

BORANG PENGESAHAN STATUS TESIS

JUDUL **EFFECT OF HEAT TREATMENT TEMPERATURE OF SN-0.7CU SOLDERS MATERIAL ON MICROSTRUCTURE**

SESI PENGAJIAN: **2007/2008**

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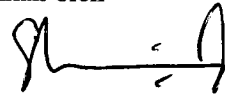
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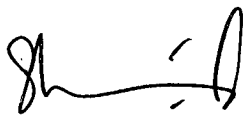
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**EFFECT OF HEAT TREATMENT TEMPERATURE ON SN-0.7CU SOLDERS
MATERIAL ON MICROSTRUCTURE**

NABHAN BIN ISMIL

**A report submitted in partial fulfillment
of the requirements for the award of
the Degree of Bachelor of
Mechanical Engineering**

**Faculty of Mechanical Engineering
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NOVEMBER 2007

I declare that this Project report entitled "*Effect of Heat Treatment Temperature of Sn-0.7Cu Solders Material on Microstructure*" is the result of my own research except as cited in the references. The report has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

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Date : 27 November 2007

**This thesis is dedicated to
My beloved father Ismil bin Eman
My beloved mother Che Pura binti Abd Rashid
My beloved brothers Mohd Faisal bin Ismil & Habil bin Ismil
My beloved sisters Raihan binti Ismil & Izzati binti Ismil**

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ABSTRACT

Soldering plays the most important role for joining technology in electronic industry. The conventional tin-lead solders have been used for a quite long time in electronic industries. However, since lead is a toxic element and harmful to individual health and environment, many researchers have proposed lead-free solder to protect individual health and environment as well. The objective of this study is to analyze the microstructure at the solder and base metal interface. This study investigates the interfacial reactions between Sn-0.7Cu solder material and copper substrate before and after aging at 100°C, 150°C and 200°C for 1 hour, 3 hours and 5 hours. Copper substrates are connected to each other by manual soldering. After soldering, the intermetallic compound formed at the interface is Cu_6Sn_5 intermetallic compound. The thickness of the intermetallic compound increases as the aging time and temperature increased. Intermetallic compound layer must be kept at sufficient thickness because excessive amount of intermetallic compound can generate defect and affect the solder joint reliability.

ABSTRAK

Pematrian memainkan peranan yang penting untuk teknologi penyambungan di dalam industri elektronik. Pateri timah-plumbum yang biasa telah lama digunakan di dalam industri elektronik. Walau bagaimanapun, plumbum merupakan unsur toksik dan berbahaya kepada kesihatan individu dan alam sekitar dan ramai penyelidik telah mencadangkan pateri bebas plumbum untuk menjaga kesihatan manusia dan juga alam sekitar. Objektif projek ini adalah untuk menganalisa struktur mikro pada permukaan pateri dan logam asas. Projek ini menyiasat tindak balas di permukaan antara pateri Sn-0.7Cu dan logam asas sebelum dan selepas aging pada 100°C, 150°C and 200°C untuk 1 jam, 3 jam dan 5 jam. Logam asas disambungkan sesama sendiri menggunakan teknik pateri secara manual. Selepas pematrian di buat, intermetallic compound yang terbentuk adalah Cu_6Sn_5 . Ketebalan intermetallic compound tersebut bertambah apabila masa dan suhu aging meningkat. Jalur intermetallic compound mestilah ditentukan pada ketebalan yang memadai kerana amaun intermetallic compound yang melampau boleh menyebabkan kerosakan dan memberi kesan kepada keutuhan penyambungan pateri.

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LIST OF SYMBOLS AND ABBREVIATIONS

μ	-	Micro
SEM	-	Scanning electron microscopy
EDX	-	Energy dispersive X-ray
IMC	-	Intermetalli compound
Sn	-	Stanium
Cu	-	Copper
Pb	-	Plumbum
Ag	-	Argentum
Au	-	Aurum
Zn	-	Zinc
Ni-P	-	Nickel- Phosphorous
IC	-	Integrated circuit
PCB	-	Printed circuit board

CHAPTER 1

INTRODUCTION

1.0 Introduction

Printed circuit board is used widely in microelectronic industry. This circuit board is the brain behind computers and large electronic systems. Today's integrated circuits are smaller in size and the fabrication technology becomes more advanced. Soldering is the most important joining technology in microelectronics. Solder plays an important role to provide electrical, thermal and mechanical continuity in electronic assemblies. Tin-lead solders have been used in microelectronic industry for decades. However, lead is a toxic element and is harmful to individual health. According to D.R.FrearX et al. (2001), medical studies have shown that lead is a heavy-metal toxin that can damage the kidneys, liver, blood, and central nervous system.

This tin-lead solder has sufficient mechanical properties besides its low cost, low melting temperature and ease of handling. (Titus Chellaih et al, 2006). The needs of green products reflect to the foundation of lead free solder which is environmental-friendly solder. Many researchers have proposed the lead free solder candidates to replace the conventional Sn-Pb solder but it is quite hard to find the lead-free solder that suitable for meeting all requirement including mechanical properties, manufacturability and cost as well.

Many researches have been conducted to investigate the interfacial reactions at the solder joint which is between the solder and the pad such as copper pad. Intermetallic compound is growth at the interface of the solder joint. For long term

aging, the existence of intermetallic compound is varying for the grain size, thickness and its effect to the solder reliability.

Work by Aditya Kumar et al. (2005) has shown that, Cu reacts rapidly with Sn of the solder forming thick layers of Cu–Sn intermetallics. The intermetallic compound over-growth problem is bad in the case of lead-free solders, used due to the legislation and environmental concerns.

1.1 Project Objectives

The project objectives are:

- i. To investigate the effects of aging temperature on the growth of intermetallic compounds. Aging test will be conducted with different time and temperature. The changes on temperature and time will effect the growth of intermetallic compound in the solder material.
- ii. To analyze the microstructure at solder material before and after aging. Scanning Electron Microscope (SEM) equipped with EDX is used to analyze the microstructure.

1.2 Project Scopes

The scopes of this project are:

- i. Soldering of Sn-0.7Cu solder material and copper base metal. The copper metal is connected to other one with manual soldering.
- ii. Aging process at different time and temperature.
- iii. Analyze the microstructure of the samples before and after aging using SEM with EDX. The existence of intermetallic compound is investigated.

1.3 Problem Statement

The presence of intermetallic compound between the solder alloys and base metal ensures a good bonding. However, due to its brittle nature and weakness, the excessive growth of intermetallic compound at the interface layer will have tendency to generate structural defects to the solder joint (Peng Sun et al 2006). The microstructure will be investigated after high temperature aging at different temperature. It is well known that the thickness and grain size of intermetallic compound will increase due to increasing of aging temperature (M.J.Rizvi et al. 2006).

The effect of heat treatment process to the Sn-0.7Cu solder/Copper joint on microstructure will be observed.

1.4 Thesis Disposition

Chapter one presents the introduction of the project including general introduction of the project, objective, scope, and problem statement. The objective and scope determine the boundary and purpose of the study specifically. Thus, there will be more focus on the project. This is to make sure that the project is performed under specific scope.

Chapter two is about the literature study of the project. The information about the project is explained in this chapter. Literature review is a study to gather information on lead-free solder and the interfacial reaction between solder material and base metal. Regarding this study, the formation of intermetallic compound for various solders and base metals are known and the effect of aging time and temperature on the formation of intermetallic compound as well.

Chapter three shows the methodology of the project. The methods applied in this project are explained step by step. The flowchart included in this chapter shows

the flow of the project from beginning till the end. Each activity in conducting the experiment is clearly stated.

Chapter four presents result and discussion. The result for the experiment is obtained from the analysis of interfacial reaction between solder and base metal using SEM equipped with EDX. SEM or Scanning Electron Microscope is used to analyze the existence of intermetallic compound on the microstructure. EDX is help to determine the element exist on the microstructure. The difference situation regarding intermetallic compound is being discussed in this chapter. The aging time and temperature affected the growth of intermetallic compound formation

Chapter five presents the conclusion and recommendation. Conclusion is made due to the observation and analysis of the specimens before and after aging using SEM.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Chapter two presents the literature study of the project. This chapter is explains about the scope which is related to the project. Thus, the reader will get the idea about the whole project. Basically, this chapter consist some information about the integrated circuit which is widely used in electronic applications. Integrated circuit plays an important role in many applications such as in computer, mobile phone and other digital appliances.

Joining technology or soldering process in electronics industry is a general scope in this project. Based on the project title, Sn-0.7Cu solder material is the main focus. However, this chapter also describe about tin-lead solder that has been used earlier than lead-free solder. Aging, which is a heat treatment process also included in this chapter. Aging is done to improve the properties of the solder joint. Reflect to the aging process, intermetallic compound is growth at the interface layer of solder material and base metal. The information of intermetallic compound is included in this chapter as well.

