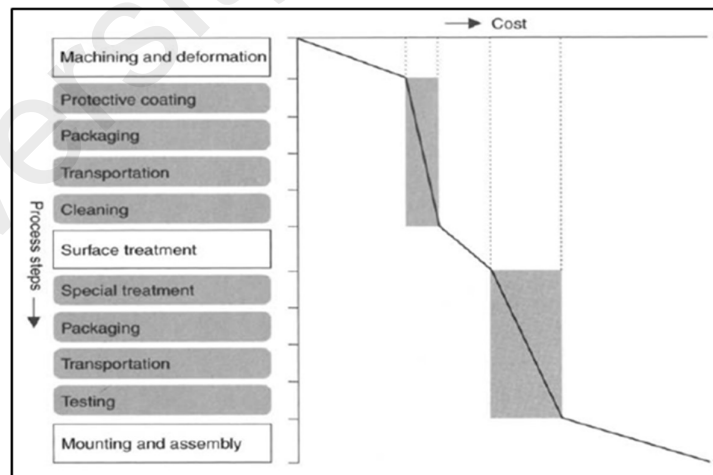


Figure 2.3: Cost reduction achieved with in-line plating

(Source: Kanani, 2007)

From this Figure 2.3, it tends to be visible that utilizing this methodology, the venture costs for capacity and transport of materials are essentially diminished. What's more, the cost incurring procedures which might be required to shield a surface being moved starting with one area then onto the next would then be able to be disposed of (Kanani, 2007).

2.4 Faraday's Law

In 1833, laws of electrolysis was set up by Faraday, which are the establishment of both the better-known disintegration and electrodeposition methods. Faraday's laws of electrolysis are quantitative connections dependent on the researches on electrochemical by Michael Faraday in 1834. The most widely recognized proclamations of Faraday's law look like the following:

Faraday's first law of electrolysis: The mass of a substance changed at an electrode during electrolysis is legitimately relative to the amount of electricity moved at that cathode. Amount of power alludes to electrical charge, ordinarily estimated in coulombs, and not to electrical flow.

Faraday's second law of electrolysis: For a given amount of power (electric charge), the mass of an essential material changed at an anode is straightforwardly relative to the component's proportional weight. The proportionate load of a substance is its molar mass separated by a number that relies upon the reaction experienced by the material. The mathematical form of Faraday's laws can be represented by

$$m = \left(\frac{Q}{F}\right) \left(\frac{M}{z}\right)$$

where m is the mass of the substance altered at an electrode, Q is the total electric charge passed through the substance, $F \approx 96.485 \text{ C mol}^{-1}$ is the Faraday constant, M is the molar

mass of the substance, and z is the valance number of ions of the substance (electrons transferred per ion). Note that M/z is the same as the equivalent weight of the substance altered. For Faraday's first law, M , F , and z are constants, so that the larger the value of Q the larger will m be. For Faraday's second law, Q , F , and z are constants, so that the larger the value of M/z (equivalent weight) the larger will m be.

In the simple case of constant current electrolysis, $Q = It$ leading to equation as below,

$$m = \left(\frac{I \cdot t}{F} \right) \left(\frac{M}{z} \right)$$

where n is the amount of substance ("number of moles") altered, $n = m/M$, and t is the total time the constant current was applied (Ivanov, Leese, & Spieser, 2015).

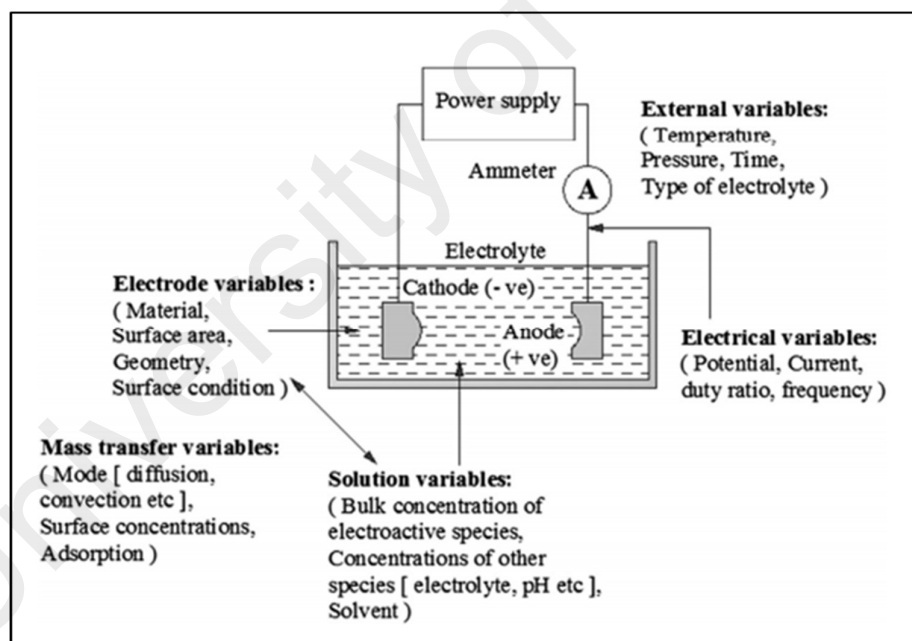


Figure 2.4: Variables affecting the rate of an electrode reaction.

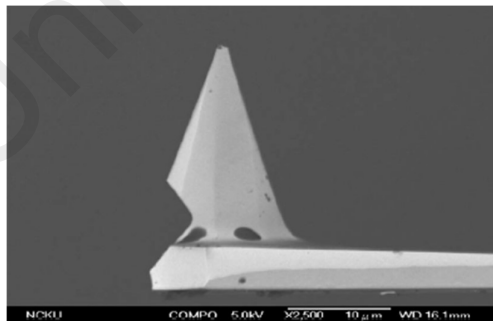
2.5 Electroplating Parameters

There are several operating parameters that will affect properties of the deposits formed. Parameters to be considered includes current density, concentrations of the electrolyte used, temperature of the bath, addition of additives and also mechanical agitation. Though individual parameters play significant role in obtaining coating deposits of certain properties, combination of parameters does play a part in optimizing the coating deposits needed. Thus, it is crucial to analyze the parameter in combination in order to obtain the expected result (Kim, Yu, Lee, & Lee, 2004).

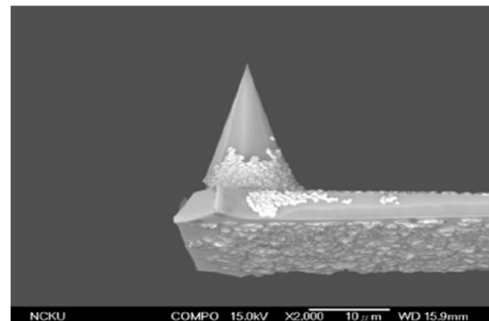
2.5.1 Current density

Current density is one significant parameter that has to be controlled in order to obtain a uniform deposition of metal on substrate. The current density utilized during the testimony assumes a noteworthy job in deciding the crystallite size and deformities in the covering and thus microstructure of the equivalent. In (Augustin, Huilgol, Udupa, & Bhat K, 2016), it is unequivocally seen that the coating covering area expanded as current density increased. At exceptionally low current density, coating is intermittent and underneath substrate is unmistakable in between the coated material. However, studies from Arun et al. also states that the with increase in current density, there is a decline in size of crystallite formed, in which this could be due to the increasing nucleation rate throughout the deposition process. Expanding current density will build the motion of diffusive radicals to the sample's surface. Hence, local extinguishing and arrangement of nanocrystallites are increasingly fast (have shorter occasions for development of nanocrystallites), and the average size nanocrytallites will be reduced. A study on the effect of current density on electrodeposition of copper has shown that a slow increase in current density has resulted in a uniform deposition of copper on the substrate. The arrangement of uniform copper deposits on the substrate could be ascribed to the

