DIFFERENT COATING TYPE ON COPPER SUBSTRATE

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DIFFERENT COATING TYPE ON COPPER SUBSTRATE

by

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Specially dedicated to my beloved family and friends
for their
unconditional love, care and support
in all stages of my life

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ABSTRACT

This thesis will deal with the effect of different coating types on solder joint strength, specifically on intermetallic compound (IMC). Coating is essential in printed circuit board application since it can protect the base material, which is Copper, from oxidation and degradation. However, the coating thickness will give different effect on solder joint strength or life. Two types of surface finish have been used in this research which is Electroless Nickel and Electroless Nickel / Immersion Gold (ENIG). The purposes of the surface finish are to provide a diffusion barrier, oxidation barrier, adhesion and solderable surface on copper substrate. For this research, solder material that will be used is Sn-4Ag-0.5Cu to form a solder joint. All samples were subjected to reflow soldering and then to the isothermal ageing at 150℃ with duration 250 hours and 500 hours. The IMCs were characterized using scanning electron microscopy and image analyser to investigate the thickness of IMC on solder joint at all samples. From the research, it was observed that the IMC growth is influenced by ageing duration where the thickness of IMC is increases with ageing.

ABSTRAK

Tesis ini akan membincangkan tentang kesan lapisan salutan yang berbeza terhadap kekuatan sambungan solder terutamanya terhadap sebatian antara logam (IMC). Lapisan salutan amat penting terhadap aplikasi di dalam papan litar kerana ianya mampu melindungi bahan asas papan litar tersebut iaitu kuprum daripada pengoksidaan dan kemerosotan. Walaubagaimanapun, ketebalan lapisan salutan boleh memberi kesan yang berbeza terhadap kekuatan sambungan solder dan jangka hayatnya. Dua jenis permukaan penyudahan digunakan dalam penyelidikan ini iaitu *Electroless Nickel* dan *Electroless Nickel / Immersion Gold* (ENIG). Tujuan penyudahan permukaan adalah untuk memberi halangan terhadap penyebaran, halangan pengoksidaan, lekatan dan memberi keboleh solderan di atas substrak kuprum. Semua sampel akan dikenakan pematerian *reflow* sebelum dikenakan penuaan isoterma pada suhu 150℃ selama 250 jam dan 500 jam. IMC akan dikarakterkan menggunakan SEM dan penganalisis gambar untuk mencari ketebalan IMC pada sambungan solder di setiap sampel. Daripada penyelidikan, didapati pembentukan IMC dipengaruhi oleh masa penuaan isoterma dimana IMC menjadi tebal apabila semakin lama dikenakan penuaan isoterma.

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LIST OF ABBREVIATIONS

Ag	Silver
Au	Gold
CTE	Coefficients of Thermal Expansion
Cu	Copper
ENIG	Electroless Nickel / Immersion Gold
IC	Integrated Circuit
IMC	Intermetallic Compound
Ni	Nickel
PCB	Printed Circuit Board
SEM	Scanning Electron Microscopy
SMD	Surface Mount Device
Sn	Tin
TAB	Tape automated bonding
UV	Ultraviolet

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Printed circuit boards (PCB) are electronic circuits that are created by mounting electronic components on a nonconductive board, and produce conductive connections between them. The creation of circuit patterns is accomplished using both additive and subtractive methods. The conductive circuit generally is a copper, although aluminum, nickel, chrome, and other metals are sometimes used. The majority of printed circuit boards today are made from purchased laminate material with copper already applied to both sides. Copper that is unwanted for the board will be removed by various methods leaving only the desired copper traces, this is called subtractive. PCB can also be produced using an additive method where traces are added to the bare substrate and this is a complex process of multiple electroplating steps.

The performance and reliability of an electronic product depends largely on the electronic packaging. Electronic packaging is the manufacturing step that packages a semiconductor chip so it is protected and can be connected to other electronic components in electronic equipment. Thus, it is the bridge that interconnects the system to the next level. The interconnection bonding technologies used in electronic packaging has been evolving from wire bonding, tape automated bonding (TAB) to the latest flip-chip interconnection bonding.

Soldering is a process in which two or more metal items are joined together by melting and flow a filler metal (solder) into the joint. The filler metal which has a lower melting point than the work piece will flowing into the joint and solidify to join the metals. For the types of solder, lead free solder Sn-4Ag-0.5Cu is used due to its environmental friendly.

Among the factors that are important to ensure the strength and reliability of the solder joint is the intermetallic compound (IMC) that is formed during soldering. The IMC is formed between the solder and substrate surface finish and functions to provide mechanical, thermal, and electrical connections through the solder joint.

The IMC formation in the solder joint depends on the substrate surface finish metallization and the solder alloy used. The purpose of surface finish is to protect the substrate base metal from oxidizing and limits the diffusion of solder into underlying metal. There are many types of surface finish systems that exist in PCB manufacturing and the most practiced by industry is electroless nickel / immersion gold (ENIG). ENIG is a surface finish that consists of a thick layer of electroless nickel on the top of copper substrate and a thin layer of gold on the top of nickel surface. ENIG mostly used in industry because it offers better planarity, improved corrosion resistance to the base material and excellent wettability.

1.2 OBJECTIVES

The main objective of this research is to determine the effect of coating type on intermetallic compound thickness. Coating is essential in printed circuit board application since it can protect the base material, which is Copper, from oxidation and degradation. However, the coating thickness will give different effect on solder joint strength or life. Therefore, the current work will focus different coating type on solder joint, specifically in intermetallic compound thickness.

1.3 SCOPE

The copper substrates are plated with Electroless Nickel and Electroless Nickel / Immersion Gold (ENIG) surface finish. The substrate then will be forming a solder bump on surface finish by using lead-free solder (Sn-3Ag-0.5Cu) SAC 305. The solder joints produced are then subjected to isothermal ageing for several durations. The IMCs formed are examined using different characterization tools.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter will provide an overview of electronic packaging, printed circuit board, intermetallic compound (IMC), surface finishes which is focus on electroless nickel and electroless nickel/ immersion gold (ENIG), soldering, solder alloy and also solder joint fatigue.

2.2 ELECTRONIC PACKAGING

Electronic Packaging refers to the method of enclosing, protecting or providing physical structure to either electronic components, assemblies of components or finished electronic devices. Packaging is the bridge that interconnects the ICs and other components into a system-level board to form electronic products. The integration of many circuits or components on a single chip is defined as an integrated circuit (IC). ICs are classified by their material and composition, number of transistor elements, degree of integration, manufacturing method, principles of operation and device type. An IC can be a single component such as a power amplifier or they can have many components such as a fully integrated microprocessor used in modern PCs and high performance servers and workstations. IC or chip is packaged into an electronic packaging consisting of a small black epoxy encasement that allows the device to be handled without damaging it and to be soldered onto a circuit board.

Electronics packaging often involves a series of different electronics packages. For example, a series of integrated circuits which each of it is in their own electronics package, are soldered onto a circuit board along with other devices, such as resistor, diode and amplifier, which each of it also in their own electronics package. The circuit board itself also can be considered an electronics package as well, as it provides a place and method to connect the integrated circuits, diodes and resistors as well as a stable structure that can be attached to a framework. The framework, too, is an electronics package, because it’s providing the structure needed to collect the circuit boards into a larger and single assembly. On the smaller end of the scale, primary electronic components such as resistors and CPU chips are usually packaged in plastic or epoxy, and sometimes glass also is used for it. If the component emits interference or is required to endure high temperatures, it can be placed in an additional outer electronic package made of metal. An important part of packaging primary electronic components is the means that the package provides for connecting the components to other components (Tummala, 2001).

Circuit board itself also can be electronic package. The type of package used usually is determined by the use of the assembly or the conditions in which it will be used. For example, a circuit board can be screwed onto a framework that simply holds it in place or be encased in plastic or resin in order to make it waterproof. It can also be enclosed in a sheet, cast or machined metal case to prevent it from being affected by circuit noise, or the case can be airtight to create a hermetic seal that prevents the assembly from being affected by atmospheric conditions.

2.3 PRINTED CIRCUIT BOARD (PCB)

PCB is a self-contained module of interconnected electronic components found in devices ranging from common pagers, or beepers, and radios to sophisticated radar and computer systems. The circuits are formed by a thin layer of conducting material deposited, or "printed," on the surface of an insulating board known as the substrate. Individual electronic components are placing on the surface of the substrate and soldering to the interconnecting circuits. There are three major types of printed circuit board construction which is single-side, double-side, and multi-layer. (Babak, 2005)

Single-side boards have the components on one side of the substrate. When the number of components becomes too much for a single-side board, a double-side board may be used. Electrical connections between the circuits on each side are made by drilling holes through the substrate in appropriate locations and plating the inside of the holes with a conducting material. The third type, a multi-layer board, has a substrate made up of layers of printed circuits separated by layers of insulation. The components on the surface connect through plated holes drilled down to the appropriate circuit layer. The base material for printed circuit board is generally fiberglass, and the conductive connections are generally copper and are made through an etching process (Babak,2005). Table 2.1 shows the steps of manufacturing process for PCB.