2.2 Integrated Circuit (IC)

An integrated circuit (IC), sometimes called a chip or microchip, is a semiconductor wafer on which thousands or millions of tiny resistors, capacitors, and transistors are fabricated. An IC can function as an amplifier, oscillator, timer,

counter, computer memory, or microprocessor. Only a half century after their development was initiated, integrated circuits can be found anywhere.

Computers, cellular phones, and other digital appliances are now important parts of the structure of modern societies. That is, modern computing, communications, manufacturing and transport systems, including the Internet, all depend on the existence of integrated circuits. Indeed, many scholars believe that the digital revolution brought about by integrated circuits was one of the most significant occurrences in the history of mankind.

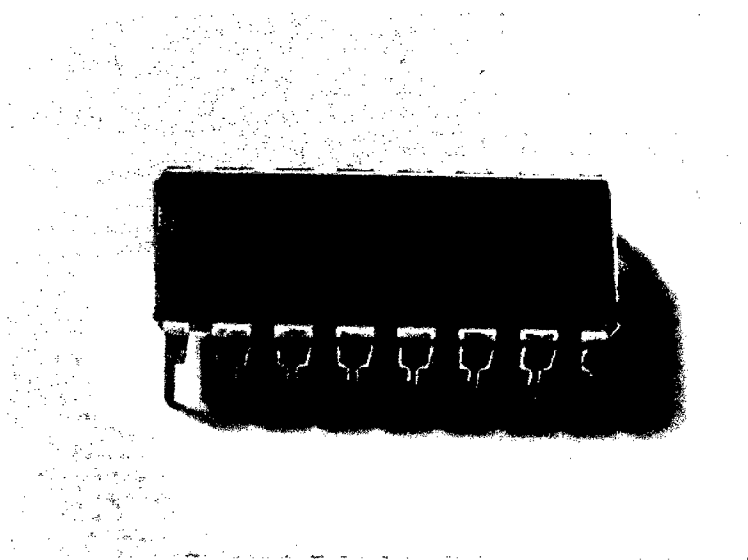


Figure 2.1: Integrated circuit

2.3 Soldering in Electronic Industry

Soldering is the most important joining technology in microelectronics. This is due to the emerging of flip-chip technology. Flip-chip interconnects are the electrical and mechanical connections between the semiconductor integrated circuit and the package (or board for direct-chip attach). Soldering is a significant joining process of solder material. According to (Cristina Anderson et al; 2006), solder plays

a crucial role in assembly and interconnection of electronic products. Nowadays, flip-chip technology is expanding widely.

The size of integrated circuit migrated to smaller sizes over the years, allowing more circuitry to be packed on each chip. This makes the integrated circuit more compact and more resistors, capacitors and transistors can be fabricated inside the integrated circuit. Due to this increased capacity per unit area, the production cost can be decreased and/or increase functionality. In the last fifty years, the electronic industry has relied mainly on one type of solder (Sn-Pb solder) in the manufacturing of computer chips, circuitry and other electronic equipments (Sang Won Kim et al; 2004).

The function of solder is to join 2 or more metals at temperatures below their melting point. Solder provides a metal solvent action between the solder and metal(s) being joined. This "solution" of metal in the solder results in an intermediate alloy being formed. This provides metal and electrical continuity. Soldering is primarily used to provide a convenient joint to ensure electrical contact or seal against leakage. Solders typically do not provide high mechanical strength. Soldering is used extensively in the electronics industry printed circuit boards.

2.3.1 Benefits of Tin-Lead Alloys

The benefits of Tin-Lead alloys are:

- i. Lowest cost joining method compared to welding, brazing, mechanical joining
- ii. Very fast method, easy, low skill level required
- iii. Versatile heat sources - soldering iron, torch, oven
- iv. Minimal effect on metals jointed
- v. Easy to take joints apart
- vi. Low melting point
- vii. High electrical conductivity
- viii. Relatively strong joint at and below room temperature

Table 2.1: Sn-Pb alloy mechanical properties

Tin (wt. %)	Tensile Strength (MPa)	Shear Strength (MPa)	Elongation (%)	Elastic Modulus (GPa)	Izod Impact Strength (J)	Stress to produce 0.01%/day creep rate (kPa)
0	12	12	55	18.0	8.1	1700
5	28	14	45	18.5	9.5	1400
10	30	17	30	19.0	10.8	
20	33	20	20	20.0	15.0	
30	34	28	18	21.0	16.3	790
40	37	32	25	23.7	19.0	
50	41	36	35	26.9	20.3	860
60	52	39	40	30.0	20.3	
63	54	37	37	31.5	20.3	2300
70	54	36	30	35.0	19.0	

Tin, lead and their alloys, due to their low melting temperatures and wide availability, are the most commonly used solder materials. As can be seen from the above table, the 63% tin 37% lead solder alloy results in the maximum tensile strength, shear strength, impact strength, and resistance to creep. This 63-37 composition is also known as the eutectic point of the alloy, where the alloy behaves like a pure metal having a single melting (solidification) temperature (176°C / 349°F). This is a good operational feature. Once the solder melts on application of heat, it solidifies immediately on removal of heat, without going through a pasty stage like other alloys. This allows for predictable soldering and fast cycle times.

2.4 Lead- Free Solder

According to (Jeong-Won Yoon and Seung-Boo Jung; 2005), a new environmental requirement has been the new challenges to the electronics industry. Electronic products and assemblies must be environmental-friendly. This can be fulfilled by make them lead-free. For the first phase, lead will be eliminated from the solder. This is the first step to make the products toward the environmental needs. Instead using tin-lead solder in joining technology, lead-free solder takes place as a solder for joining in the electronic industry. Regarding to the need of green product, lead-free solder is widely used in electronic industry.

Since lead is a toxic element and is harmful to individual health and to the environment, the development of lead-free solder has been a great concern in recent years (Xin Ma et al; 2005). A lot of researches have been conducted to find the promising lead-free solder that can replace tin-lead solder in soldering process. Usually, lead-free solder consists of binary and ternary phases. The ternary phases of lead-free solder such as Sn-Ag-Cu and Sn-Ag-Bi and for binary phases, such as Sn-Ag, Sn-Cu, Au-Sn, Sn-Zn, Sn-Bi and Sn-Sb. Jeong-Won Yoon (2005) has stated that among many lead-free solder alloys, a eutectic Sn-Cu solder (Sn-0.7wt. %Cu) is considered the most promising candidate alloy to replace the eutectic Sn-Pb solder. And this type of solder might be suitable for high temperature applications such as in automotive industry. This study is focusing on Sn-0.7Cu as a solder material.

2.5 Aging

Important basic properties such as strength, hardness, ductility, and toughness as well as resistance to wear are greatly influenced and modified by alloying elements and by heat treatment processes. The common example of property improvement is heat treatment. It modifies microstructures and thereby produces a variety of mechanical properties. Aging is one type of the heat treatment processes. Aging is relies significantly on time and temperature. This relationship must be observed in order to obtain desired properties. Alloys must be kept at elevated

temperature for hours to allow precipitation to take place. This time delay is called aging.

2.6 Intermetallic Compound

Intermetallic compounds are complex structures consisting of two metals in which solute atoms are present among solvent atoms in certain proportions, thereby forming a new chemical compound. Intermetallic compounds are strong, hard and brittle. They have high melting points and, strength at elevated temperatures, good oxidation resistance and relatively low density. Because of these reasons, they are candidate for advanced-gas turbine engines.

Typical examples are the aluminides of titanium (Ti_3Al), nickel (Ni_3Al) and iron (Fe_3Al). Intermetallics is the short summarizing designation for such intermetallic phases and compounds, i.e. chemical compounds between two or more metals with crystal structures which differ from those of the constituent metals. In a mechanical context, such compounds often offer a compromise between ceramic and metallic properties when hardness and/or resistance to high temperature are important enough to sacrifice some toughness and ease of processing. They can also display desirable magnetic, superconducting and chemical properties, due to their strong internal order and mixed (metallic and covalent/ionic) bonding, respectively. Intermetallics of aluminum and gold are a significant cause of wire bond failures in semiconductor devices and other microelectronics devices.

The inter-metallic layer must form between the solder material and the base metal. This is to enhance the good bonding otherwise the solder simply solidifies over the base metal without forming any bond. However, high ambient temperatures on the solder joint result in the growth of unwanted intermetallic compounds which weaken the strength of the solder joint due to their brittleness and weakness (Seong-Boo Jung et al; 2005).