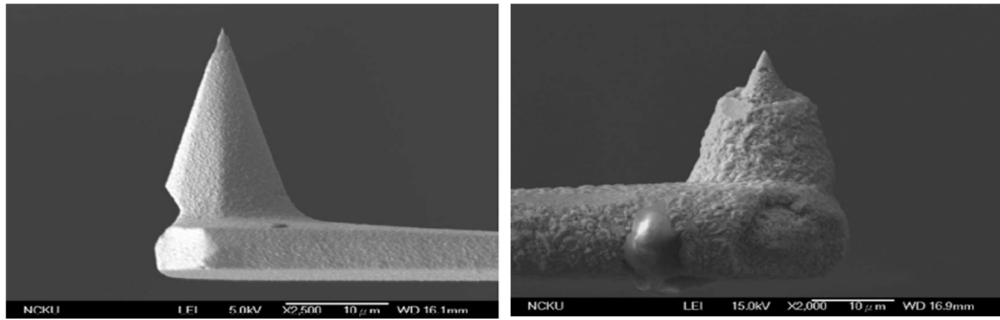
improved mass transfer of copper particles with the rise in current density (Lin, Chen, & Mohanty, 2008). A recent study from the Journal of Materials Engineering and Performance has proven that with increasing current density, the thickness of deposited metal layer increases linearly. An increase in current density from 4 to 10mA/cm² gives an average thickness of deposited layer between the range of 0.93 to 15.98μm (Jung, Sharma, Kim, Choo, & Jung, 2015). The number density of copper ions will increase and therefore the deposition rate is increased with a rise within the electrode current density. this can be in line with the opposite reports that the presence of a lot of metal ions at higher electrode current density will increase the deposition rate appreciably. The current dispersion at the cathode surface greatly affects the nature of the coating. The current circulation is commonly isolated into primary and secondary current dispersion. The source of the essential distribution is in the geometry of the surface and its impact on current flow. No polarization is contemplated, just the size, shape, and distance of the plated sample, anodes, and tank dividers and surfaces, the conductivity of the metal ions, electrolyte, and anodes. Edges and corners will have higher current distribution, prompting a thicker coating thickness or even burned coating. Despite what might be expected, hollows or breaks will have a slenderer covering than normal or may even be left exposed.



(a) Current density: 0.03A/dm², Time: 300s



(b) Current density: 0.3A/dm², Time: 540s



(c) Current density: 0.6A/dm², Time: 300s

(d) Current density: 0.6A/dm², Time: 900s

Figure 2.5: SEM Imaging of Copper deposition at varied current density and time

Source: (K.Lin et al., 2008)

2.5.2 Plating Time

Plating time is another parameter that has to be controlled in parallel with current density. Many studies have proven that plating time adversely affects the thickness of electrodeposited metal on the substrate (Jung et al., 2015). The increase in coating thickness with increasing plating time suggests that there is a linear increase in deposition rate with time. Figure 2.6 also depicts clearly the correlation between plating time and thickness of coating on the substrate.

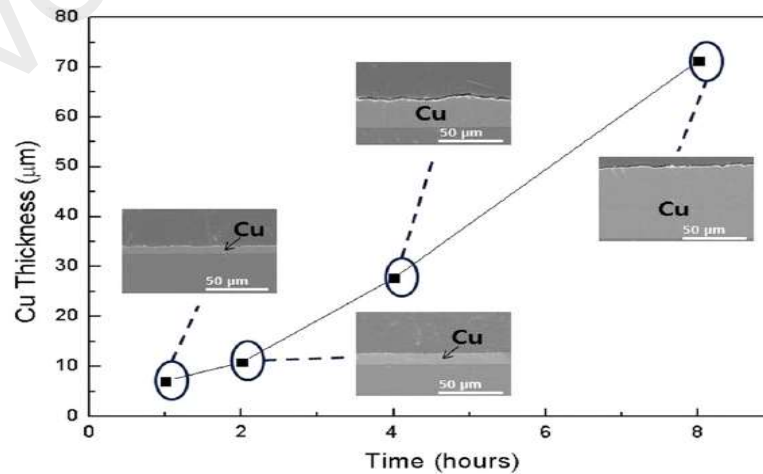


Figure 2.6: Cu thickness at different plating times

(Source: Jung et al., 2015)

2.6 Surface Roughness

Surface roughness is the proportion of the finely dispersed smaller scale abnormalities on surface which is made out of three parts, in particular waviness, form and roughness (Ali & Hung, 2016). Roughness parameter can be measured in either two-dimensional (2D) or three-dimensional (3D) structure. However, in current publications emphasis has been placed in the significance of 3D surface morphology and topography in science and engineering practices. This parameter is normally linked with the grain size of electrodeposited coating (Saitou, 2010). Surface roughness parameters of the material have a huge job in the development of contact angle hysteresis, the genuine infinitesimal deviations of incline superficially structure hindrances due to pin the movement of the contact line and change the contact angle (Hebbar, Isloor, & Ismail, 2017). It is a significant parameter that influences the tribological conduct of surfaces, e.g., asperity interlocking, or distortion can expand friction (Freyman, Zhao, & Chung, 2007). Amplitude parameters are the most significant parameters to describe surface geography. They are utilized to gauge the vertical qualities of the surface deviations.

2.6.1 Arithmetic Average Height (R_a)

Arithmetic average height factor is the most commonly used roughness parameter in quality control. It is also known as the centre line average (CLA). It is characterized as the normal total deviation of the harshness anomalies from the mean line more than one examining length as appeared in figure below.

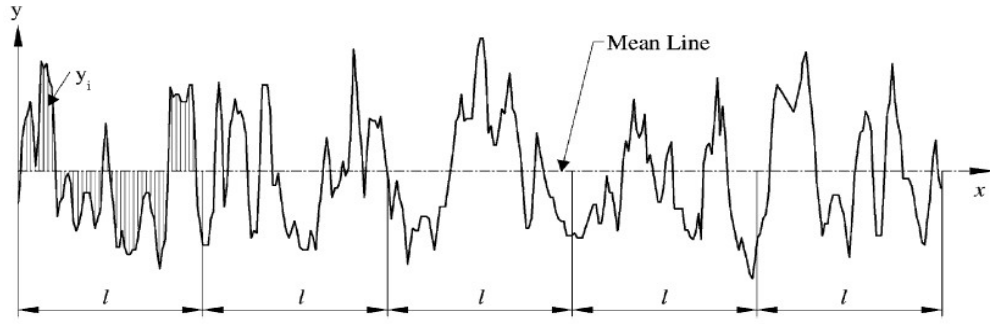


Figure 2.7: Definition of arithmetic average height

(Source: Gadelmawla et.al, 2002)

The statistical definition and numerical average height parameter are as shown below.

$$R_a = \frac{1}{l} \int_0^l |y(x)| dx$$

$$R_a = \frac{1}{n} \sum_{i=1}^n |y_i|$$

(Source: Gadelmawla et.al, 2002)

2.6.2 Root Mean Square roughness (R_q)

R_q is one significant parameter in defining surface roughness. It is defined as the standard deviation of distribution of surface heights. This factor is much more sensitive as compared to R_a to vast deviation from the average line. The statistical definition and mathematical application of this factor are as shown below. The RMS mean line is the line that partitions the profile with the goal that the whole of squares of the deviation of the profile range from it is equivalent to zero.

$$R_q = \sqrt{\frac{1}{l} \int_0^l \{y(x)\}^2 dx}$$

$$R_q = \sqrt{\frac{1}{n} \sum_{i=1}^n y_i^2}$$

(Source: Gadelmawla et.al, 2002)

Diverse assembling procedures produce distinctive surface qualities. Likewise, various applications require distinctive surface properties. Thus, surface parameters are extraordinary and wide running. Every one of these parameters shows a specific property of the surface and it could be the most significant for specific application.