Table 2.1: Manufacturing Process for PCB

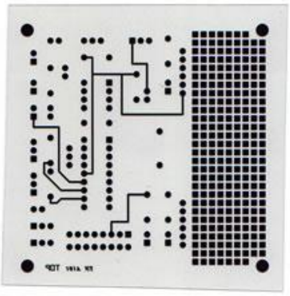
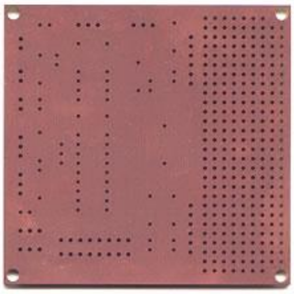
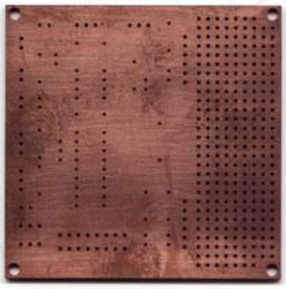
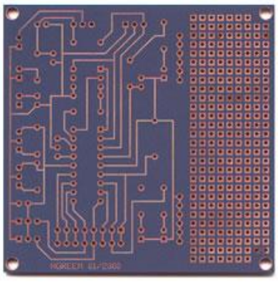
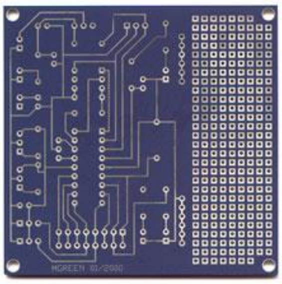
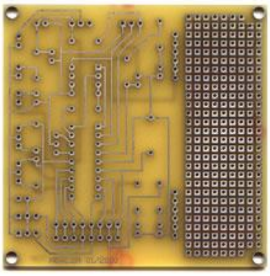
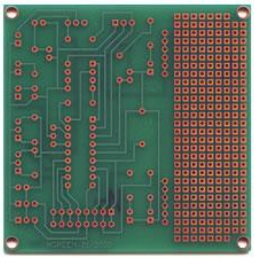
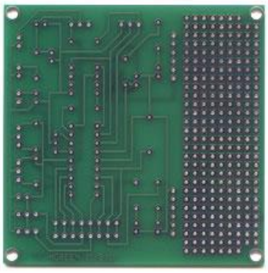
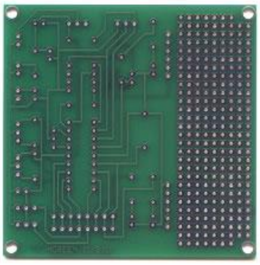
Step	Description	Picture
Step 1: Film Generation	The film is generated from the design files (Gerber files) which are sent to the manufacturing house. One film is generated per layer.	
Step 2: Raw Material	Using industry standard 0.059" thick, copper clad with two sides. Panels will be sheared to accommodate many boards.	
Step 3: Drill Holes	Using CNC machines and carbide drills to drill holes according to the drill spec sent to the manufacturing house.	
Step 4: Electroless Copper	Apply thin copper deposit in holes barrels.	
Step 5: Apply Image	Applying photosensitive dryfilm (plate resist) to panel. Using light source and film to expose panel. Develop selected areas from panel.	

Table 2.1 Continued; Manufacturing Process for PCB

Step 6: Pattern Plate	Doing an Electrochemical process to build copper in the holes and on the trace areas. Apply tin to surface.	
Step 7: Strip and Etch	Remove the dry film, and then etching the exposed copper. The tin protects the copper circuitry from being etched away.	
Step 8: Solder Mask	Applying a solder mask area to the entire board with the exception of solder pads.	
Step 9: Solder Coat	Applying solder to pads by immersing into tank of solder. Hot air knives level the solder when removed it from the tank.	
Step 10: Nomenclature	Applying white letter markings using screen printing process	

Source: Babak Kia (2005)

2.4 ELECTROLESS NICKEL

Electroless is a chemical process which promotes continuous deposition of a metal onto a surface through an oxidation-reduction chemical reaction, without the use of an external electrical potential. An internal reducing agent donates electrons to the positively charged metal ions in solution, thereby reducing the metal and promoting its deposition onto the catalyzed metal surfaces of the substrate. This reaction is considered auto-catalytic because it will continue to plate in the presence of source metal ions and a reducing agent until the board is removed from the plating bath. This gives the plated part a very uniform deposit and the plating thickness can be controlled easily, even on complex shapes and internals (Okinaka, 1974).

Therefore, electroless nickel is the autocatalytic deposition of nickel from an aqueous nickel salt solution onto a substrate and the corresponding oxidation of hypophosphite anions to phosphite ions with the evolution of hydrogen gas at the catalytic surface

The purposes of electroless nickel plating are to improving the corrosion resistance, providing a uniform and dense coating, increasing the surface hardness of the material, and in many cases, maintains the same surface finish the material had before plating.

Electroplating bath consists of a series of components to produce a desired layer of nickel thickness as shown below:

a) Source of nickel ions

   The metal source serves as the provider of nickel ions. It is introduced as metal salts and the most widely used and preferred source of nickel is nickel sulphate. Other nickel salts include nickel chloride, nickel acetate and the ideal source of nickel ions is nickel salt of hypophosphorus acid, $Ni\ (H_2PO_2)_2$.

b) Reducing agent

   Reducing agents for electroless nickel plating are usually phosphate or boron based with the former more widely used. The reducing agents provide reaction energy for the nickel to deposit.

c) Complexing agent

Complexing agents assist in preventing the pH of the electroless nickel solution from decreasing too fast. They are also required to prevent precipitation of nickel salts into precipitated phosphites and reduce the concentration of free nickel ions. There are several types of complexing agents used and they include citrate, glycolate or lactate ions which come in monodendate, bidendate, tridendate and quadridentate forms of anions. The rate of the nickel deposition is proportional to the stability constant of the complexing agent. The lower plating bath is generally explained by the ‘tying-up’ process of the nickel ions and releases only a small fraction of free nickel ions.

d) Stabilizer

Stabilizers are added to reduce the spontaneous decomposition rate of the Electroless Nickel plating solution. Stabilizers raise issues of decreased deposition rate of the plating process. Many Electroless Nickel plating processes have employed stabilizers which can be divided into the following groups (Mallory, 1990):

i. Compound of group VI elements
ii. Compound Containing oxygen
iii. Heave metal cations
iv. Unsaturated organic acids

e) pH adjuster

Rapid increase of the hydrogen ion concentration in the plating bath will increase the acidity of the bath. This can be measured by the decrease of the pH value. Reduced pH will slow down the plating rate considerably and eventually stop the plating. The impairing of hypophosphite reduction power in low pH condition causes this condition. In addition, the newly plated nickel deposition will dissolve into the highly acidic plating solution at an increasing dissolution rate and eventually equal the nickel deposition rate. Therefore, it is necessary to adjust and maintain the pH value throughout the plating process in order to obtain a satisfactory thickness (Pecht, 1990). The adjustment of pH can be done by periodic or continuous addition of a soluble alkali hydroxide or a soluble alkaline salt.

Figure 2.1 shows the formation of Nickel ions when Copper substrate was subjected with Electroless Nickel plating. Nickel ions will form a Nickel layer and coat the Copper substrate.

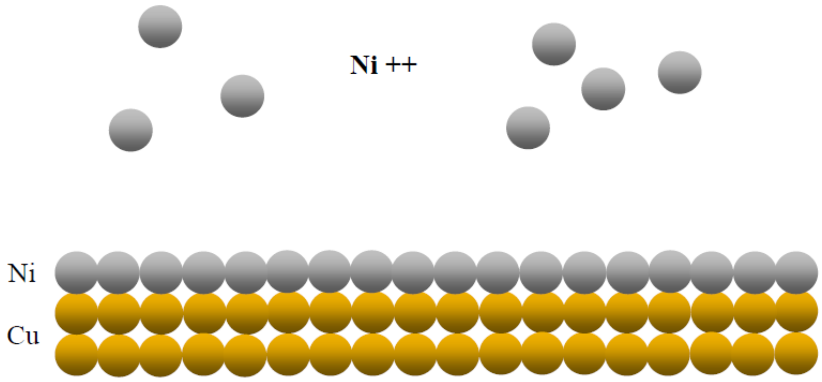


Figure 2.1: Electroless Nickel Plating

Source: Dan Slocum (2003)

2.5 ELECTROLESS NICKEL / IMMERSION GOLD (ENIG)

ENIG is an electroless nickel layer capped with a thin layer of immersion gold. It is a multifunctional surface finish, which is applicable to soldering, aluminum wire bonding, press fit connections, and also as a contact surface. The immersion gold protects the underlying nickel from oxidation or passivation over its intended life. The gold layer is very thin and not intended to provide the main structure of the track. It just acts as a protective coating for the nickel to prevent it tarnishing before it is soldered. Gold is extremely resistant to corrosion so ENIG has several good points. It can be touched with bare fingers without tarnishing, has a very long shelf life, and the pads or tracks are very flat and square-edged, which is something that very important for fine pitch surface mount part.

ENIG is formed by the deposition of electroless nickel on a catalyzed copper surface followed by a thin layer of immersion gold. The IPC ENIG Specification 4522 specifies that the electroless nickel thickness shall be 3 to 6 µm. The minimum immersion gold thickness shall be 0.05 µm which are at four sigma (standard deviation) below the mean, the typical range is 0.075 to 0.125 µm. Higher gold thickness would normally require extended solution dwell time and /or increased solution temperature (Dan, 2003).

The ENIG deposition process is fairly complex; it requires a clean copper surface free of solder mask residues as well as free of any copper/tin intermetallic (tin is used as an etch resist and is stripped before ENIG). Solder mask for ENIG plating must be adherent and completely cured (cross-linked) to withstand the high temperature and prolonged dwell in the electroless nickel bath and in the immersion gold bath (Dan,2003). Figure 2.2 shows the formation of Nickel ions and gold ions when Copper substrate was subjected to the ENIG plating.

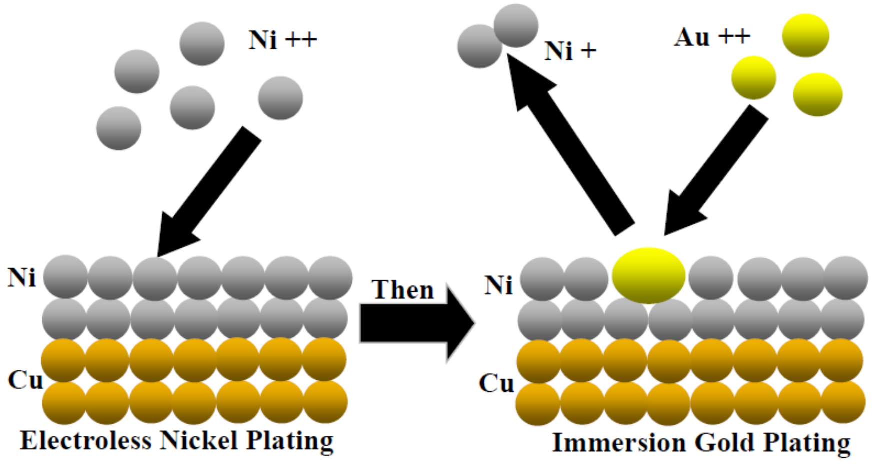


Figure 2.2: Electroless Nickel / Immersion Gold Plating

Source: Dan Slocum (2003)

2.6 INTERMETALLIC COMPOUND (IMC)

IMC have often been observed at or near the solder/substrate interface as well as in the interior of solder joints. Its can be defined as a mixture in specifics proportion of two metallic elements that form a periodic crystalline structure different from those of the original elements. Unlike conventional metal alloys, the particular structure of intermetallic compounds is caused because of the respectively larger strength of bounding between the respective unlike atoms than between like atoms. This particular structure of intermetallic compounds gives them some mechanical properties such as high melting points and great strength (particularly at high temperatures), but poor ductility (Sandström, 2002).