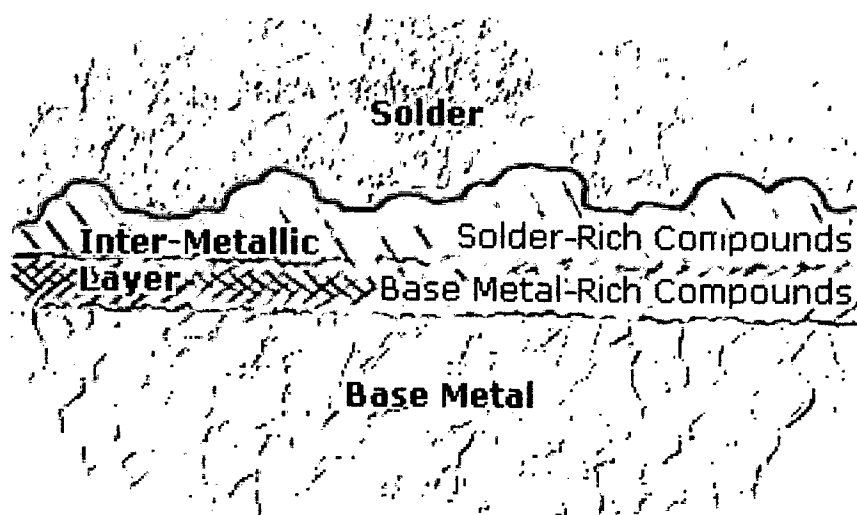


Figure 2.2: Intermetallic layer between solder and base metal

Within each inter-metallic layer, there are actually a number of different compounds formed by the solder materials and the base metal. These compounds are typically quite brittle and will adversely affect the integrity of the solder joint. As the joint is subject to stress, thermal cycles, vibration, or shock, the inter-metallic layers are usually where it starts to fail. Since the inter-metallic layers are inevitable, it is best to keep it as thin as possible.

2.7 Previous Study

This section is to show several journals that have similarity with the project. These journals are basically about interfacial reaction at solder and substrate interface during high temperature aging.

2.7.1 Interfacial Reaction and Mechanical Properties of Eutectic Sn-0.7Cu/Ni BGA Solder Joints during Isothermal Long-term Aging

Jeong- Won Yoon and Seung-Boo Jung (2005) were studied on interfacial reaction of eutectic Sn-0.7Cu/Ni solder joints during isothermal aging. They were investigated the interfacial reactions and growth kinetics of intermetallic compound (IMC) layers formed between Sn-0.7Cu solder and Au/Ni/Cu substrate at aging temperature of 185°C and 200°C for aging times up to 60 days. The solder ball and the substrate was bonded in a reflow process by place it in a reflow machine at maximum temperature of 250°C for 60 seconds. After reflow process, the intermetallic compound that formed at the interface was $(\text{Cu, Ni})_6\text{Sn}_5$.

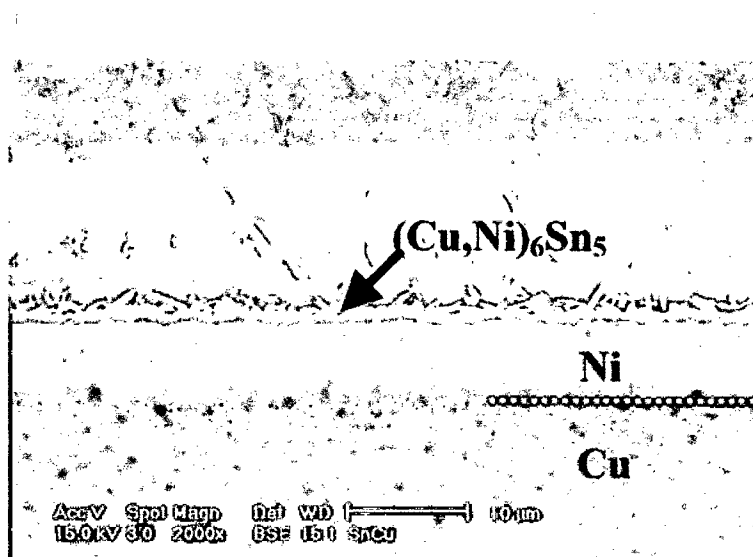


Figure 2.3: SEM micrograph of the Sn-0.7Cu solder/Ni interface reflowed at 250°C for 60 seconds

After aging at 185°C for 3 days, two intermetallic compound $(\text{Cu, Ni})_6\text{Sn}_5$ and $(\text{Ni, Cu})_3\text{Sn}_4$ were observed. These intermetallic compounds also growth at the interface after aging at 200°C for 1 day. Due to the restriction of supply of Cu atoms, the growth of $(\text{Ni, Cu})_3\text{Sn}_4$ intermetallic compound consumed the $(\text{Cu, Ni})_6\text{Sn}_5$ intermetallic compound at aging temperature of 200°C. This is caused the decreasing of Cu diffusion to the interface. Besides, the growth of the upper $(\text{Cu, Ni})_6\text{Sn}_5$ intermetallic compound is because of the availability of Cu in solder matrix.

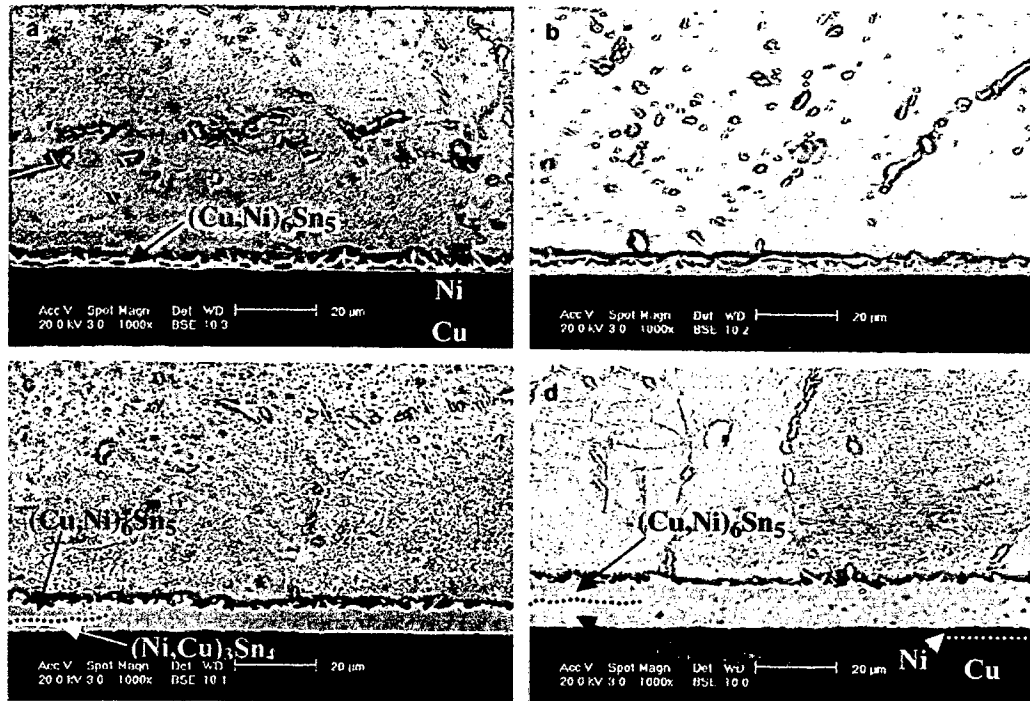


Figure 2.4: SEM micrograph of the Sn–0.7Cu solder/Ni BGA joints aged at 185 °C for various times; (a) 1 day, (b) 3 days, (c) 15 days and (d) 50 days.

After aging at 185 °C for 50 days, a very thick and uniform intermetallic layer was formed as in figure 2(d).

Figure below shows the SEM micrograph after aging temperature of 200°C at various times. A thick intermetallic compound layer was formed after aged for 1 day. A thicker $(\text{Ni}, \text{Cu})_3\text{Sn}_4$ intermetallic compound was observed after aged for 50 days. At this stage, $(\text{Cu}, \text{Ni})_6\text{Sn}_5$ intermetallic compound was not observed.