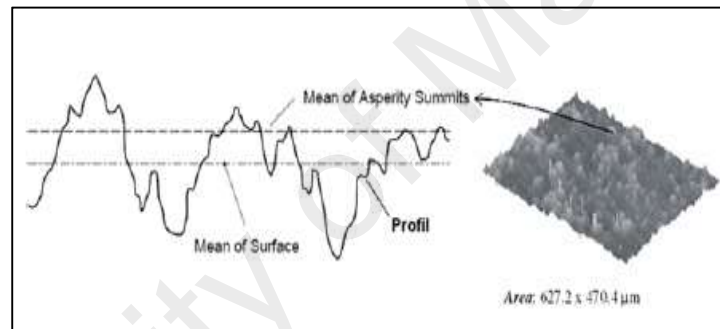


Figure 2.8: Surface morphology of solid substance

(Source: Rachman, Risdiyanto, Khayam, & Suwarno, 2013)

2.7 Deposition Process

At the point when metallic material is plunged into the electrolyte arrangement in a plating cell, a potential difference will be produced among anode and cathode. At the point when no current is gone through the cell, the potential difference relies upon the electrode potential, temperature, and the concentration, or all the more effectively, the exercises of the species associated with redox reactions. The activities of the ions may contrast from their concentrations due to the interatomic impacts and particularly because of the intricate agents. The action of complex particles is much lower than straightforward

or hydrated particles or ions, which affects electrode potentials. At the point when the outside power source is exchanged on, the potential between terminals will change to another value. The terminals become energized. At the cathode surface, the concentration of the metal particles will be diminished. A dispersion layer will shape on the surface of the terminal. Its thickness in a solution relies upon agitation, deposition rate of the metal particles, and temperature. Regularly, the thickness is a few divisions of a millimeter. The applied current density influences the rate of deposition. The dispersion does not give enough mass exchange through the layer, and a potential distinction over the dissemination film will be framed. Inevitably, when the potential at the outer power source is raised, the concentration of the deposited particles will be zero at the metal surface and the restricting current density has reached. The current density will become further increased just by side responses, for example, hydrogen development. Utilizing potentials higher than at constraining current density, the surface coating quality will endure effects significantly, prompting 'burnt' surfaces or then again fine powdery deposits on the substrate surface. At the outside of the metal, there is a layer that is a couple of atoms thick, where hydrated metal particles are adsorbed on oppositely charged metal surfaces. It is known as the Helmholtz twofold layer after the creator of the model. The charge transfer happens inside this locale. At the point when the charge transfer happens, the neutral metal particles will diffuse superficially toward the metal cross section and take shape to have a lively positive state, as shown in figure below. Explicitly adsorbed anions and surfactants may affect the crystal structure (Yli-Pentti, 2014).

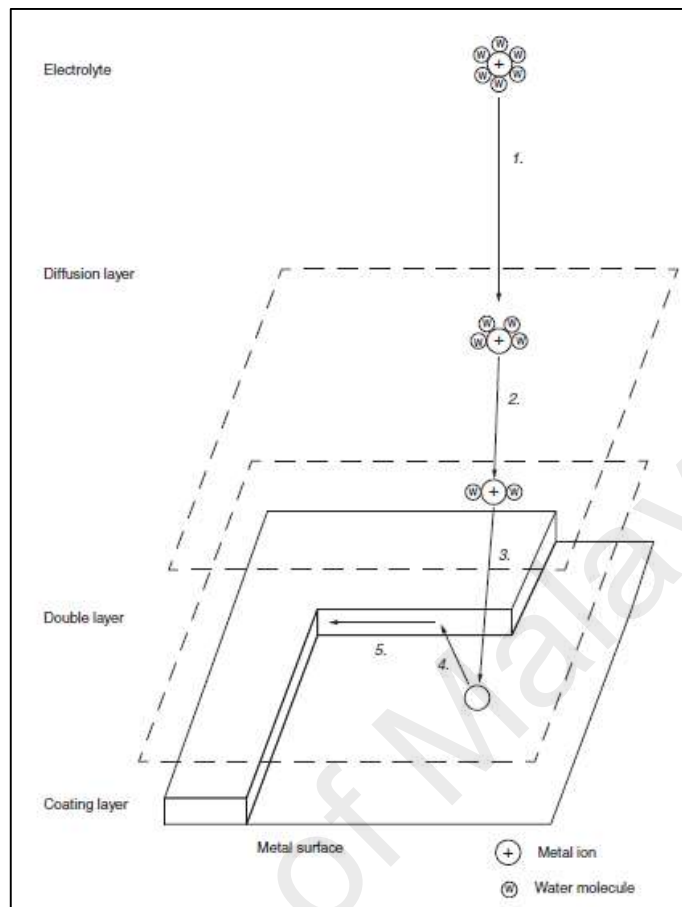


Figure 2.9: Processes during bright plating

(Source: Yli-Pentti, 2014)

Figure above shows five steps or processes during bright plating. The first process shows the convection of hydrated metal ion from bulk solution. Then, diffusion happens towards the cathode surface through diffusion layer, where part of the hydrated water molecules will be lost. Thereafter, charge transfer happens in the double layer, metal ions will be neutralized electrically, and more hydrated water will be lost. Following this, surface diffusion of metal atom happens and finally crystallization into coating metal lattice occurs.

CHAPTER 3: METHODS AND MATERIAL

3.1 Introduction

This chapter describes the process and equipment by which substrate, which is the bonded porcelain is silver plated. This process includes cleaning the bonded porcelain, spray painting, washing with deionized water prior to dipping into the electroplating bath at varied current densities and plating time. In addition to this, adhesion of the coating toward the substrate is tested using the tape test. Also, the surface roughness of plated samples was obtained using the Digital Surface Roughness tester TR100. Scanning electron microscopy (SEM) were used to view and analyze thickness of coating produced at varied current density and plating time of coated samples that passed the tape test. The process flow of the work is schematically shown below.



Figure 3.1: Process flow chart

3.2 Substrate Preparation (Cathode)

Substrate used in this study is bonded porcelain, each with a plating surface area of 1.0 dm². The bonded porcelain was obtained from Royal Selangor (Selangor Pewter).

Samples are checked for any surface defects or uneven surfaces on the body. If there are any uneven surface present on the substrate body, sandpaper of grade 800 and 1200 is used to smoothen even out the surface. It is then weighed and the weight of all the samples obtained is engraved at the back of the samples using a hand tool. A small hole is drilled at the back of the sample to attach a hook. After drilling, hook is then attached to the sample accordingly. It has to be ensured that this hook is secure enough to hold the sample. Pictures are as shown below in Figure 3.2.

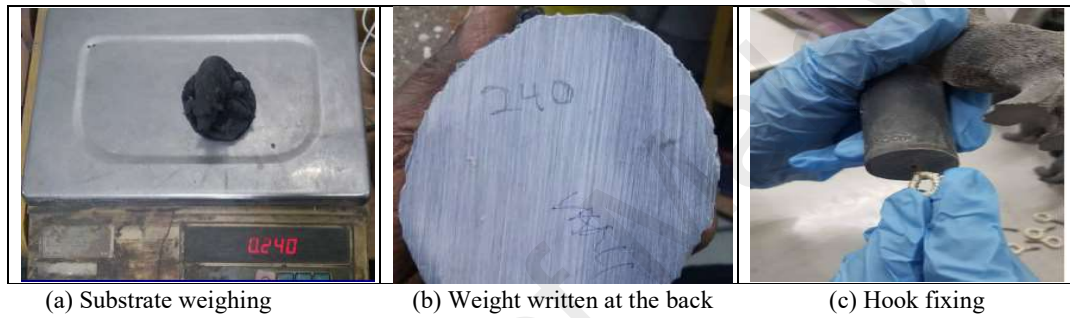


Figure 3.2: Substrate preparation (phase 1)

A copper wire is then attached to the hook and a knot is tied at the end. This wire will be the conductor between the rectifier and substrate in order to allow the current flow. Following this, the samples are cleaned using clean water and soap to remove dust particles or dirt from the surface of substrate. Pictures are as shown below in Figures.

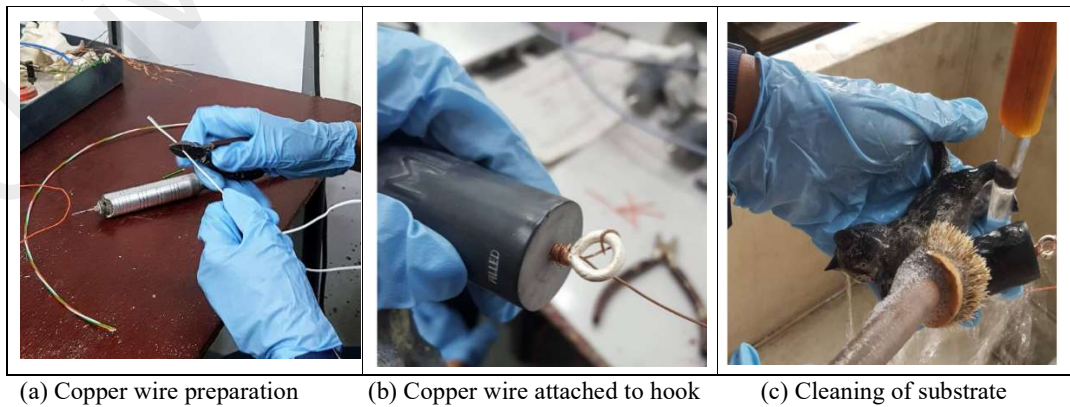


Figure 3.3: Substrate preparation (phase 2)

Samples are then left to air dry for at least 30 minutes. To ensure samples are fully dried, a pneumatic air blow gun is used to blow off excess water from the surface of samples to be plated. After that, dried samples were painted with silver paint using a paint brush. Samples are further painted using a spray paint gun to ensure the entire surface of the sample is covered with silver paint. In this study, substrates have been painted with silver paint to ensure the substrates used are conductive. Only conductive samples are able to go through the electroplating process. After spray paint, the samples are then allowed to dry for not more than 30 minutes. Once the paint has dried, samples are rinsed in water rinse tank prior to dipping into electroplating bath.