Intermetallic compounds usually form between two metal elements that comprise of a limited mutual solubility through diffusion. These compounds possess a composition of a certain stoichiometric ratio of the two elements (Gilleo, 2004). The new composition has a different crystal structure from those of their elemental components. The interface of tin and copper when examined in a cross section would reveal a superimposed layer consisting of compounds having certain copper/tin ratio as shown in Figure 2.3.

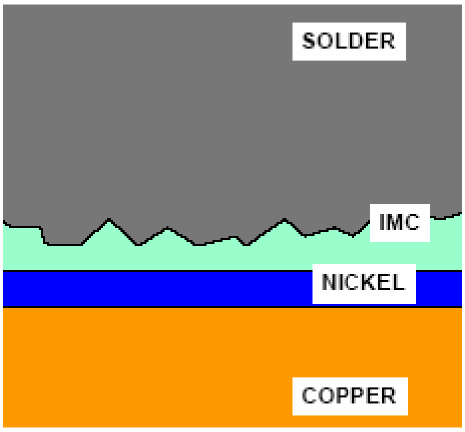


Figure 2.3: Formation of IMC layer between solder and copper

Source: Madeni (2003)

There are some key facts about intermetallic formations that should be illustrated in order to prevent reductions in reliability, solderability and yield. Intermetallics are necessary, but it can result in embrittled joints and unsolderable components or circuit boards.

When solder comes in contact with a common metal substrate for a sufficient amount of time at a high enough temperature, intermetallic compounds may form. Below a solder’s liquidus temperature, formation is primarily a solid state diffusion process and thus depends highly on temperature and time. While solder is in a molten state, the solubility of the element from substrate into molten solder accelerates the rate of intermetallic formation.

There are various factors which influence the intermetallic formation, the composition of the compound and its morphology:

i. The metallurgical reactivity of a solder with a substrate
ii. Soldering (reflow) peak temperature
iii. Dwell time at peak temperature
iv. The surface condition of a substrate
v. The post- soldering storage and service conditions

Apart from above factors, the selection of solder alloy and surface finish play important role as well.

2.6.1 Types of IMC formations in Solder Joints

Formation of IMC at the solder interface is primarily governed by the material of the solder, surface finish and substrate metal pad. The most commonly found IMCs are from copper-tin lead-frame, where the copper is the base material and the tin comes from solders. Lead rarely forms intermetallic in solder joints but commonly encountered elements that may form IMC with tin which include copper, nickel, silver and gold.

The possible IMC forms at the interfacial of solder and substrate is determined by solder material used, substrate material and surface finish covered on the substrate. Table 2.2 shows the possible intermetallic phases in various lead frame system.

Table 2.2: Possible intermetallic phases in various lead-frame system

Lead-frame system	Intermetallic
Copper-tin	Cu_3Sn, Cu_6Sn_5
Nickel-tin	Ni_3Sn_2,Ni_3Sn_4,Ni_3Sn
Gold-tin	AuSn,$AuSn_3$,$AuSn_4$
Silver-tin (Lead-free solder)	Ag_3Sn

Source: Madeni (2003)

The most common solder/substrate intermetallic compound is tin/copper (Sn/Cu). The growth of the total intermetallic layer is influenced by the synergy information and growth between η (Cu_6Sn_5) and ε (Cu_3Sn) phases. Cu_6Sn_5 (η- phase) is the first to form at lower temperatures (< 170°C) and is essential for good solder adhesion to the copper substrate. This compound grows rapidly but once the metals solidify, it will slow sharply. The ε-phase has been shown to grow at the expense of the η-phase, and has higher activation energy of formation, so growth of ε is more predominant at higher temperatures. The mechanisms for intermetallic formation are also greatly influenced by the interdiffusion characteristics of tin and copper.

In Nickel/gold-tin system, the eutectic solder forms a strong attachment to underlying nickel by forming the Ni_3Sn_2, Ni_3Sn_4, Ni_3Sn intermetallic while the Au dissolves into the molten solder, and forms fine, needle-shaped $AuSn_4$ intermetallic precipitates that are retained in a dense distribution in the bulk of the solder joint after it has solidified (Glazer, 1991).

When lead free solder, such as Sn/Ag or pure Sn is used on a copper substrate, the Ag_3Sn plate intermetallic is formed while Cu_6Sn_5 compound forms as the bottom layer. Large Ag_3Sn plate formation is found to be substantially reduced in alloys with a silver content less than 3 wt % (Sung, 2004). Because of the relatively low fraction (3-5 wt%) of alloying elements, these intermetallic structures comprising a small portion of the area within the solder joint. The morphology are varies, exhibiting a round, lath like, blocky, or needle like structure. Studies have shown, however, that intermetallic structures at the interface, such as tin-copper, grow slower in some tin-based lead-free solders than with their leaded counterpart. It is believed that lead plays a part in enhancing intermetallic growth when subjected to thermal exposure.

2.7 SOLDERING

Soldering is a process in which two or more metal items are joined together by melting and flow a filler metal (solder) into the joint, the filler metal having a lower melting point than the workpiece. Soldering basically is use to make electrical connection between electrical device like transistor, diodes, capacitors, resistance and etc. Its also provide physical connection between the component and its supporting printed circuit board (Woodgate, 1983).

Solder chemically reacts with other metals to form different alloy. There are four basic element involve in soldering process; base metal, flux, solder and heat. A base metal is a metal that contacts the solder and forms an intermediate alloy. When attaching electronic components to a printed circuit board, the component's leads or pins and board's metallic circuitry at the base metals and will contact with the solder. Many metals, such as copper, bronze, silver, brass, and some steels, readily react with solder to form strong chemical and physical bonds. There is a direct relationship with the level of surface oxidation on the base metal and how readily solder will react with it. The more oxidation is present, the weaker the solder bond will be (Hwang, 2002).

Flux is often applied as a liquid to the surface of the base metals prior to soldering. The first and primary purpose of flux is to stop the base metals from oxidizing while they are being heated to the soldering temperature. The flux covers the surface to be soldered, shielding it from oxygen and thereby preventing oxidation during heating. Most fluxes have an acidic element that is used to remove the oxidation already present on the base metal. Using a strong acid, it would be possible to virtually completely clean off the oxidation layer. When the liquid solder is applied, the flux must readily to move out of the way so the solder can come directly contact with the base metal. During this process some of the flux inevitably combines with the solder.

Solder is typically transported and sold in solid form. Common forms of solder including chips, bars, and wire (often with a core of flux), each of it which has advantages in different soldering processes. A common process called reflow soldering is use for a solder paste. Solder paste is a substance with a cream-like consistency made up of solder, flux, and some carrying medium. The material most commonly used for solder in the electronics industry is a tin-lead alloy. Tin-lead alloys have relatively low melting point and can be produced at a low cost in comparison with other alloys with similar properties.

2.7.1 Soldering Methods

There are various soldering techniques developed throughout the growth of electronic assembly to promote the process capability and productivity. It will be categorized whether it is performed by hand or by machine. For circuit board, generally the soldering process will fall into two main categories, which are wave soldering and reflow soldering. For wave soldering, it is primarily used for soldering through-hole components on PCBs where the electronic components are inserted prior to application of solder. For reflow soldering, it is used for soldering SMD (Surface Mount Device) components on to PCBs. The electronic components are mounted after the application of solder.

2.7.1.1 Wave Soldering

Wave Soldering is a large-scale soldering process by which electronic components are soldered to a printed circuit board (PCB) to form an electronic assembly. This technique is used for both through-hole printed circuit assemblies and surface mount. Basically there is a tank which heated at high temperature to hold the molten solder. The components are inserted to the PCB and the loaded PCB is placed on a motorized, edge hold conveyor where it is fluxed, preheated and skimmed over the crest of the solder wave. As the PCB continues past the wave, it cools and solder solidifies and complete solder joint formation (Clyde, 2001).

A standard wave solder machine consists of four zones which are the fluxing zone, the preheating zone and the soldering zone. An additional fourth zone, cleaning, is used depending on the type of flux applied.

a. Fluxing

   In fluxing zone, the fluxer applied flux underside of the PCB. This is a key sub-process where uniform and sufficient quantity of flux application requires for a good solderability. The two common fluxing methods used are spray fluxing and foam fluxing.

a. Preheating

   Preheating is the next step to activate the flux and remove any flux carrier solvents. The purpose of this zone is to prevent thermal shock on the PCB which sudden high temperature expose will potential cause failure on board reliability and it is also essential for flux to reach enough temperature for its activation, reaction and disruption of oxides and tarnishing on the metals of the materials to be soldered (Clyde, 2001).

b. Wave Soldering

   The solder waves from the tank of molten solder contact the bottom of the board, and sticking to the solder pads and component leads via surface tension. This process is sometimes performed in an inert gas atmosphere to increase the quality of the joints. Figure 2.4 and Figure 2.5 shows the principle and the process flow of wave soldering.