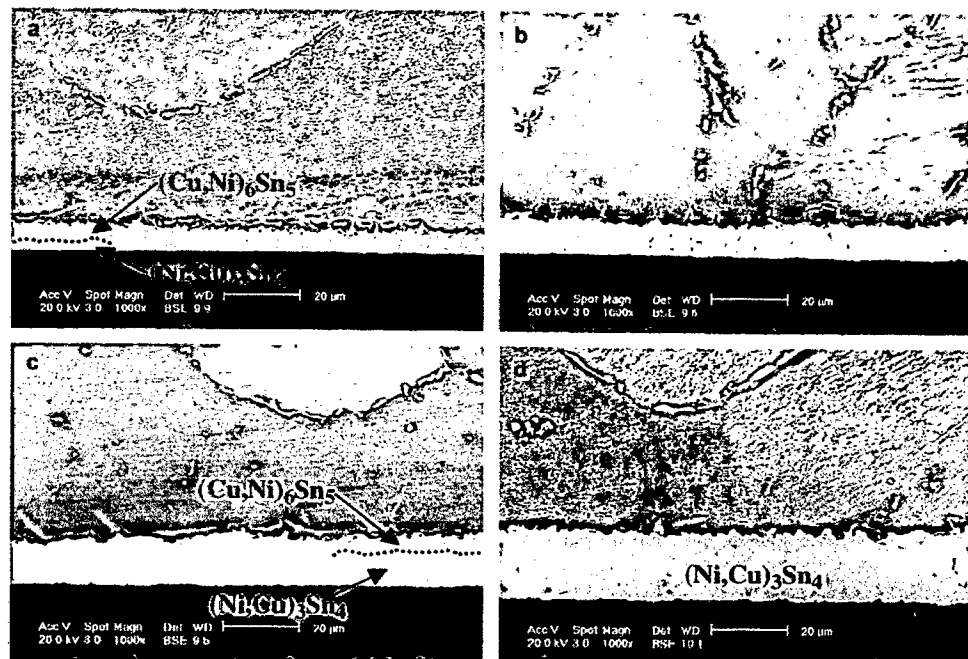


Figure 2.5: SEM micrograph of the Sn–0.7Cu solder/Ni BGA joints aged at 200 °C for various times; (a) 1 day, (b) 6 days, (c) 15 days and (d) 50 days.

Figure 2.6 shows the SEM micrographs of the Sn–0.7Cu solder/Ni/Cu interface aged at 200 °C for 60 days. Ni layer of the substrate was completely consumed after aged for 60 days. As the intermetallic compound layer contacted with Cu layer, a Cu_3Sn intermetallic compound layer was formed underneath $(\text{Ni,Cu})_3\text{Sn}_4$ intermetallic compound layer.

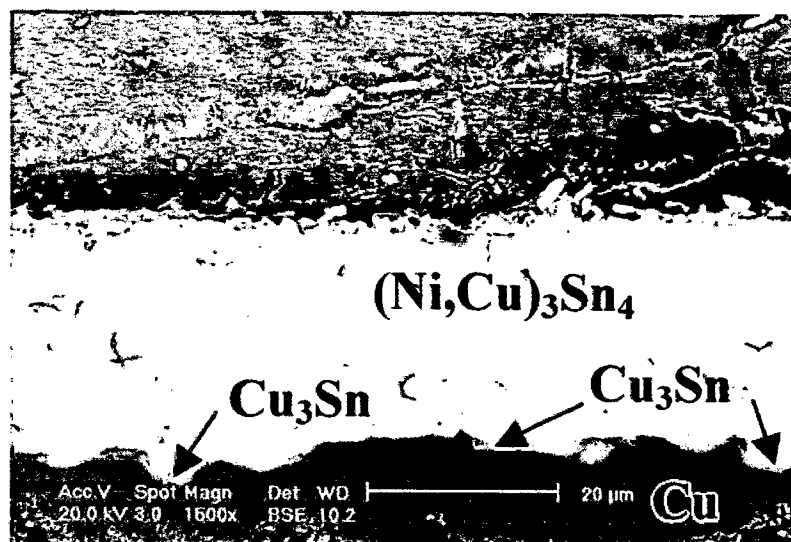


Figure 2.6: SEM micrographs of the Sn–0.7Cu solder/Ni BGA joints aged at 200 °C for 60 days.

As a conclusion from the journal, after the entire Au layer dissolved into the molten solder during reflow process, a $(\text{Cu, Ni})_6\text{Sn}_5$ intermetallic compound was formed resulted by reaction between Ni layer and the molten solder. After aging at 185 °C for 3 days, two intermetallic compounds layers $(\text{Cu, Ni})_6\text{Sn}_5$ and $(\text{Ni, Cu})_3\text{Sn}_4$ were observed. A Small amount of Au contained in the upper $(\text{Cu, Ni})_6\text{Sn}_5$ intermetallic compound layer. As the aging time increased, the growth of intermetallic compound layers $(\text{Cu, Ni})_6\text{Sn}_5$ and $(\text{Ni, Cu})_3\text{Sn}_4$ also increased. Two intermetallic compounds $(\text{Cu, Ni})_6\text{Sn}_5$ and $(\text{Ni, Cu})_3\text{Sn}_4$ were observed after aged at 200 °C for 1 day. The growths of the intermetallic compound layers are much faster than aging temperature at 185 °C. At this temperature, these two intermetallic compound layers are growth after 3 days. But just 1 day for aging temperature at 200 °C. The growth of intermetallic compound is faster as the aging temperature increased.

2.7.2 High Temperature Aging Study of Intermetallic Compound Formation of Sn-3.5Ag and Sn-4.0Ag-0.5Cu Solders on Electroless Ni(P) Metallization

Peng Sun et al. (2006) were studied on intermetallic compound formation of Sn-3.5Ag and Sn-4.0Ag-0.5Cu solder pastes on electroless Ni(P) metallization. The solder pastes that used are Sn-3.5Ag and Sn-4.0Ag-0.5Cu and the substrate was a copper pad deposited with electroless Ni-P immersion Au surface finish. Reflow soldering was done using reflow oven with nine different temperature zones. After high temperature aging for Sn-3.5Ag solder pastes at 150°C, Ni₃Sn₄ intermetallic compound were found. The samples were aged at 150°C for 168, 500 and 1000 hours. After aged at 1000 hours, Ni₃Sn₄ particles lost contact to the electroless Ni (P) layer and clear gap was observed after high temperature aging. Intermetallic compound formation between the solder and the substrate usually improves the wettability of liquid solder. A thin and uniform intermetallic compound layer is an essential requirement for good bonding.

Figure 2.7 shows the cross-sectional SEM images of Sn-3.5Ag solder joints after aged at 150°C for various times. The interfacial reaction resulted in the formation of large particles of Ni-Sn intermetallic compounds. The intermetallic compounds were detected just above the interface. As shows in figure 2.7(c), after 1000 hours aging, there was a clear gap existing between the intermetallic compound layer and the electroless Ni(P) layer. This would affect the solder joint reliability. As shown in figure 2.7(a) and 2.7(b), the Ni-Sn intermetallic compound layer was separated away from the interface. This phenomenon was clearly observed in figure 2.7(c).

The voids were also observed after aging for 168 hours. The clear gap that existed after aging for 1000 hours is resulted by the growth and interconnection of the voids. The intermetallic compound layer becomes thick during aging.

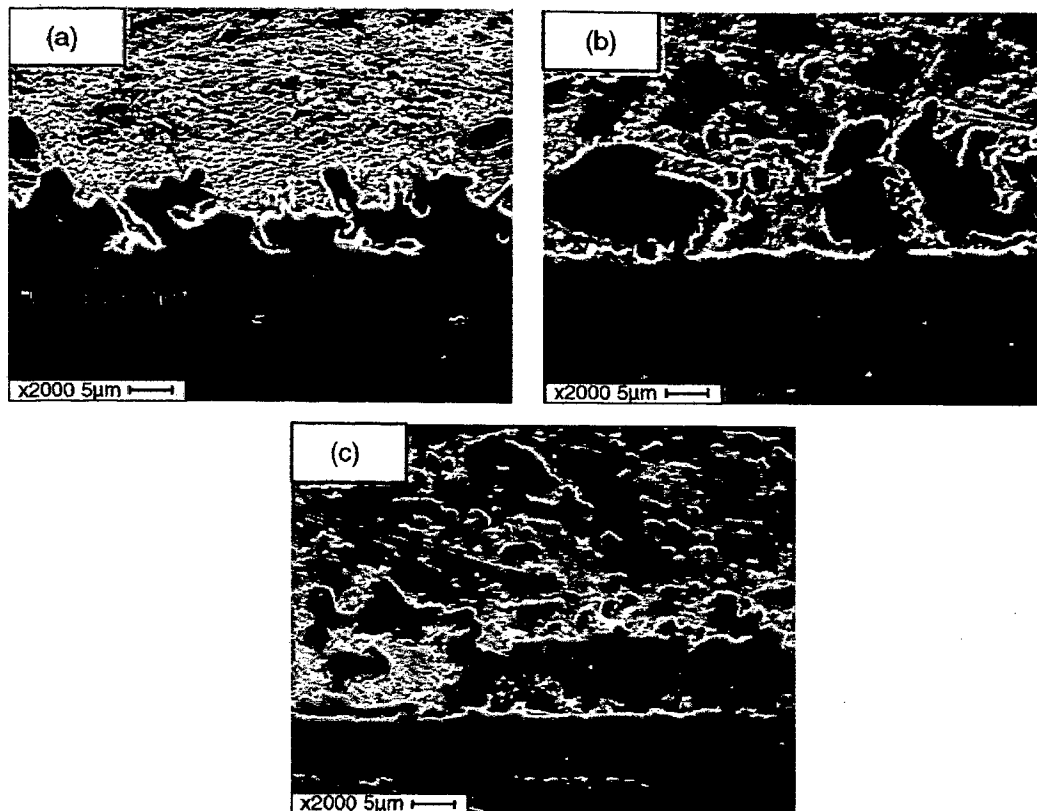


Figure 2.7: Cross-sectional SEM images of Sn-3.5Ag solder joints interface after different aging times at 150°C: (a) 168h, (b) 500h and (c) 1000hours

Two kinds of intermetallic compound were found after high temperature storage aging of Sn-4.0Ag-0.5Cu solder joint at 150°C for 168, 500 and 1000hr are $(\text{Cu,Ni})_6\text{Sn}_5$ and $(\text{Ni,Cu})_3\text{Sn}_4$.