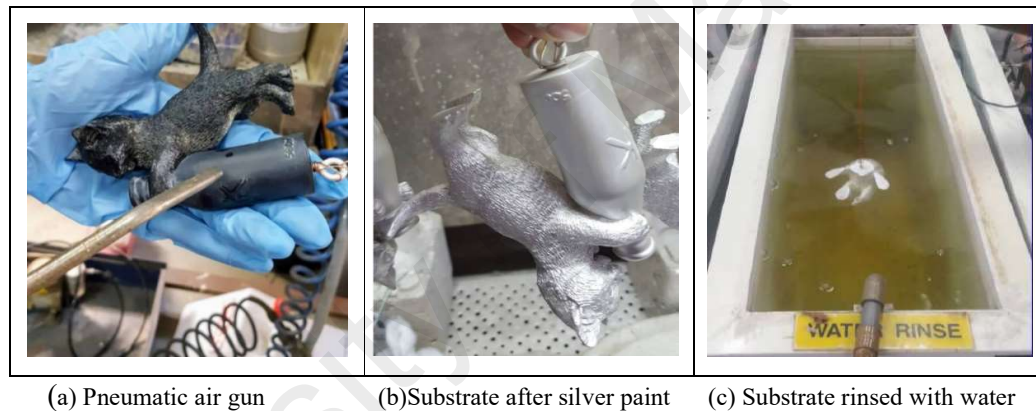


Figure 3.4: Substrate preparation (phase 3)

3.3 Taguchi Approach

In this study, the Taguchi model is employed to determine the optimum combination of plating parameters namely current density and plating time to obtain a smooth surface finish with minor or no surface defects. Orthogonal array L9 has been used to identify the set of parameters for this study based on the array selector of Taguchi model referring to Table 3.1. Three levels having equal spacing within the operating range of current plating conditions were selected for each factor studied. Range of parameters are as shown in Table 3.2 below.

Table 3.1: Array selector

L E V E L S	PARAMETERS											
		2	3	4	5	6	7	8	9	10	11	12
	2	L4	L4	L8	L8	L8	L8	L12	L12	L12	L12	L16
	3	L9	L9	L9	L18	L18	L18	L18	L27	L27	L27	L27
	4	L16	L16	L16	L16	L32	L32	L32	L32	L32		
	5	L25	L25	L25	L25	L25	L50	L50	L50	L50	L50	L50

(Source: Kondapalli, Chalamalasetti, & Damera, 2017)

Table 3.2: Process parameters and their levels

Factor	Level 1	Level 2	Level 3
A (Time, hr)	1	2	3
B (Current, A/dm²)	3	4	5

3.4 Silver bar (Anode) Preparation/Casting

Silver anodes are typically provided with a virtue of 99.97% or higher. Two grades of silver bar are commonly available, a normal grade and a premium quality grade. For the best plating outcomes, the fantastic evaluation anodes ought to be utilized. These anodes are produced using chosen silver grains or silver melts and are prescribed by the anode providers for ideal execution in the plating bath.

Contaminations in silver anodes can cause the arrangement of dark film and chipping which result in plate unpleasantness. In this study, silver anode was prepared using 99.98% silver grain. These silver grains were melted in a crucible in a furnace. Molten silver is then poured into a mold which forms the ingot shape. The molten silver will then

solidify over time. The silver bar is then left to further cool down before being used in the electroplating bath.

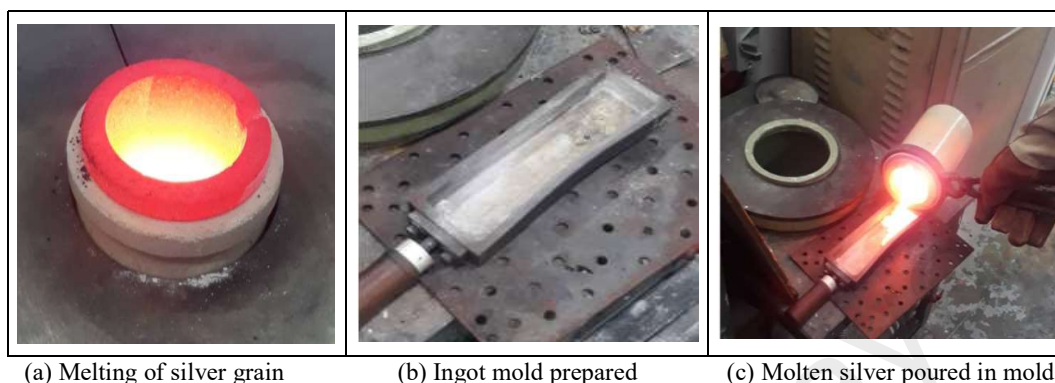


Figure 3.5: Silver anode preparation

3.5 Electrolyte (Cyanide bath) Preparation

The experiment was carried out in an electrolyte bath with 5000ml silver electrolyte solution in a 6000ml tank. The chemicals used to prepare this silver electrolyte includes potassium silver cyanide, silver cyanide and brightener (Silvor '003). Compositions of the silver bath is as shown in the table below. Temperature of the bath is maintained at 25 – 30°C.

Table 3.3: Electrolyte composition

Parameter	Value
Potassium silver cyanide salt (g/L)	30g/L
Potassium cyanide (g/L)	100g/L
Brightener (g/L)	0.05g/L

(Source: Blair, 2015)

3.6 Electroplating Process

The process of electroplating begins with placing the silver anode in the electrolyte bath which is connected to a rectifier. Silver anodes are placed in a steel basket with synthetic fabric woven bag. The weave of the texture ought not confine arrangement stream to cause anode polarization. After the anode has been placed in the bath, painted substrates that has been rinsed are placed in the electrolyte bath by tying the knot at a metal rod with the connected copper wire. Knot has to be tightened to ensure the substrate does not drop into the tank.

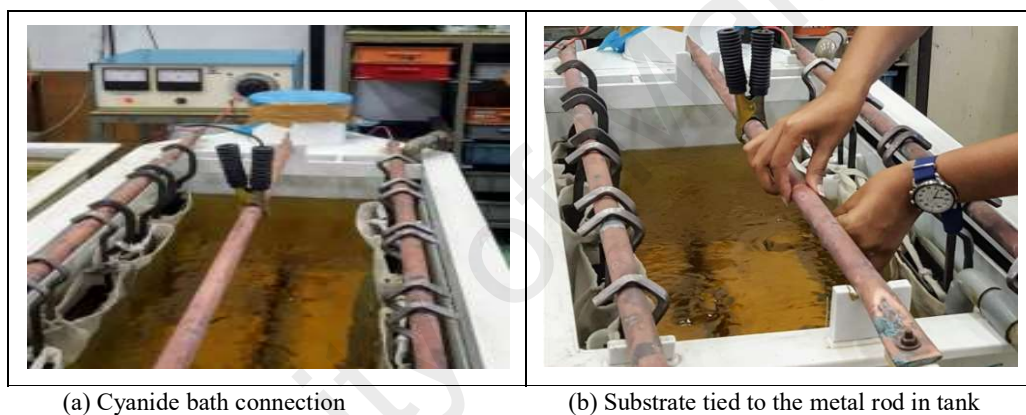


Figure 3.6: Electroplating process

The effect of current density and plating time is studied together. Current density was varied from 3A/dm^2 to 5A/dm^2 (with 1A/dm^2 variation in between) and plating time was varied from 1 hour to 3 hours (with an hour variation in between). After plating is completed, substrate is rinsed with clean water to remove excess chemicals. Also, rinsing prevents the substrate from discoloration.

3.7 Coating Characterization

Coating characterization of obtained samples are done by visual observation for any surface defects, tape test method to analyze the adhesiveness and SEM imaging to obtain the thickness of coating deposited on each sample to optimize the parameters accordingly.