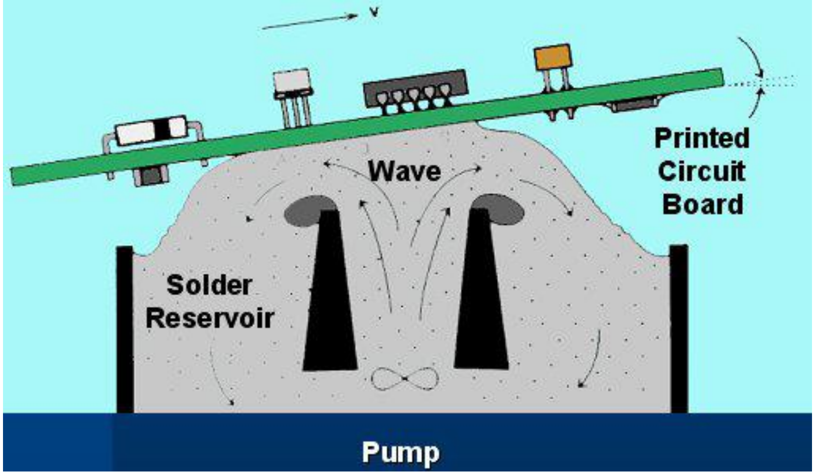


Figure 2.4: Schematic showing the principle wave soldering

Source: Clyde (2001)

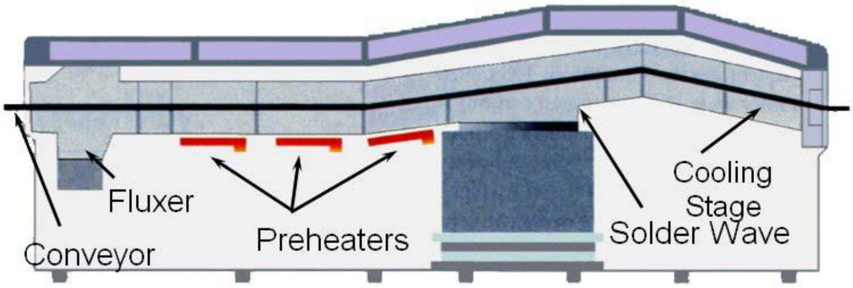


Figure 2.5: Wave soldering process flow

Source: Clyde (2001)

2.7.1.2 Reflow Soldering

Reflow soldering is the most common means to attach a surface mounted component to a circuit board. The process consists of applying solder paste to a circuit board, placing the devices onto the paste, and then conveyed the board through an oven with successive heating elements of varying temperatures. The purpose of the reflow process is to melt the powder particles in the solder paste, wet the surfaces which will be joined together, and solidify the solder to create a strong metallurgical bond.

In reflow soldering process, each component is placed on the board with the metallization on the die aligned with the corresponding lands on the board, and then the solder paste printed onto PCBs re-melts and forms the solder joints. During reflow, the heat profile tracks the work temperature against time.

The solder profile, also known as thermal profile is one of the key variables in the manufacturing process that significantly impacts product yield. There are four major zones consists in the reflow furnace: preheat zone, soaking zone, reflow zone and cooling zone. To set-up a soldering profile, parameters for each zone such as temperature settings, cooling speed, conveyor speed and fan speed must be considered (Bentzen, 2000). This reflow profile will ensure better wettability and solderability between solder and surface for intermetallic formation (Clyde, 2001). The reflow profile is not only product specific, it is also flux dependent. Different pastes require different profiles for optimum performance.

i. Preheat Zone

The objective at preheat zone is to increase the temperature at a control rate to minimize any thermal damage to the component and board. The heating rate in this zone is around 2 to 4°C/ sec. At this stage, preheating of the substrate and components are initialized. The flux also becomes chemically active. The ramp gradient is important in this stage. Solder ball formation will occur if the gradient too steep where these will decrease local solder volume and affect the solder bonding. It is also to avoid thermal shock for sensitive components such as ceramic chip.

ii. Soak Zone

During the soak phase the temperature rises slowly. At this phase, the flux flows onto the metal surfaces to react away surface oxides and acts as a barrier to prevent oxidation. The ramp rate in this zone is very low to avoid the flux activate fully too early which could dry out. If this temperature is not reached during the soaking period, only partial cleaning of the solder spots is performed and wetting problems can occur.

iii. Reflow Zone

At reflow zone the temperature and time control in this stage is essential because the formation of molten solder and metal surface take place. The temperature must be high enough for adequate flux action and obtain good wetting. The temperature should not too high which will damage the components and substrate. To avoid a too thick intermetallic layer, the ideal solder temperature is

30 - 40 ºC above the solder paste melting point. Time control is also important to avoid damage for temperature sensitive components and avoid excessive intermetallic growth which will lower the solder joint fatigue resistance as IMC is brittle in nature.

iv. Cooling Zone

The cooling zone is a phase where solidification of solder joint occurs. The cooling rate should be as fast as possible to ensure the grain size of the solder is smaller and also for the solder joint to perform a strong bonding between the solder pad and the component terminal. Hence it will promote the fatigue resistance of the solder joint.

Figure 2.6 show the reflow profile for lead free solder. From reflow profile, the better wettability and solderbility between solder and surface for IMC formation can be ensured.

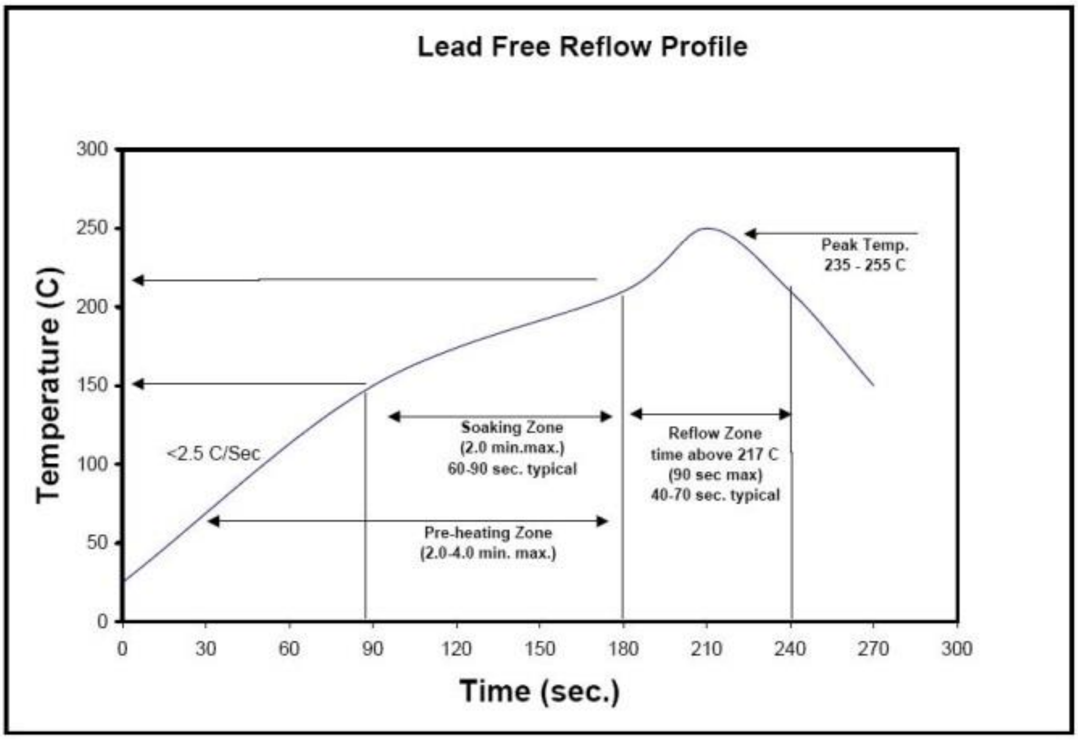


Figure 2.6: Reflow Profile

Source: Prasad (1996)

2.8 SOLDER ALLOY

A solder is a fusible metal alloy with a melting point or melting range of 90 to 450°C (200 to 840°F), used in a process called soldering where it is melted to join metallic surfaces. It is generally useful in electronics and plumbing. Alloys that melt between 180 and 190°C are the most commonly used.

Soft soldering is characterized by the melting point of the filler metal, which is below 400°C (752°F). The filler metal used in the process is called solder. Different combinations of tin, lead and other metals are used to create solder. It is depend on the desired properties of the combination. The most popular combination is 60% tin, 39% lead, and 1% alloys. This combination is strong, has a low melting range and melts and sets quickly. A higher tin composition giving the solder higher corrosion resistances, but raises the melting point.

Hard soldering or silver soldering, performed with high-temperature solder containing up to 40% silver, is also often have a form of brazing, since it involves filler materials with melting points in the vicinity of, or in excess of, 450°C. In silver soldering (hard soldering), the purpose is generally to give a nice and structurally sound joint, especially in the field of jewelry. In silvers smiting or jewelry making, special hard solders are used so that it will pass assay. They containing a high proportion of the metal that being soldered and lead is not used in these alloys. These solders also come in a varieties of hardness, known as 'enameling', 'hard', 'medium' and 'easy'. Enameling solder has a high melting point, which close with the material itself, to prevent the joint desoldering during firing in the enameling process.

A number of solder materials, especially zinc alloys, are used to soldering aluminum metal and alloys and some of lesser extent steel and zinc. This mechanical soldering is similar with low temperature brazing operation which is the mechanical characteristics of the joint are reasonably good and it can be used as a structural repairs for those materials.

Lead-free solder has a higher melting point, slower flow rates and sometimes can cause leeching in the iron components. Higher melting points and the decreased flow rates will require a longer contact period between the solder and the connections in order for the solder to completely fill the holes. Higher melting points also require a melting pot made from more stable material. The melting pot and ducts also need to be made from material that will prevent dissolution.

Table 2.3 below shows the commonly used lead-free solder alloy and its properties. Among these lead-free solder alloy, SnCu0.7, Sn3.5Ag0.5Cu, Sn3.8Ag0.7Cu and SnAg3.5 are better solder alloy because of the better wetability and solderability.

Table 2.3: Commonly used lead-free solder alloy

Lead Free Solder Alloy	Solidus (°C)	Liquidus (°C)
SnAg3.5	221	221
SnCu0.7	228	228
SnCu3.0	228	300
Sn3.8Ag0.7Cu	217-218	217
Sn3.9Ag0.6Cu	217-218	217
Sn10In3.1Ag	204	205
91.8Sn3.4Ag4.8Bi	211	213
96.2Sn2.5Ag0.8Cu0.5Sb	215	217
SnZn8Bi3	189	199

Source: Karl (1995)

2.9 SOLDER JOINT FATIGUE

Solder joint fatigue is resulting from the application of cyclical stresses. It is often considered to be the largest and most critical failure categories, since it is encountered in many different situations that are difficult to control. Solder joint fatigue failure is attributed primarily to stresses brought by temperature swings and mismatches between the coefficients of thermal expansion (CTE) of the mounted devices' solder joints and the application board. The extent of the deformation depends on the design of the joint. Usually the deformation of the solder in the joints is rather large, of the order of 1%, and the movements are slow, with typical cycle times measured in hours. Referred to as ‘low cycle fatigue’, a cracking from this type of deformation may be observed in all kinds of soldered joints and this is the main cause of eventual crack formation in initially good joints.