Table 2.2: Summary of journals

Author(s)	Year	Field	Scope of study
Peng Sun et al	2006	Material Science and Engineering	Study on formation of intermetallic compound in Sn-Co-Cu, Sn-Ag-Cu and eutectic Sn-Cu solder joints on electroless Ni(P) immersion Au surface finish after reflow soldering
Jeon-Won Yoon and Seung-Boo Jung	2006	Alloys and compounds	Study on interfacial reactions between Cu/Sn-Ag-Cu/Cu (ENIG) sandwich solder joints during isothermal aging at 150°C up to 1000 hours.
M.J. Rizvi et al	2006	Alloys and Compounds	Study on the growth behavior of intermetallic layer with or without adding 0.3wt% Ni into the Sn-0.7Cu solder during wetting reaction on Cu-substrate and thereafter in solid-state aging condition.
Sinn-Wen Chen et al	2003	Electronic Materials	Study on the Ni/Sn/Ni and Ni/Sn-0.7wt.%Cu/Ni couples reacted at 200°C for various length of time. To examine the interfacial reactions, fracture surfaces and the tensile strength of the specimens.
Sang-Won Kim et al	2005	Alloys and Compounds	Study on the interfacial reaction and morphology change of intermetallic compound between eutectic Sn-Cu solder and Ni substrate under various reflow and cooling conditions.
Jeong-Won Yoon and Seung-Boo Jung	2005	Microelectronics	Study on the interfacial reactions and growth kinetics of intermetallic compound layers formed between Sn-0.7Cu solder and Au/Ni/Cu substrate at aging temperatures of 185°C and 200°C for aging times up to 60 days.
Xin Ma et al	2003	Material Science and Engineering	Study on the development of Cu-Sn intermetallic compound at the solder/Cu joint interface using Sn-3.8Ag-0.7Cu and Sn-2Ag-0.8Cu-0.6Sb alloys during solid-state thermal aging at 125°C.

CHAPTER 3

METHODOLOGY

3.1 Introduction

In order to completing this project, the methods used will be explained in this chapter. This chapter consist a flowchart which is experimental flowchart. Besides, the flowcharts show the project flow step by step.

This project begins with cutting the sheet metal which is copper into desired dimension. Every specimen is polished to remove dirt and other particles to ensure its cleanliness. This is important for the specimens to be observed then. Next, every single specimen is combined to another specimen by manual soldering technique. Following soldering, the samples were aged at different time and temperature. Finally, the microstructure of the aged samples were observed using SEM. The growths of intermetallic compound layers were observed. The microstructure of before-aging samples were observed using SEM as well. The experiment flowchart can be seen in figure 3.1 as below.

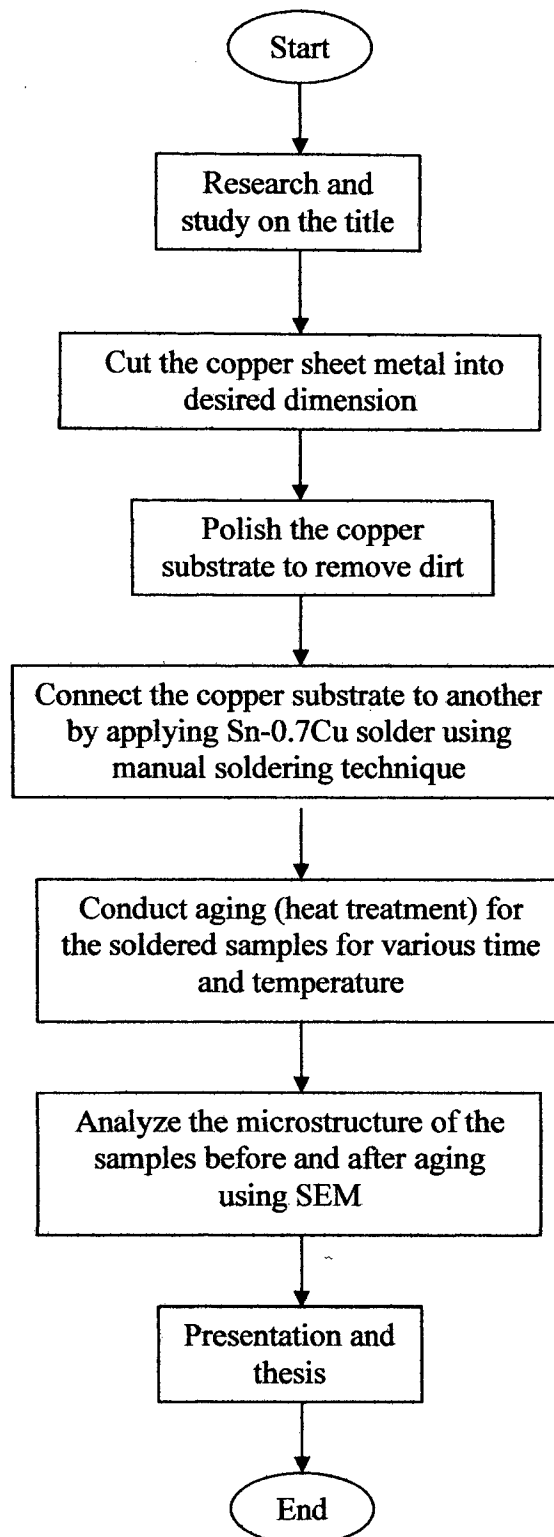


Figure 3.1: Experiment flowchart

1.2 Research and Study

In order to conduct the experiment, a lot of research and study has been performed. The information is gathered from journals, articles, books and other sources. The study scopes including the understanding of lead free solder, which is widely used in electronic industry and automotive industry as well. The study scope also includes soldering process, aging and the growth of intermetallic compound for various lead free solder and base metal. Furthermore, the methods of soldering, polishing and aging are also being defined.

3.3 Experimental Procedures

The experiment can be divided into five steps which are cutting, polishing, soldering, aging and analysis. There are 21 single copper metal specimens and the dimension of each specimen is 5cm x 3 cm and its thickness is 1 cm. Each two of the specimens is connected to each other by soldering and the total of the specimens after soldering are 12 specimens.

3.3.1 Cutting

The sheet metal with 1 cm in thickness is cut by the dimension (5 cm x 3 cm) using shear cutting machine as in figure 3.2. The copper sheet metal after cutting can be seen in figure 3.3. As mentioned before, there are 21 specimens that are used in the experiment.