3.7.1 Visual Observations

Visual observations are conducted on coating on the substrates. This includes colour, smoothness or roughness of coating, appearance and adhesion of coating to substrate. Visual observations are also done before and after the tape test to determine the adhesion of coating to substrate base.

3.7.2 Tape Test

A 25 mm wide semi-transparent, cellulose weight delicate sticky tape (3M make, type 250YT) for a length of 75 mm was used for the tape test (Gupta et al., 2018). Adhesion of coating to the substrate was assessed based on any strip off of the coating by percentage of area removed by the tape. Tape test was repeated for all the coated samples after performing a cross-hatch. Surface of the substrates are ensured to be clean and free of minor surface defects and uneven surfaces. Six to eleven parallel cuts are made on the coating surface with 2mm space cuts. All cuts are made to about 20mm long at a steady state of motion in which there is just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. After the cuts are made, any detached flakes or ribbons of coatings are removed with a soft brush or soft tissue. Before initiation of testing, two complete laps of the tape are removed and discarded at each day of testing. Thereafter, a 75mm piece of tape is cut and the center of the tape is placed at the area of grid and smoothen by finger or with the eraser at the end of pencil. After smoothening and within 60s of application, free end of the tape is pulled off quickly without breaking the tape at an angle as close as possible to an angle of 180. The grid area on substrate is inspected for any peel off visually. Rate of adhesion is reported based on the classification of adhesion test results from ASTM International Standard Test Method D3359-09 2014. Depiction of the tape test is as shown in the figure below..

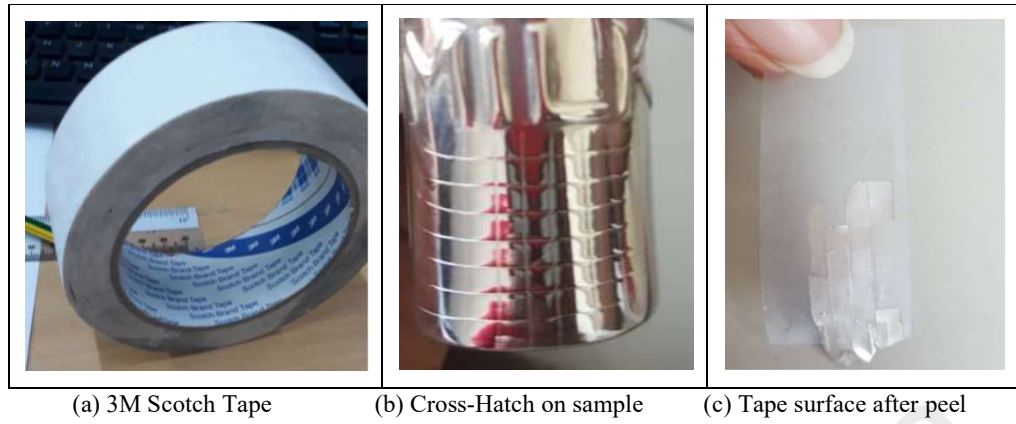


Figure 3.7: Tape test method

3.7.3 Surface Roughness Test

Surface roughness of the plated substrates were tested using the Digital Surface Roughness tester TR100. Before initiating the measurement, the device is calibrated accordingly to avoid errors during measurement since the micro stylus of this device is very sensitive. For measurement, the device is placed onto the measuring surface which is our substrate. Once start button is pressed, the micro-stylus scans over the coating and measured the surface roughness. R_a = roughness average value is immediately displayed on the device once scanning is completed. Roughness average value is obtained when the profile heights are measured from a reference line which is shown in the figure below. The formula used to obtain the arithmetic mean is stated in equation below. Each coated substrate with varied current density and plating time was measured and recorded accordingly.

$$R_a = CLA = AA = \frac{1}{L} \int_0^L |z - m| dx$$

(Source: Gadelmawla, 2002)

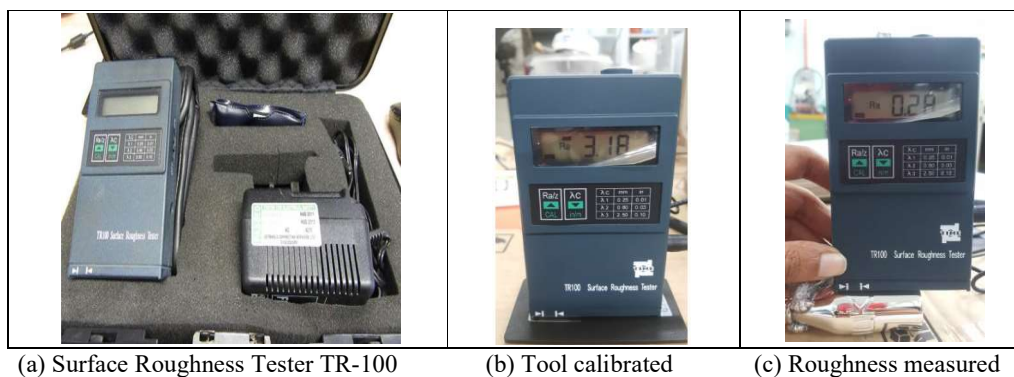


Figure 3.8: Surface roughness test

3.7.4 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) was used to view and measure the coating thickness of substrates that has been electroplated with varying current density and plating time. Cross-sections were made on substrates in order to obtain the sample piece for SEM. Samples obtained were embedded in EpoFix resin and left to harden overnight. This is known as the mounting step. Mounting enables easier handling of the samples during examination. Hardened epoxy resin containing sample is polished with abrasive polishing paper starting with grade 800, followed by grade 1200 and finally grade 2000 to achieve a smooth surface. Smooth surface of the sample will ensure a good viewing result is obtained under the microscope. Care has to be taken to not over polish the sample surface. Polished samples are then placed on the metal stub. Sample preparation for SEM is as shown in figure below.

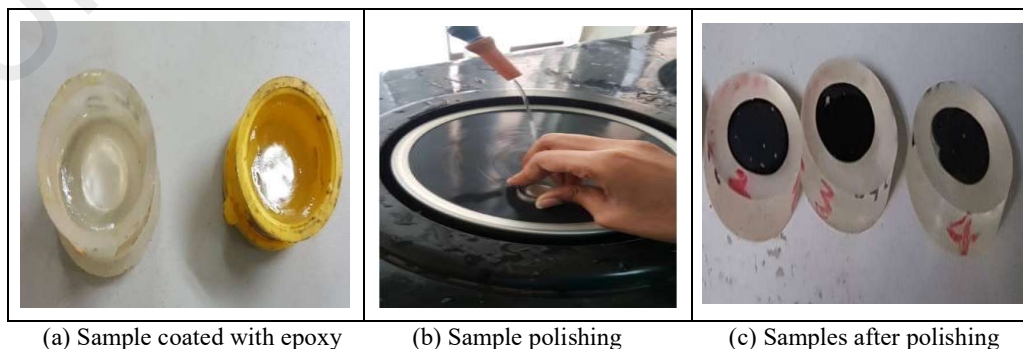


Figure 3.9: SEM sample preparation

CHAPTER 4: RESULT AND DISCUSSION

4.1 Result and discussion




This chapter will present the results on test methods that has been conducted. Discussions based on the results obtained are also laid out accordingly.