During the fatigue process, successive metallurgical phenomena occur. When the strain in the joint exceeds the plastic limit, the solder will start to creep. However, there is the complication for tin-lead solder that the material consists of two separate phases. The first phase lead has up to 19% of dissolved tin, with a face-centered cubic crystal structure and its coefficients of thermal expansion (CTE) is the same in all three directions. The second phase, by contrast, the tin phase, containing up to 2.5% of lead, has a body centered tetragonal structure, where the thermal expansion at the axis of maximum CTE is very close to that lead, but only about half this along the minimum axis.

This anisotropy causes internal stresses in the structure during slow thermal cycling. As a result of these stresses, the solder shows the phase segregation and grain growth and the weaker lead phase where the cracks start and propagate during continued thermal cycling. Before the cracks start, there is usually a change in the surface appearance of the joint, which becomes rough are often having the appearance of small steps which usually develop into cracks.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter will discuss about the methodology that will used in order to achieve the objectives of the research. The main objective of this research is to study the different coating thickness on copper substrates. The research methodology started with copper substrate preparation followed by surface finish deposition. At this stage, several thin layers of different metals were deposited onto the substrate according to the required thickness. The surface finish that will use in this research is electroless nickel/ immersion gold (ENIG). After preparing the substrate and surface finish, different sizes of lead-free Sn-4.0Ag-0.5Cu solder balls were placed onto the surface and the whole structure was subjected to reflow soldering in a resistance furnace. After the reflow soldering process, isothermal ageing was carried out to these soldered substrates with different ageing periods. After ageing, samples were prepared for characterization process using various characterization tools.

3.2 MATERIAL

The base substrate selected was a commercial oxygen free copper. Specimens were prepared with dimensions of 40 X 45 X 1 mm (width x length x thickness). The copper substrate has a laminated structure which contained a layer of polymer in the middle between two copper surfaces. The copper substrates were subjected to pretreatment process to remove dust, grease oxides and activate the copper surface before plating process. The immersion gold plating was performed right after electroless nickel without any pretreatment except rinsing in running water and drying. Figure 3.1 shows the dimension of Copper substrate.

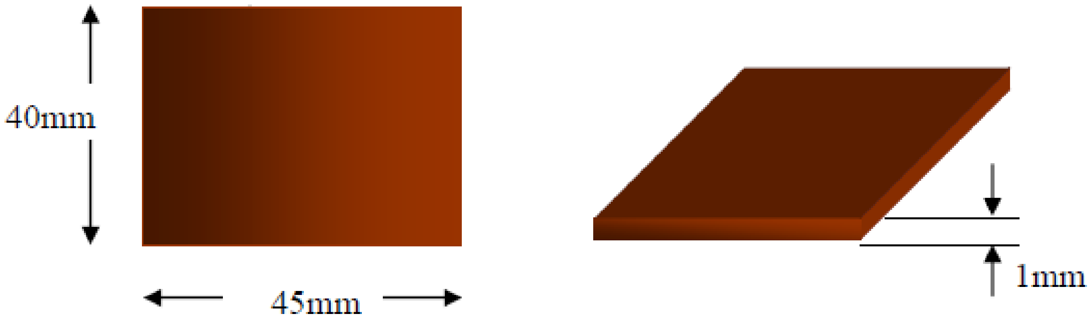


Figure 3.1: The dimension of Copper Substrate

3.2.1 Surface Finish Deposition

As previously mentioned, the study puts focus on two surface finish which are electroless Nickel and electroless Nickel/ Immersion Gold (ENIG). Electroless Nickel finishing only involve Nickel plating while ENIG finishing involves Nickel and gold plating which will provide a diffusion barrier between the copper and solder for the former and a surface passivation layer for the latter. Nickel characteristics such as hardness and solderability make this process a durable alternative PCB surface finish. Meanwhile, the thin layer of immersion gold preserves the solderability of the finish by preventing oxidation of the highly active nickel surface.

3.2.2 Surface Preparation of Copper Substrates

In order to achieve optimum and uniform electroless nickel plating, it is critical to obtain a copper substrate free from potential contaminants such as dust, oxides and grease. This was done by means of surface pretreatment as the steps below:

i. Medium alkalinity soak at 70 to 90°C for 5 minutes to eliminate contaminants.
ii. Rinsing with distilled water.
iii. Caustic cleaning with 10% NaOH at 60°C for 3 minutes to degrease.
iv. Rinsing with distilled water.

v. Acidic pickling with 10% H_2SO_4 for 1 minute to remove oxide layer.
vi. Rinsing with distilled water.
vii. Surface activation in palladium chloride + HCl solution for 3 minutes.
viii. Rinsing with distilled water.

3.3 ELECTROLESS NICKEL PLATING

The electroless nickel plating is carried out in a plating bath with stainless steel heating coils which offered constant temperature control. The bath has a capacity of 10 liters. However, plating is conducted in a one-liter Pyrex beaker placed inside the bath to minimize the amount of solution used for the plating. The remaining space left in the bath was filled with water which is used to heat up the plating solution. An aluminium rod is placed horizontally across the beaker to suspend the substrate in the solution. A double end hook-shaped aluminium rod was used to hook the aluminium clip which holds the substrate and suspend it from the aluminium rod. Schematic diagram of equipment setup for electroless nickel (boron) plating is shown at figure 3.2.

The combination of chemical composition and typical parameters for electroless nickel plating solution is shown in Table 3.1.

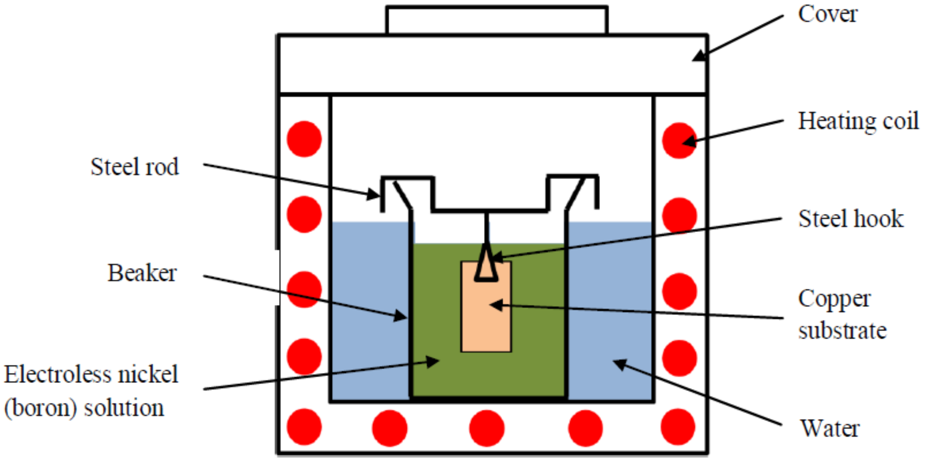


Figure 3.2: Schematic diagram of equipment setup for electroless Nickel plating.

Table 3.1: Chemical composition and parameters for electroless nickel with boron solution

Chemical	Composition
Nickel Chloride	30 g/ L
Disodium Tartarate	40 g/ L
Sodium Hydroxide	40 g/ L
Sodium Borohydride	0.2 – 1.0 g/ L (0.6 g/L)
Ethylenediame (98%)	15
Lead Nitrate	16 mg/ L
Parameter	
pH	13
Temperature	65 °C (70 °C)

3.4 IMMERSION GOLD PLATING

The final layer of ENIG surface finish is immersion gold. After the nickel coatings were plated on copper surface, the copper substrates were removed from the nickel solution bath. No surface preparation was required except rinsing in running water and drying the nickel plated substrate. This was followed by the immersion gold plating, where the copper substrates were again placed into the gold plating bath using aluminium clips and hooks as supporting structures. Immersion gold plating is conducted on to electroless nickel plated copper substrates to provide corrosion resistance features. The immersion gold plating solution was prepared according to the chemical compositions and parameters as shows in Table 3.2 (Okinaka, 1974).

Table 3.2: Chemical composition of Swan & Gostin immersion gold plating solution

Chemical	Composition
Potassium Cynoaurate ($KAuCN_2$)	2 g/ L
Ammonium Chloride	75 g/ L
Sodium Citrate, dehydrate	50 g/ L
Sodium Hypophosphite	10 g/ L
Parameter	
pH	7.0 – 7.5 (7.3)
Temperature	93 °C (95 °C)

3.5 SOLDER MASK

The purpose of solder masking lamination is to prevent molten solder from flat spreading during reflow soldering process. Before the solder mask lamination, the surface finish need to be dried to ensure adhesion between substrate and solder mask is strong. Then, a layer of solder mask was laminated onto the plated substrates by using the laminating machine as shown in Figure 3.3, right after surface finish deposition.

Figure 3.3: Solder masking laminator

After that, the solder mask will be exposed to ultraviolet (UV) light with the printed negative film for solder pad opening preparation. The exposure time is 40 seconds in the equipment. This exposure was to ensure an array of holes being made onto the solder mask upon developing in the development chemical solution. Figure 3.4 shows the UV light cure equipment. These holes were used strictly as corrals for the solder to keep it localized around the substrate land area. The following step was further exposure of solder mask (with holes) to UV light for another 2 minutes in order to cure and harden the solder mask.

Figure 3.4: UV light cure equipment

3.6 SOLDER BUMP

In this study, the Sn/4.0Ag/0.5Cu solder was used with the size of solder balls diameter is 700µm. Before solder balls placement, a thin layer of no clean flux is applied on the substrate. The primary function of applying flux prior to reflow soldering is to remove the oxide film and tarnish from the surface of solder balls and substrate surface finish. Flux applied also can improve wetting of molten solder during soldering. Then, the solder balls are manually placed and arrange at an array on each substrate.

3.7 REFLOW SOLDERING

The substrate then will be subjected to reflow soldering in the resistance furnace with peak reflow temperature of 250 °C to form solder joints and metallurgical bonding. Reflow soldering is done in a resistance furnace as shown in Figure 3.5. The reflow temperature profile for Sn-Ag-Cu lead free solder is shown in Figure 3.6.