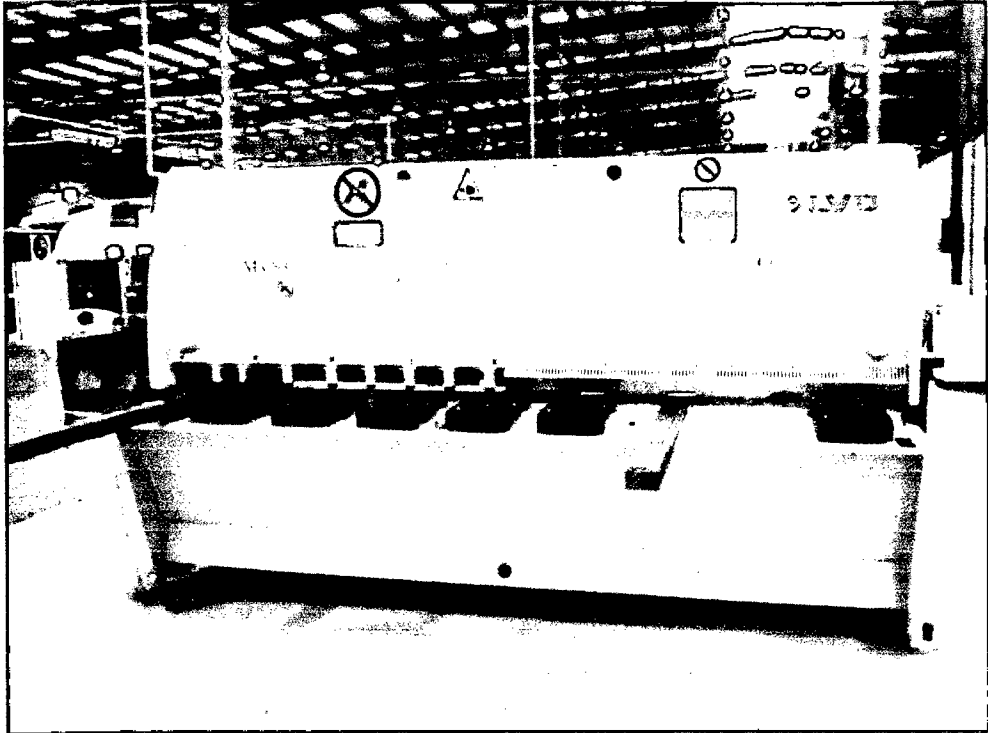


Figure 3.2: Shear cutting machine

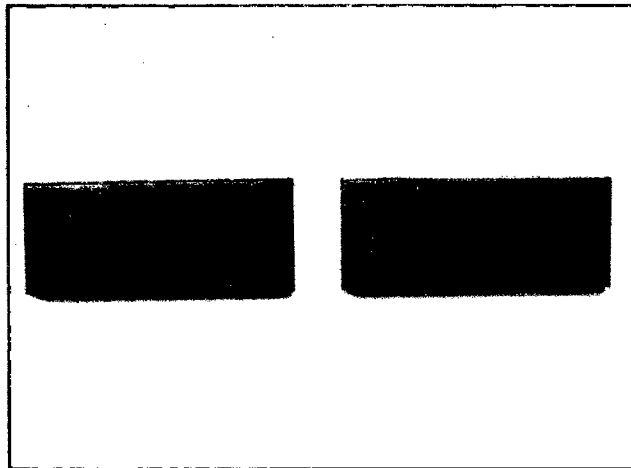


Figure 3.3: Copper sheet metal after cutting

3.3.2 Polishing

Every single specimen is polished slightly by applying ethanol to the surface of the copper metal. A piece of clean cloth is used to cleanse the copper plate surface to remove dirt. The reason behind polishing is to ensure good bonding between solder and the copper plate surface because solder cannot take part on dirty surface.

3.3.3 Soldering

After polishing, the copper plate is connected to each other by manual soldering technique. Sn-0.7Cu solder material is being soldered with copper plate to form an assembly of solder material and the copper plate. The equipments used in soldering process are Bunsen burner and Sn-0.7Cu solder. During soldering, Sn-0.7Cu solder material is melt and form contacts with the copper plate. Solder paste is applied to the copper plate surface to enhance the contact between solder and copper plate. The specimen then is being cooled for the solder to solidify. The solder joint can be seen in figure 3.4 and figure 3.5

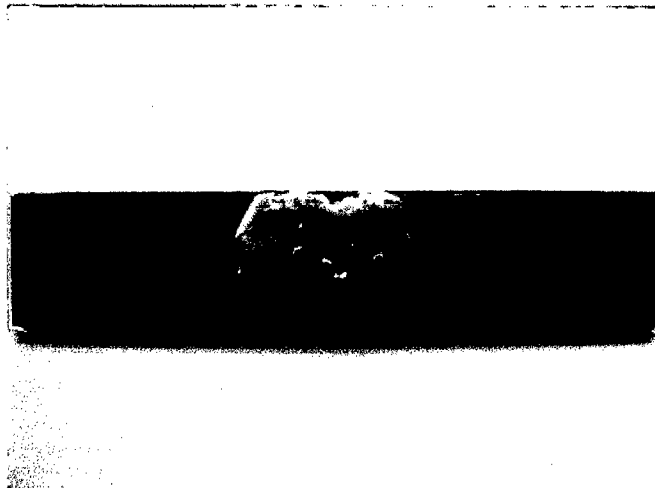


Figure 3.4: Solder joint between Sn-0.7Cu solder and copper substrate

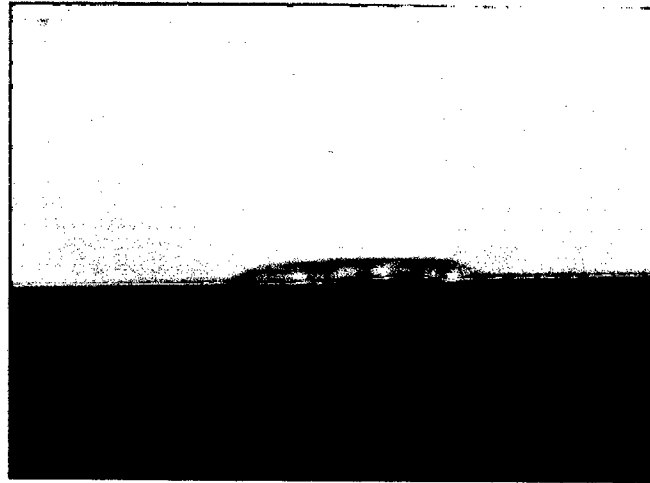


Figure 3.5: Solder joint between Sn-0.7Cu solder and copper substrate (side view)

3.3.4 Aging

Aging is a heat treatment process to improve the properties of the solder joint. The samples are placed in the furnace to perform aging. Aging is performed for various time and temperature. The temperature is set to be 100°C, 150°C and 200°C. Since the melting temperature for Sn-0.7Cu solder material is 227°C, the temperature for aging is set not higher than its melting temperature.

The time is ranging from 1 hour, 3 hours to 5 hours. There are 9 samples undergoing aging. For the first sample, aging is performed for 1 hour at 100°C and the second sample is performed for 1 hour at 150°C. The last sample for 1 hour aging is at 200°C. It is also same for 3 hours and 5 hours aging. Each sample for same aging time is performed at different aging temperature. This is to observe the growth of intermetallic compound layer when the time and aging temperature increase. The furnace used in this project is as in figure 3.6.

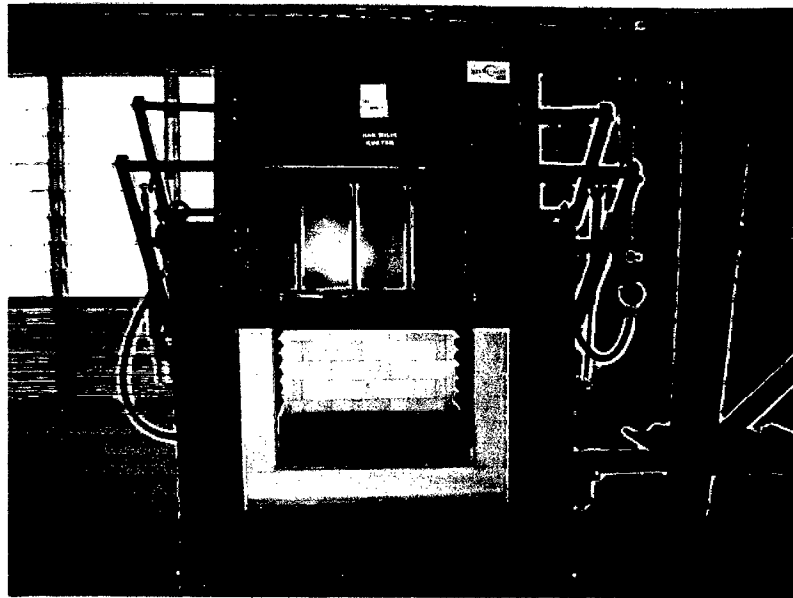


Figure 3.6: Furnace

3.3.5 Analysis

SEM is used to observe and analyze the microstructure of the samples in order to observe the growth of the intermetallic compound layer before and after aging at different time and temperature.

According to Jeong-Won Yoon and Seung-Boo Jung (2005), IMC layer grow simultaneously with aging time and the growth of IMC layer is much faster as the aging temperature increase. SEM can be seen in figure 3.7 as below.