4.2 Visual Observation

Table 4.1: Sample weight increment and visual observations

Sample	Parameter		Weight before (g)	Weight after (g)	Differences (g)	Sample Appearance
	Current (A)	Time (hr)				
1	3.00	1.00	171.250	180.383	9.133	Peel off, Dull
2	3.00	2.00	176.250	195.330	19.080	Shiny
3	3.00	3.00	185.000	213.112	28.112	Shiny
4	4.00	1.00	187.500	201.432	13.932	Minor peel off, Shiny, Uneven coating
5	4.00	2.00	197.500	226.682	29.182	Shiny
6	4.00	3.00	185.000	225.416	40.416	Shiny
7	5.00	1.00	185.000	202.655	17.655	Shiny
8	5.00	2.00	185.000	216.575	31.575	Shiny
9	5.00	3.00	170.000	218.393	48.393	Shiny

Table 4.2: Visual observations of samples

Sample	1	2	3
Picture			

Sample	4	5	6
Picture			
Sample	7	8	9
Picture			

*Table 4.2 continued

Table 4.1 above shows weight of substrate before and after electroplating and visual observation or appearance of sample surface at varied current density and plating time. The increase in sample weight after plating corresponds linearly with the increasing current density and plating time. It can be observed that with equal current density, there is an increase in weight of plated sample with increasing plating time. To illustrate further, taking the example of plating current density at 3A, it can be perceived that at plating time of 1 hour, there is an increase of 9.133g, whereas at plating time of 2 hours, there is an increase of 19.080g meanwhile at plating time of 3 hours, there is a hike of 21.112g in substrate after coating. From Table 4.1, it is also explicitly observed that with increase in current density, there is also an increase in substrate weight though plating time is maintained. At constant electroplating time of 1 hour, weight of samples obtained by varying current densities at 3A, 4A and 5A are 9.133g, 13.132g and 17.655g respectively. This is in line with the study from (Güler, 2016) which states that particle content of the coating increases linearly with the increase in current density. Least increase in weight after plating was viewed at plating conditions of 3A and 1 hour which is at 9.133g whereas highest increase in weight was obtained at plating conditions of 5A and 3 hours which is at 48.393g. Also, at current density of 3A and plating time of 1 hour, it was observed that

the substrate coating tends to peel off and crack after drying whereas at 4A current density and equal plating time which is at 1 hour, there is minor peeling effect observed. At 5A current density and a plating time of 1 hour, no peeling effect or crack was observed and much thicker coating has been produced. From literature review, it has been reported that with increase in current density, there is an increase in coating coverage (Augustin et al., 2016) on substrate surface which is in line with this study. Also, increase in weight with increasing current density and plating time is in accordance to the Faraday's Law that has been discussed in Chapter 2. No usual surface defects were observed on any samples except for samples that were plated with current density of 3A and 4A and plating time of 1 hour. These samples which showed peeling effect and crack were observed to be thin and coating was uneven on substrate surface. Mild rough surfaces were also observed with these two samples. Overall, seven out of nine of the samples appear to be shiny and an even coating was observed visually with naked eye. The results obtained on Table 4.1 indicates that at higher current density and higher plating time, there is an increase in mass transfer of Ag^+ ions thereby enhancing the deposition of Ag on surface of substrate.

4.3 Tape Test

Seven out of nine samples that has passed the visual test was used for tape test. The results obtained are presented below. As discussed in Chapter 3, the results were inspected and tabulated according to the ASTM standard of classification of adhesion. The grid below shows the surface of cross-cut area from which flaking or peeling has occurred for six parallel cuts. Percentage of area removed determines the rate of adhesion of silver coating on substrate accordingly. Results are shown in Table 4.3 below accordingly.

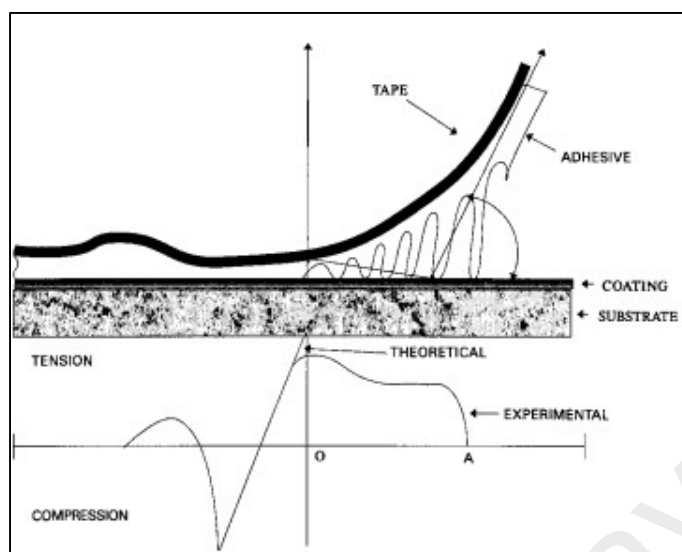
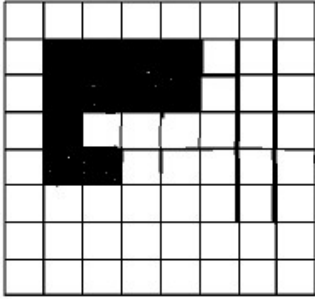
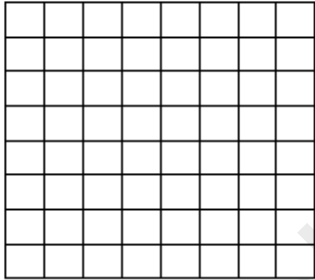
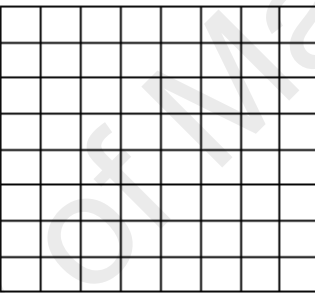
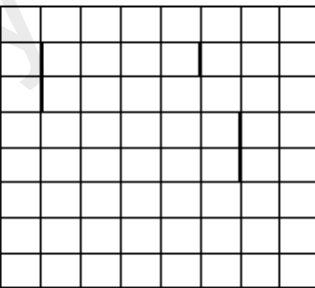
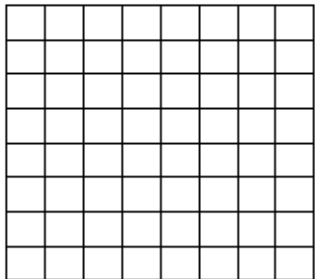
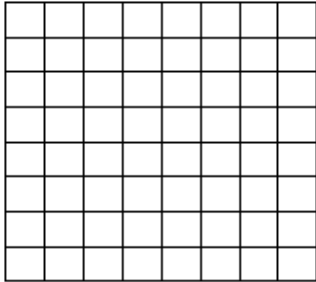


Figure 4.1: Peel Profile

Table 4.3: Classification of Tape Test (Adhesion) Test Result

Sample	Operating conditions	Surface of cross cut area observation (6 parallel lines)	Percentage of area removed
1	3A, 1 hour		Greater 77.78% (Very poor)
2	3A, 2 hours		Greater 64% (Very poor)
3	3A, 3 hours		0%

4	4A, 1 hour		Greater 69.4% (Very poor)
5	4A, 2 hours		0%
6	4A, 3 hours		0%
7	5A, 1 hour		Less than 5%
8	5A, 2 hours		0%

9	5A, 3 hours		0%
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From Table 4.3, there are 4 samples that shows flaking or peeling effect after the tape test has been conducted namely sample 1 (3A, 1 hour), sample 2 (3A, 2 hours), sample 4 (4A, 1 hour) and sample 7 (5A, 1 hour). It is an obvious observation that sample plated with operating conditions of 3A and 1 hour shows 77.78% of flaking or peeling effect which is the highest percentage recorded as compared to all the other samples followed by sample 4 (4A, 1 hour) at 69% and sample 2 (3A, 2 hours) at 64%. Sample 7 (5A, 1 hour) shows minor peeling effect which is recorded at less than 5% of total surface area tested. All the other samples show no peeling or observable flaking hence percentage of area removed recorded as 0%. For sample 1, flaking was already observed when the cutting was performed even before the tape adhesion test was conducted. This shows the adhesion on the first sample is regarded as very poor in which the coating peels and debonds just by cutting into it. The results obtained can be used to determine the adhesion of coating done toward substrate surfaces. From these, it can be deduced that sample plated at operating conditions of 3A and 1 hour shows the least adhesion of coating to substrate surface. Also, substrate plated at 4A and 1 hour shows slightly higher percentage of flaking as compared to sample 2, plated at operating conditions of 3A and 2 hours. However, substrate plated at 5A and plating time of an hour shows very minor flaking effect. It can inferred that effect of current density and plating time play equally significant role when these 2 samples are compared. This is due to substrate plated at higher current density and lower plating time tends to have more flaking effect as

compared to substrate plated with lower current density and higher plating time. It is evident that higher current density contributes to a thicker deposition of silver coating on substrate surface over time. This is agreed upon the statement which states that increasing current density increases the current efficiency in the bath and hence increases the deposition rate of silver ions in the electroplating bath (Lin et al., 2008).

4.4 Surface Roughness Analysis

In this study, the data for surface roughness was obtained using the Digital Surface Roughness tester TR100. Three measurements were taken for each substrate sample and an average of the readings were also recorded. Table 4.3 shows the tabulation of data recorded.