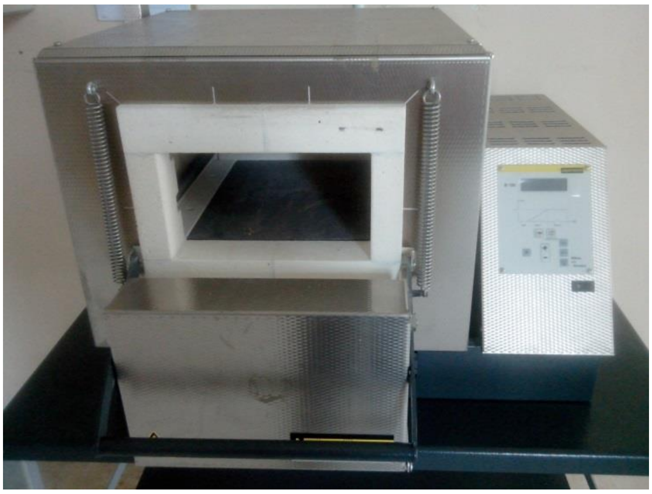


Figure 3.5: Resistance furnace for reflow soldering

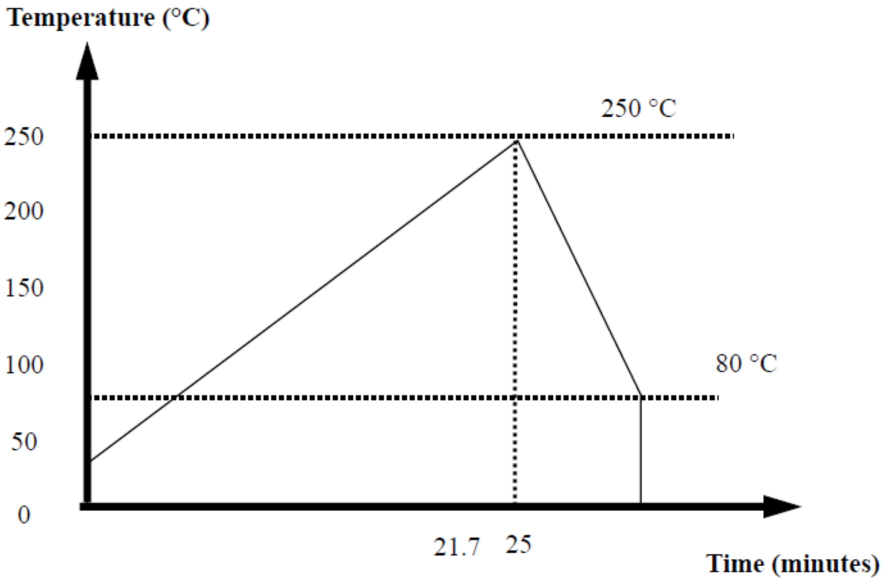


Figure 3.6: Reflow temperature profile

3.8 ISOTHERMAL AGEING

Isothermal ageing is a type of reliability test to accelerate the growth of intermetallic compounds (IMCs) within the solder joints. In general, the component is subjected to a fixed high temperature under a period of time to examine the effect of long term exposure to high temperature on the growth of intermetallic compounds. In this study, the specimen was subjected to isothermal aging at 150°C for 0, 250 and 500 hours using the same furnace.

3.9 CHARACTERIZATION

Characterization is performed on the samples to observe and identify the microstructure, morphology, thickness and composition of the material and the IMC formed. Only one set of specimen preparation procedure for this experiment which is for characterization of specimens at their cross section.

3.7.1 Characterization of Specimens Cross Sections

After the specimens were cut from each category, they were cold mounted into holdable specimens**.** Then, samples mounted were grinded on successive silicon carbide papers with various grits, followed by polishing process to refine the solder joint surface. The sample will be etching with 5% HCl in 95% ethanol about 15 second after polishing process.

The cross sections were observed by using Nikon optical microscope at various magnifications at different locations. The thickness of the IMCs layer at the solder joint was measured with the aid of the image analyzer and SEM.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

Solder joint reliability is one of the major concerns in the flip chip type electronic packaging because the formation of IMC at the interface between lead-free solder and surface finish has huge impact on the packaging interconnection. Hence, it is essential to investigate the effect of IMC thickness toward the solder joints in the package. As discussed in the literature, a thin layer of IMC is essential to form a strong and reliable solder joint, where as excessive growth of IMC is unwanted due to its hard and brittle nature, and also other factors such as CTE mismatch, stress concentration and consumption of surface finish which would reduce the reliability of the joints. In this chapter we will discuss the effect of coating type on IMC thickness during reflow soldering and isothermal ageing treatment between Sn-3Ag-0.5Cu on Copper, Electroless Nickel and Electroless Nickel/Immersion Gold (ENIG) surface finish.

4.2 THICKNESS OF IMC

In order to form a strong and reliable solder joint, intermetallic compound formation is essential. However, thick intermetallic layer is detrimental to the solder joint integrity due to its brittle nature and poor conductivity. In this research the thickness of the IMC formed in each sample was measured using the image analyzer and SEM. Five measurements have been taken from each sample and the mean value was calculated to be considered as IMC thickness and the coating thickness for that particular sample. Table 4.1, Table 4.2 and Table 4.3 shows the IMC thickness at Copper, Electroless Nickel surface finish and ENIG surface finish.

Table 4.1: IMC and coating thickness for Copper samples

Ageing Duration (hours)	Cu (μm)	Cu at solder joint (μm)	IMC (μm)
0	19.8	18.0	1.8
250	19.8	16.9	2.9
500	19.8	15.7	4.1

Table 4.2: IMC and coating Thickness for Electroless Nickel samples

Ageing Duration (hours)	Cu (μm)	Ni (μm)	Cu at solder joint (μm)	Ni at solder joint (μm)	IMC (μm)
0	20.4	1.2	19.9	1.2	0.4
250	20.4	1.2	19.5	1.3	1.4
500	20.4	1.2	18.1	1.5	2.1

Table 4.3: IMC and coating thickness for ENIG samples

Ageing Duration (hours)	Cu (μm)	Ni (μm)	Au (μm)	Cu at solder joint (μm)	Ni at solder joint (μm)	IMC (μm)
0	22.1	2.5	0.9	18.8	2.1	0.8
250	22.1	2.5	0.9	18.0	2.5	1.6
500	22.1	2.5	0.9	17.2	3.0	2.4

Copper on solder joint at every sample decreased due to increasing of ageing duration. The decreases of Copper at solder joint can be seeing at Figure 4.1, Figure 4.2 and Figure 4.3.

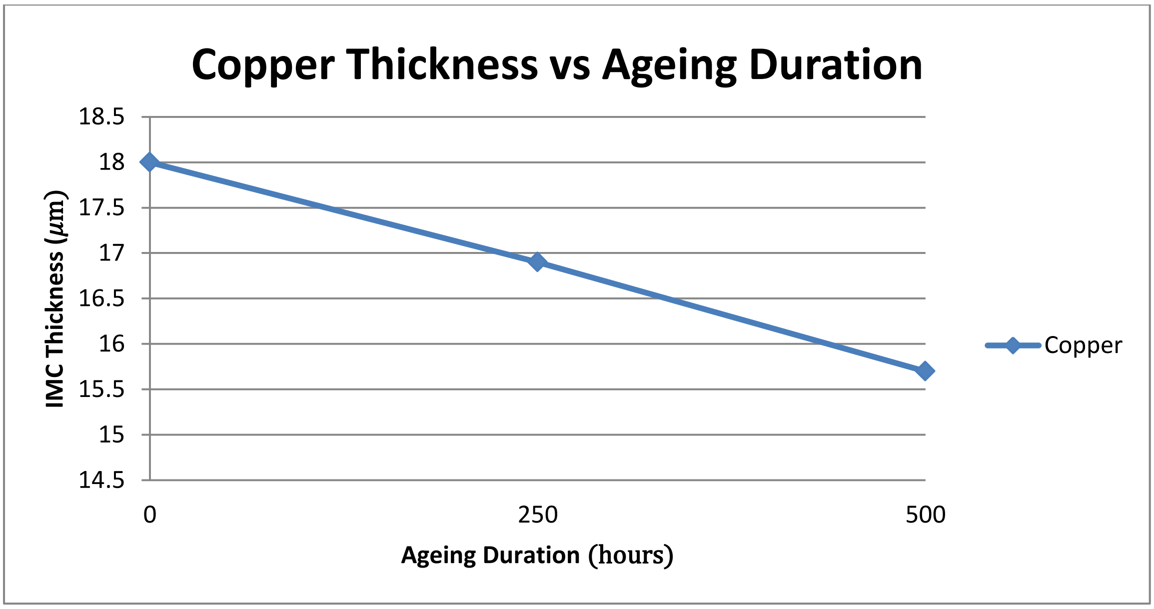


Figure 4.1: Reduction of Copper thickness at solder joint on Copper sample

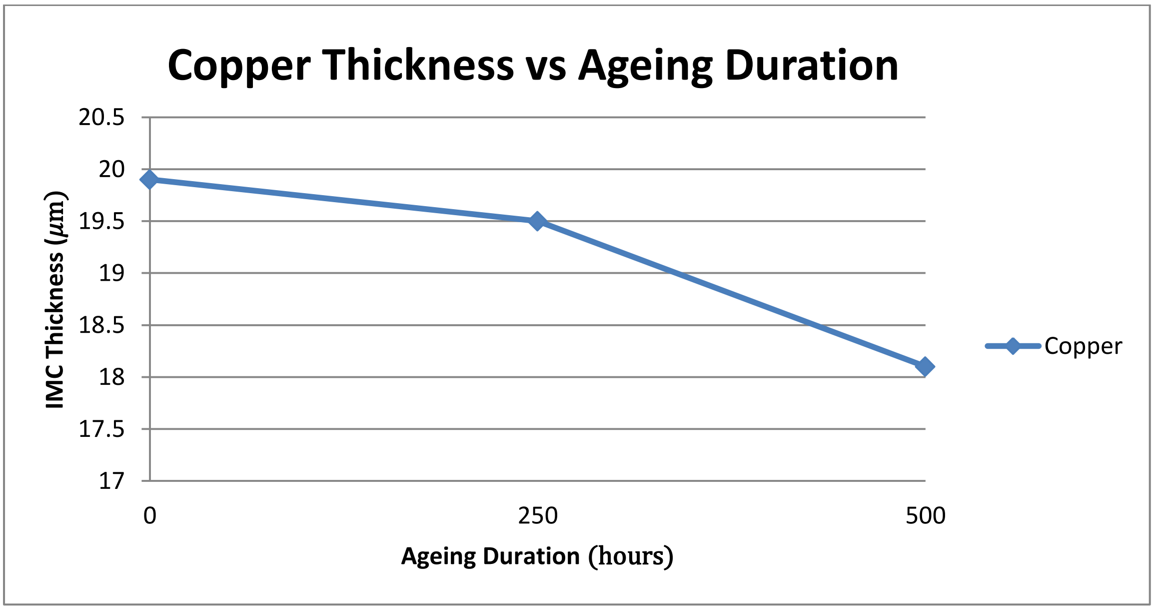


Figure 4.2: Reduction of Copper thickness at solder joint on Electroless Nickel sample