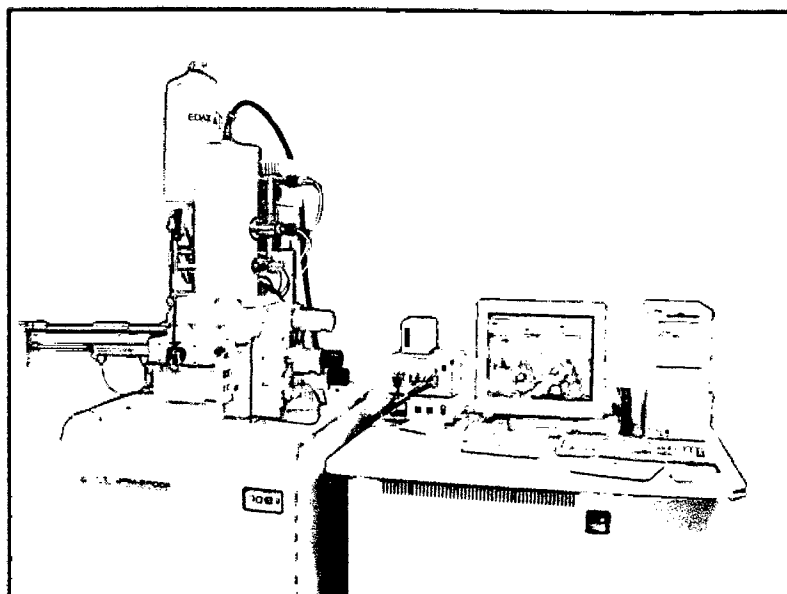


Figure 3.7: SEM (Scanning Electron Microscopy)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

Chapter 4 presents the result of this project based on the experiment conducted. The result is obtained from the investigation of the effects of aging time and temperature on the growth of IMC layer. From the experiment, aging was conducted on the samples with different time and temperature. Thus, the morphology change of the IMC growth is investigated.

SEM is used to analyze the presence of IMC layer between solder material which is Sn-0.7Cu and the copper plate. The samples were cut to perform SEM analysis. EDX is used to determine the elements exist on the microstructure. Five samples were analyzed which are the sample before aging, aging for 1 hour at 100°C, aging for 1 hour at 200°C, aging for 5 hours at 100°C and aging for 5 hours at 200°C.

4.2 Results

The SEM and EDX analysis is performed for specimens before and after aging. The results obtained show the different morphology of intermetallic compound.

4.2.1 Before Aging

As-soldered sample is observed using SEM. This is to investigate the effect of soldering on the presence of IMC layer.

4.2.1.1 As- Soldered Joint

The microstructure of 'before aging' sample can be seen in figure 4.1. The formation of uniform intermetallic compound layer is found at the interface of solder material and the base metal. The thickness for as-soldered sample is found to be $5.359 \mu\text{m}$.

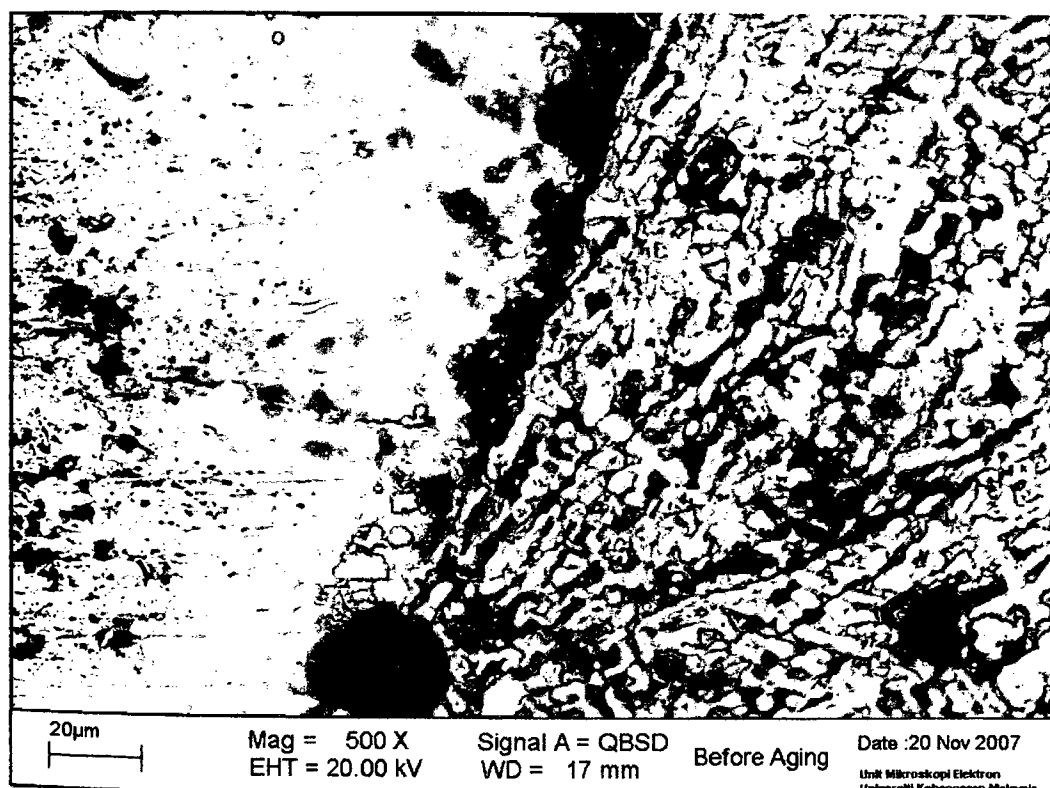


Figure 4.1: SEM micrograph of as-soldered joint

Base on the atomic percentage in EDX analysis as in table 4.1, the short supply of Sn from solder to the copper metal is the reason why Cu_6Sn_5 intermetallic compound is not found. Below are the results from EDX analysis.

Table 4.1: EDX element analysis of IMCs layer for as-soldered joint at interface

Element	App Conc.	Intensity Corrn.	Weight %	Weight % Sigma	Atomic %
C K	17.64	0.4932	30.81	1.29	66.99
O K	3.97	0.4819	7.10	0.77	11.58
Cl K	1.35	0.7512	1.55	0.16	1.14
Cu K	38.24	0.8912	36.97	0.98	15.20
Zn K	1.97	0.8949	1.90	0.45	0.76
Sn L	16.19	0.8196	17.02	0.61	3.75
Pb M	4.29	0.7946	4.65	0.59	0.59
Totals			100.00		

The graph below shows the presence of elements of IMCs layer for as-soldered joint at interface.

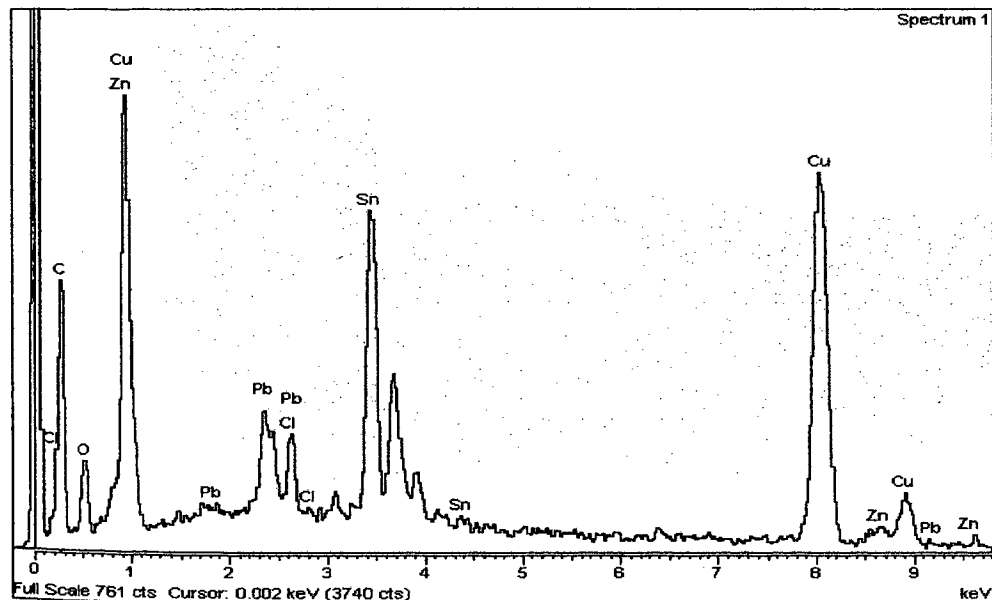


Figure 4.2: EDX element analysis of IMCs layer for as-soldered joint at interface

4.2.2 After Aging

After soldering, the samples were put in the furnace for certain time and temperature to perform aging. The morphology change of the IMC growth is investigated with different time and temperature.

4.2.2.1 Aging for 1 hour at 100°C

For the second sample, aging is performed for 1 hour at 100°C. The intermetallic compound layer can be seen in figure 4.3. The formation of uniform intermetallic compound layer is clearly seen between solder material and base metal. As the solder joint exposed to heat in aging process, the thickness of intermetallic compound layer is increase as well.