Table 4.4: Surface roughness test result

Current Density (A)	Time (hr)	Surface Roughness, Ra (unit)			
		1 (Top)	2 (Mid)	3 (Side)	Average
3	1	2.38	0.36	0.73	1.157
3	2	1.76	0.65	0.26	0.890
3	3	1.32	0.20	0.28	0.600
4	1	1.38	0.24	3.76	1.793
4	2	2.52	0.20	0.50	1.073
4	3	0.29	0.39	0.33	0.337
5	1	0.60	0.50	0.23	0.443
5	2	3.24	0.23	0.29	1.253
5	3	3.92	0.23	0.24	1.463

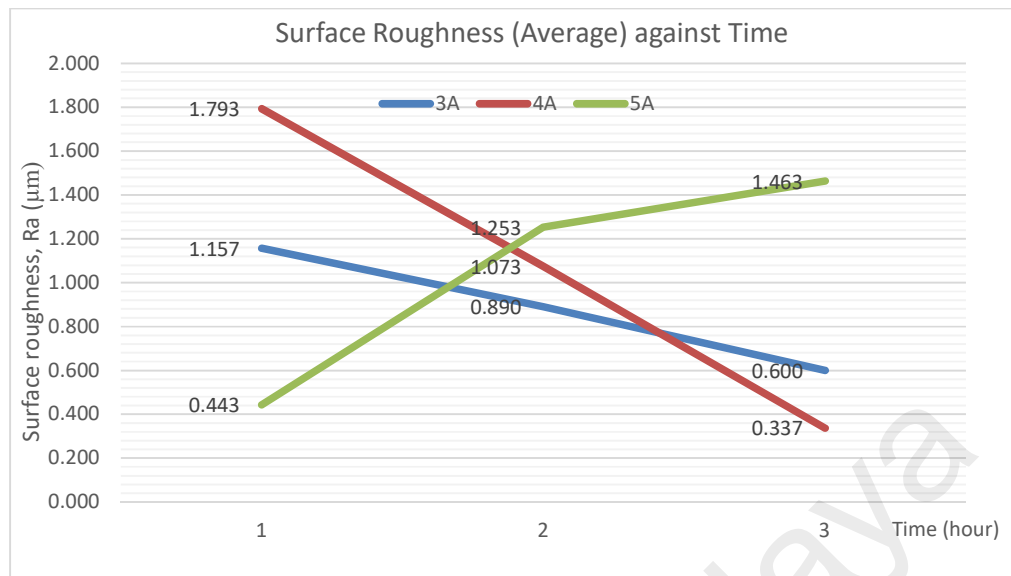


Figure 4.2: Graph of surface roughness (average) against time

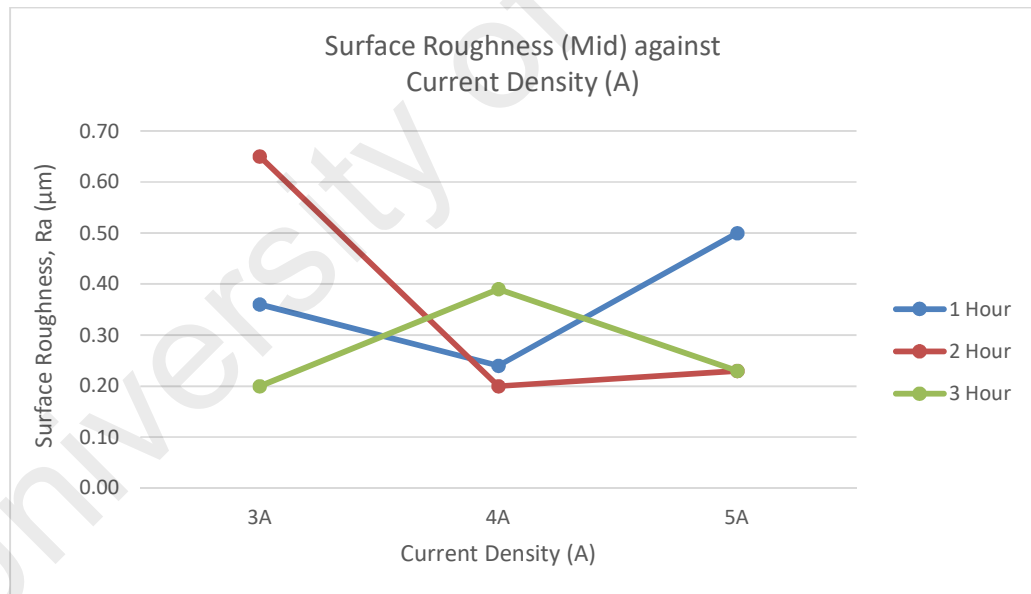


Figure 4.3: Graph of surface roughness (mid) against current density

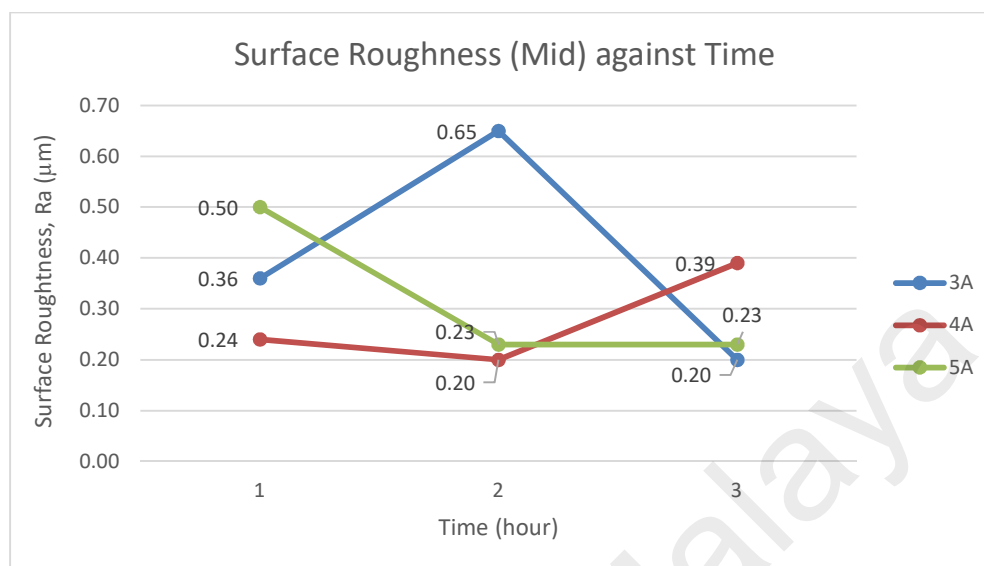


Figure 4.4: Graph of surface roughness (mid) against time

In general, smaller values of surface roughness measurement show a smoother and even surface whereas bigger values give indications of rougher surfaces. Based on average results obtained from Table 4-3, sample plated at 4A and 3 hours shows the smallest value of surface roughness which is at 0.337. This is followed through by substrate plated at 5A and 1 hour giving surface roughness value of 0.443 and finally substrate plated at plating conditions of 3A and 3 hours shows a reasonable smooth surface giving a value of 0.600. The highest roughness measurement was obtained at sample plated at 4A and plating time of 1 hour which is at 1.793 on average. However, referring to these average data gained, we are unable to deduce the correlation between surface roughness and plating conditions of current density and plating time. This is further explained by the data obtained for roughness measurement at the top of the samples which shows exceptionally higher values as compared to other parts of the substrate namely side and middle area of sample surface. It can be hypothesized that surface roughness before plating at the top area of the substrate was higher as compared to the middle and side area of the substrate. This could have resulted in the exceptionally

higher roughness value at particularly one area on the substrate surface (Jung et al., 2015). Since deposition rate is defined as the ratio of coating thickness to deposition time, it could be reasoned that due to edge effect, the deposit roughness and deposition rate at the sample edges are larger than in the middle. Thus, surface roughness measurements at the middle of the substrate surface were further analyzed.