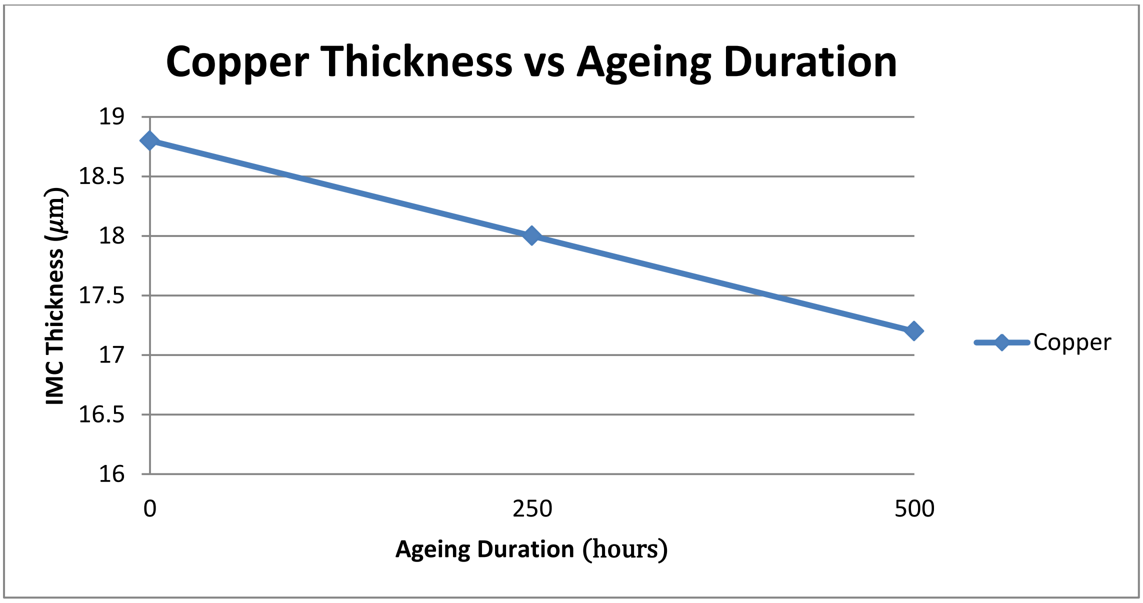


Figure 4.3: Reduction of Copper thickness at solder joint on ENIG sample

From Figure 4.4 and Figure 4.5, the graphs show the thickness of Nickel on solder joint at Electroless Nickel and ENIG sample become thicker when the duration of ageing increase. For gold layer at ENIG sample, the entire layer has been dissolve rapidly into the molten solder during reflow soldering, allowing the Nickel layer to be exposed the molten solder.

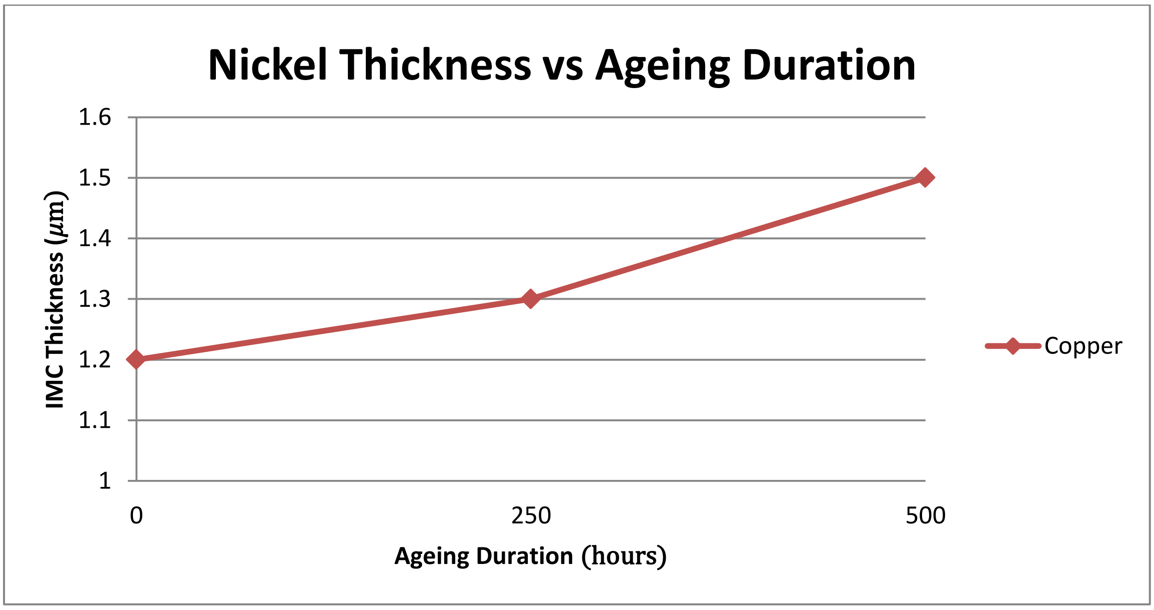


Figure 4.4: Nickel thickness at solder joint on Electroless Nickel sample

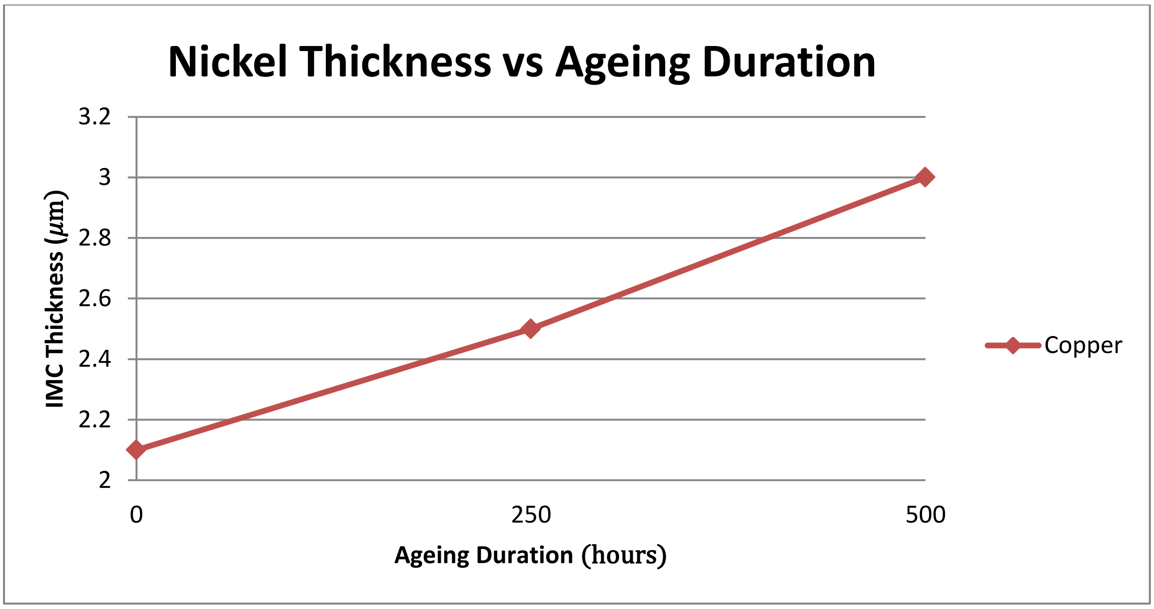


Figure 4.5: Nickel thickness at solder joint on ENIG sample

Based on the data from Table 4.4, two graphs have been plotted to show the IMC thickness formed for samples with different coating types and ageing duration respectively. In this research, the bare Cu samples act as reference to be compared with Electroless Nickel and ENIG samples. Figure 4.6 and Figure 4.7 shows the thickness of IMC against different coatings type graphs. From the Figure 4.6, the thickness of IMC from Copper is thicker than Electroless Nickel and ENIG.

Table 4.4: Mean value of IMC thickness on different coating type

Coating Type	**Ageing Duration (μm)**		
	0 hours	250 Hours	500 Hours
Copper Substrate	1.8	2.9	4.1
Electroless Nickel	0.4	1.4	2.1
ENIG	0.8	1.6	2.4

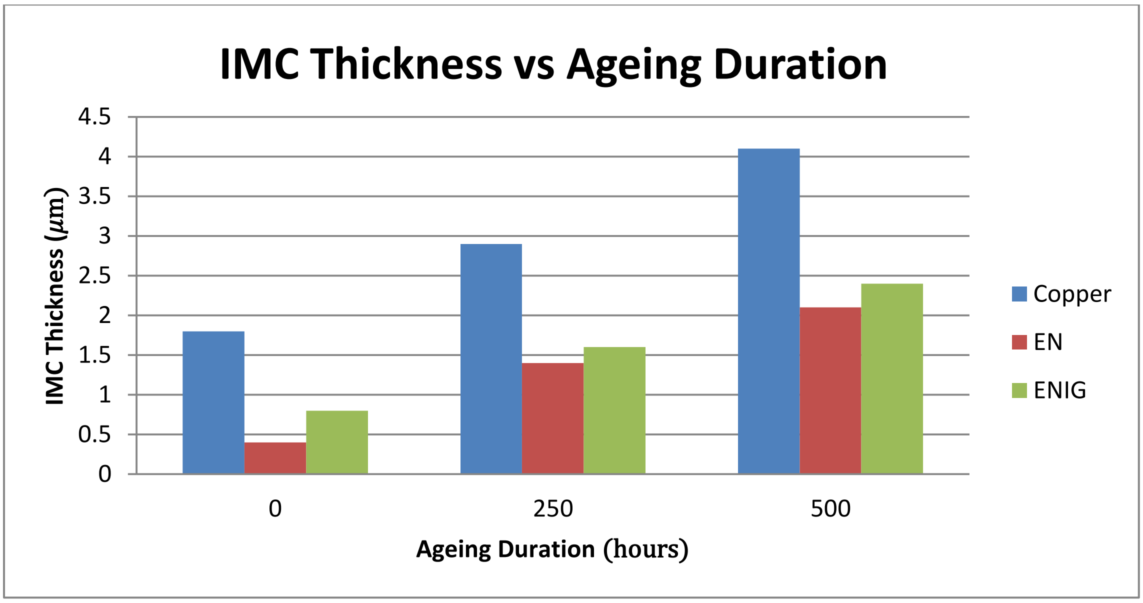


Figure 4.6: IMC Thickness on Different Coating Types

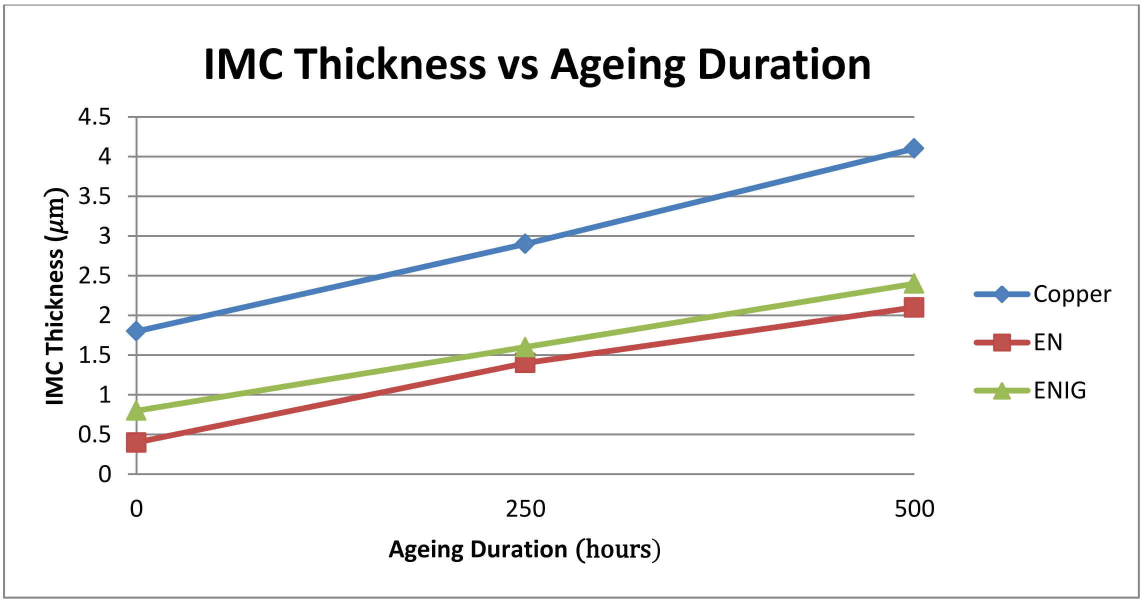


Figure 4.7: IMC Thickness versus Ageing Duration

Electroless Nickel and ENIG have Nickel as one of the layer for the coating. From background research, Nickel has slower reaction rate with molten Sn than Cu, hence it is known as an effective diffusion barrier to prevent rapid inter diffusion between solder and copper base material.

Figure 4.8, Figure 4.9 and Figure 4.10 below show the IMC at solder joint on different coating types. IMC layer will form at the solder joint between solder and

Copper substrate. For Electroless Nickel and ENIG sample, IMC layer will form with Nickel layer between solder and Copper substrate.

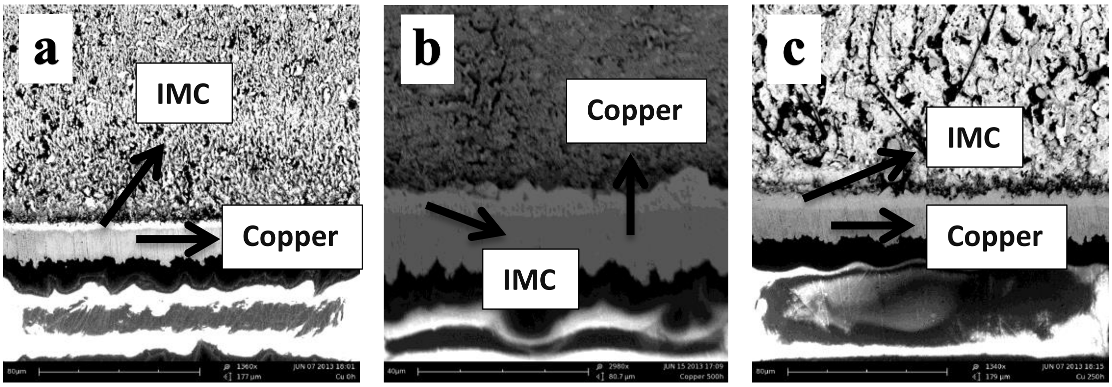


Figure 4.8: IMC at Copper substrate sample at ageing duration (a) 0, (b) 250 Hours, (c) 500 hours

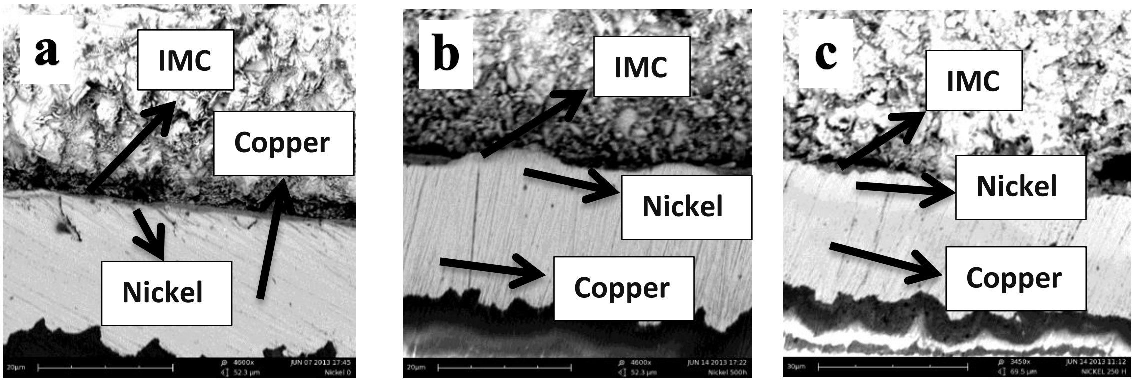


Figure 4.9: IMC at Electroless Nickel surface finish sample with ageing duration (a) 0, (b) 250 hours, (c) 500hours

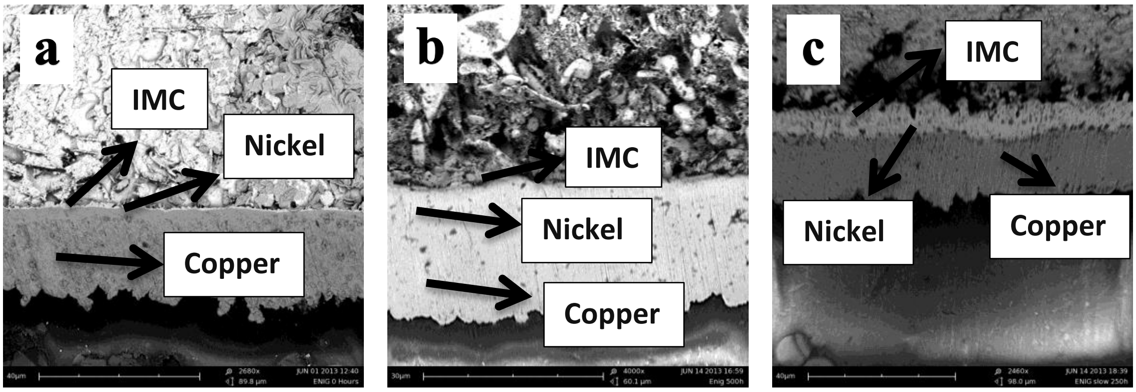


Figure 4.10: IMC at ENIG surface finish sample with ageing duration (a) 0, (b) 250 hours, (c) 500 hours

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

As a conclusion, the duration of ageing are important for the formation of IMC at solder joint. From the experiment, the IMC layer thickness increases with ageing duration. The IMC thickness at Copper sample after reflow is 1.8μm and increasing to 2.9μm after isothermal ageing at duration 250 hours and 4.1μm at ageing duration 500 hours. For Electroless Nickel sample, the IMC thickness after reflow is 0.4μm. It is increases to 1.4μm after ageing at duration 250 hours and 2.1μm after ageing at duration 500 hours. IMC thickness for ENIG sample is 0.8μm and increasing to 1.6μm when the sample is subjected to the isothermal ageing with duration 250 hours and increasing to 2.4μm when the duration is at 500 hours. From the result, it is shows that even the duration of ageing treatment is in short period, it still can lead the increasing of IMC Thickness.

Other than that, IMC at copper sample is thicker than IMC at Electroless Nickel and ENIG. Thicker IMC layer will lead to solder joint failure where fracture occurs at the interface between solder and IMC layer due to mechanical forces such as vibration and expansion caused by variation in temperature. For a good metallurgical bonding between solder and substrate, a thin and uniform IMC layer is needed.

From the experiment also, copper substrate that have Nickel layer as their coating will give a good diffusion barrier to prevent rapid inter diffusion between solder and copper base material

5.2 RECOMMENDATION

For recommendation for this research, do the further ageing treatment for a longer periods to analyzes the effect of IMC on surface finish. Ageing treatment will lead to an increasing IMC thickness even the duration is in short period. So, try to investigate whether the increasing of IMC thickness have a limit or will increase due to increasing ageing duration and the effect at solder joint strength.

Other than that, for recommendation for this research also, use other surface finish such as ENEP, ENEPIG, Silver and etc. to investigate the IMC thickness on solder joint and the effect on surface finish.

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