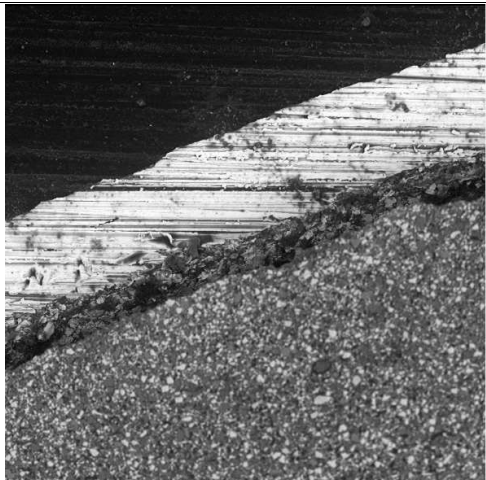
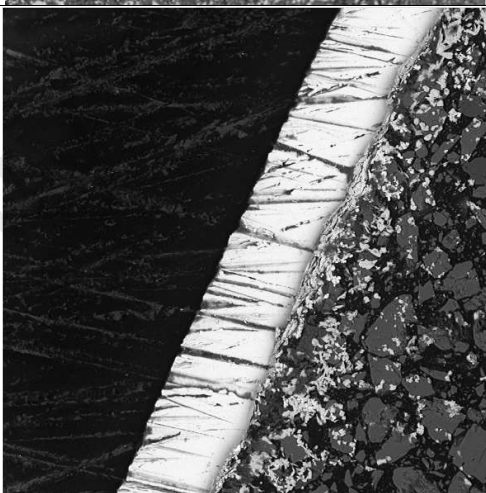
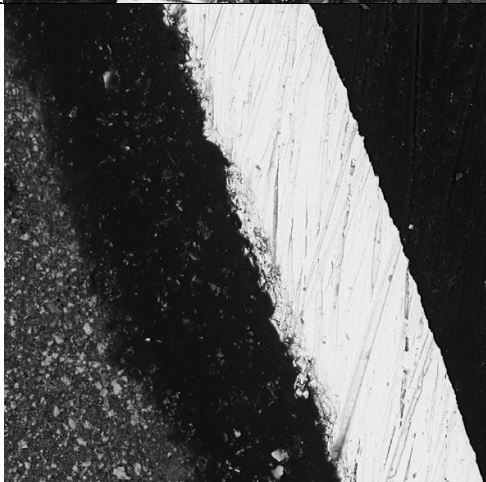
In Figure 4.4, there is a trend that is quite obvious. When current density is increased from 3A to 4A, it is observable that the surface roughness value reduced at plating time of 1 hour and 2 hours. Increasing current density further to 5A results in reduction of surface roughness value for all. This can be explained in accordance to the theory that states with increasing current density, the deposition rate increases. The hike in current density leads to overpotential and hence results in the increase in nucleation rate. The nucleation rate is low when the current density is relatively small. With a low nucleation rate, it provides the crystal nucleus with bigger space to grow resulting in formation of uneven surfaces. However, when there is an increase in deposition rate along with current density, crystal nuclei will grow synchronously and uniformly resulting in an even surface formation. On the other hand, it is a noteworthy point to be taken into account that when the current density is too high, micro-discharge normally takes place at a very sharp angle in view to the reason of lack of discharge of metallic ions near the cathode. This would result in larger grains formation and surface coarsening (Ren, Yin, Wang, Volinsky, & Tian, 2013). This is observed in results obtained for plating condition of 1 and 2 hours, as the current density increased from 4A to 5A, the surface measurements increased too.

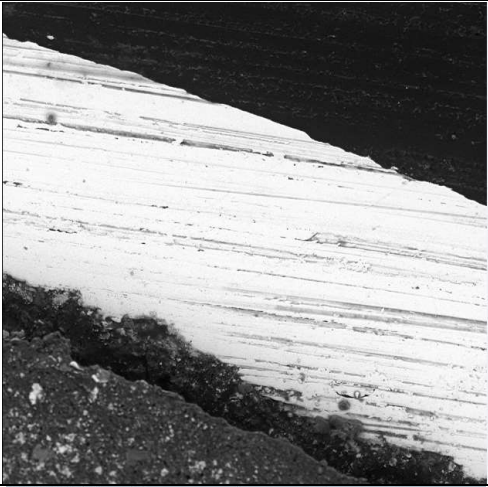

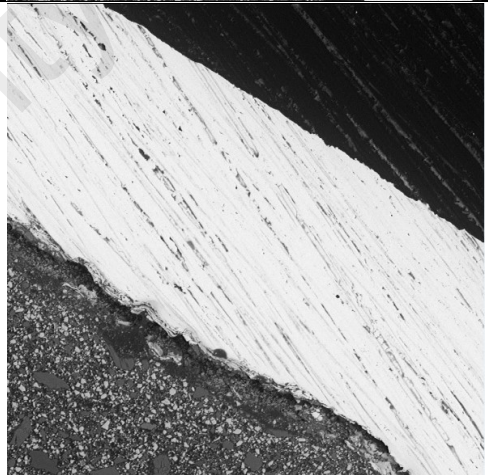
4.5 Scanning Electron Microscope (SEM) Analysis

For SEM analysis, sample 1 (3A, 1 hour) and sample 4 (4A, 1 hour) was not analyzed in view of the results from tape test which showed very poor adhesion of coating to substrate surface. Thus, SEM results for all the other samples showing the coating

thickness obtained by viewing the cross-section of substrates are tabulated here. For this analysis, images at magnification of 2000X was used to compare.

Table 4.5: SEM Images

Sample	Plating Conditions	Image	Coating Thickness (μm)
2	3A, 2 hours		30.437
3	3A, 3 hours		54.957
5	4A, 2 hours		50.979

6	4A, 3 hours		66.436
7	5A, 1 hour		40.396
8	5A, 2 hours		56.482

9	5A, 3 hours		122.683
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The thickness obtained on each sample are also tabulated in Table 4.3. Highest thickness was obtained for sample 9, substrate plated at 5 A and 3 hour which is at 122.683 μm . This is parallel with the results obtained for weight of silver deposited on sample 7 which also shows the highest among the rest. The thinnest coating was observed on sample 2 (plating conditions: 3A, 2 hours) which is at 30.437 μm . Though substrate plated at 3A and 1 hour was not analyzed, it can be deduced that the thickness of its coating was thinner as compared to sample 2 the coating peeled off and flaked even before the tape test was conducted. This again proves that with increasing current density, there is an increase in deposition of silver on substrate surface. Also, from the SEM images, it is quite obvious that as the current density increases, the coating seems to be denser and compact on substrate surface as compared to coating at lower current density (Ren et al., 2013). Along with that, it was also noticeable that silver coatings at higher density shows a brighter appearance as compared to those of lower current density and plating time though with naked eye, all the samples coatings had nearly the same brightness and shininess.

CHAPTER 5: CONCLUSION

5.1 Conclusion

The present study aims at optimizing the electroplating parameters, namely current density and plating time for Royal Selangor and investigating the surface defects of plated substrates. This electroplating study was done for a cyanide bath silver plating which is currently being practiced in Royal Selangor. Substrates were electroplated at a varying current density ranging from 3A to 5A and plating time of 1 hour to 3 hours in accordance to the factorial design model and Taguchi method. Visual observations of surface defects and coating appearance was done for all the samples to serve as the visual quality inspection. Following that, adhesion of the coated silver was measured by doing the tape test which complies to the ASTM standard test method. The findings obtained from the aforementioned investigations give a good indication of adhesion of deposited silver on our substrate. Apart from that, measurements of surface roughness using the Roughness Tester TR-100 was obtained and coating thickness was analyzed to obtain the optimized parameter for silver plating of bonded porcelains (substrate).

The findings from the surface roughness measurement shows that with a lower current density of 3A, there is a tendency to have a rough and uneven coating surface on plated substrate. However, as the current density increases to 4A, the surface roughness reduces and a more even coating was formed. Exceeding the optimum level of current density and plating time results in a rougher and crackled surface and this was observed in samples plated at current density of 5A and plating time of 1 hour. These results were confirmed via surface roughness analysis done on all the plated samples. Based on SEM analysis on the coating thickness of silver deposits on substrate, the thickest or highest attribution of thickness was observed at samples plated at 5A and plating time of 3 hours. This result indicates the highest deposition occurs at highest current density and plating time. SEM

image of this sample also exhibited a brighter and denser coating image compared to the other samples.

In the present study, samples plated at current density of 4A and plating time of 3 hours exhibited the most reasonable result for all the test methods and investigations conducted. It has a medium surface roughness at 0.36 μm and passed the surface adhesion test. Also, the weight of silver deposited on the substrate is about 40.416g which corresponds to the approved weight of silver deposits on substrate in Royal Selangor. Thus, based on the results obtained from all the investigations done, it can be concluded that current density of 4A and plating time of 3 hours are the optimized electroplating parameters for bonded porcelain silver plating in Royal Selangor.

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