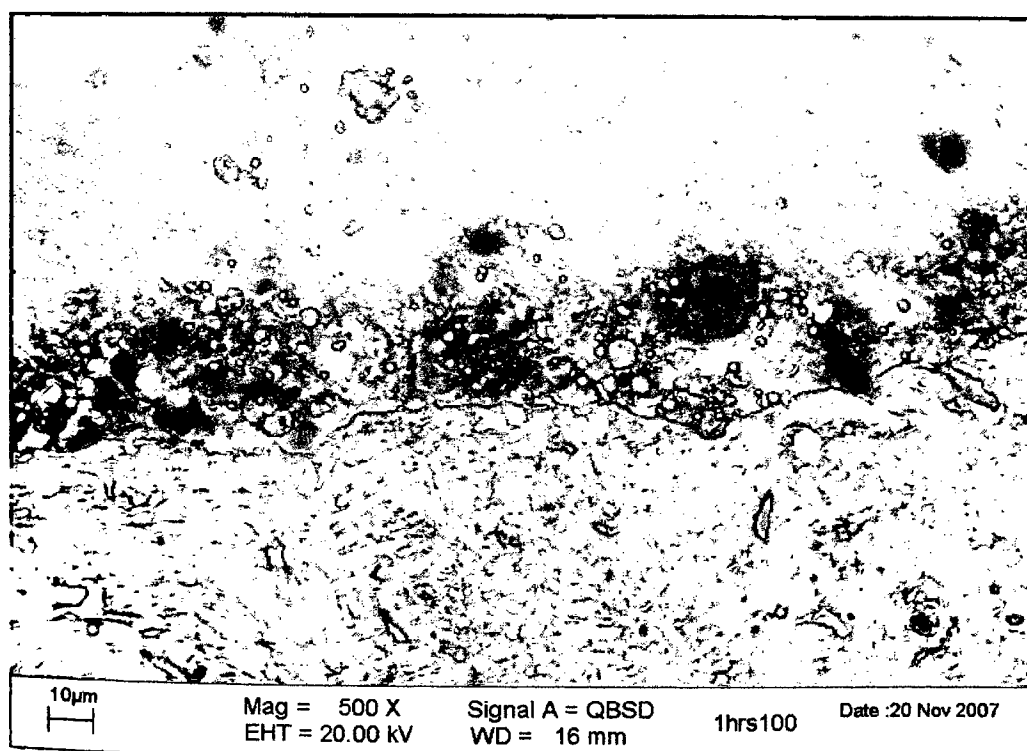


Figure 4.3: SEM micrograph of IMCs layer after aging for 1 hour at 100°C

From EDX analysis as in table 4.2, the atomic percentage for Sn and Cu shows that the intermetallic compound found is Cu_6Sn_5

Table 4.2: EDX element analysis of IMCs layer for aging for 1 hour at 100°C at interface

Element	App Conc.	Intensity Corrn.	Weight %	Weight % Sigma	Atomic %
C K	16.84	0.5410	32.49	1.58	62.21
O K	7.53	0.4999	15.73	1.20	22.61
Cl K	1.75	0.7365	2.48	0.25	1.61
Fe K	8.54	0.8984	9.92	0.57	4.09
Cu K	5.85	0.8558	7.14	0.66	2.58
Zn K	7.89	0.8608	9.57	0.87	3.37
Sn L	9.31	0.7900	12.31	0.70	2.38
Pb M	8.32	0.8386	10.36	0.90	1.15
Totals			100.00		

The graph below shows the presence of elements of IMCs layer for aging for 1 hour at 100° at interface.

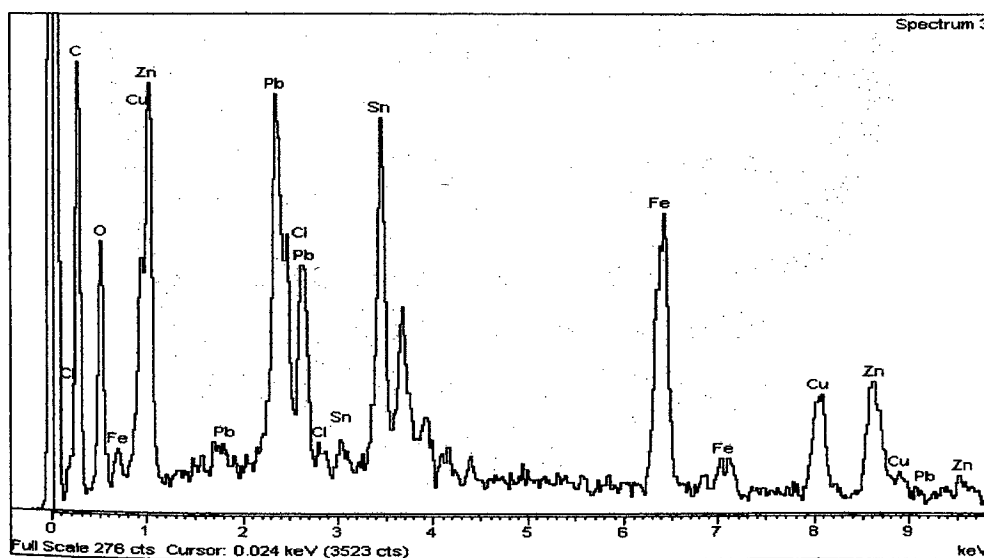


Figure 4.4: EDX analysis of IMCs layer after aging for 1 hour at 100°C

4.2.2.2 Aging for 5 hours at 100°C

Aging is performed for 5 hours at 100°C for the third sample. The SEM micrograph for the sample is seen in figure 4.5. At this stage intermetallic compound layer is clearer than the sample aging for 1 hour at 100°C. The continuous heat supply at the interface layer resulting faster growth of IMCs layer.

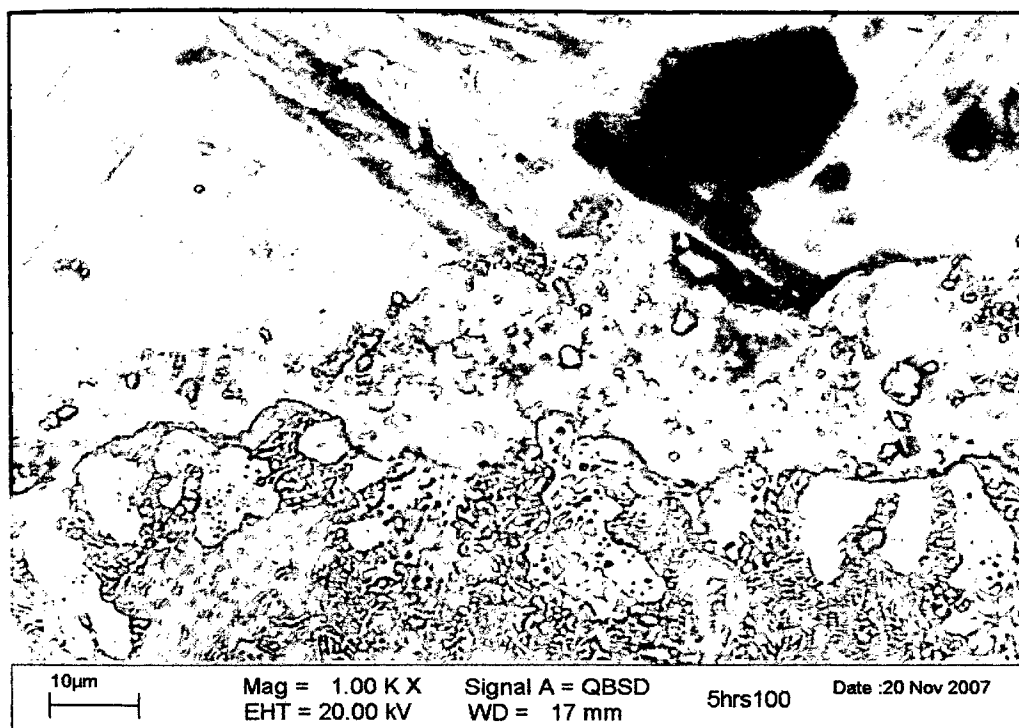


Figure 4.5: SEM micrograph of IMCs layer after aging for 5 hours at 100°C

The result from EDX analysis confirmed that the IMCs layer found is Cu_6Sn_5 . The atomic percentage of Sn and Cu elements can be seen in table 4.3 as below.

Table 4.3: EDX element analysis of IMCs layer for aging for 5 hours at 100°C at interface

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	15.62	0.5065	28.90	1.30	65.58
O K	4.47	0.4387	9.54	0.91	16.26
Cl K	4.04	0.7014	5.39	0.29	4.14
Cu K	11.85	0.9036	12.28	0.63	5.27
Zn K	3.54	0.9114	3.64	0.52	1.52
Sn L	16.07	0.7628	19.74	0.71	4.53
Pb M	19.14	0.8746	20.50	0.90	2.70
Totals			100.00		

The graph below shows the presence of elements of IMCs layer for aging for 5 hours at 100° at interface.

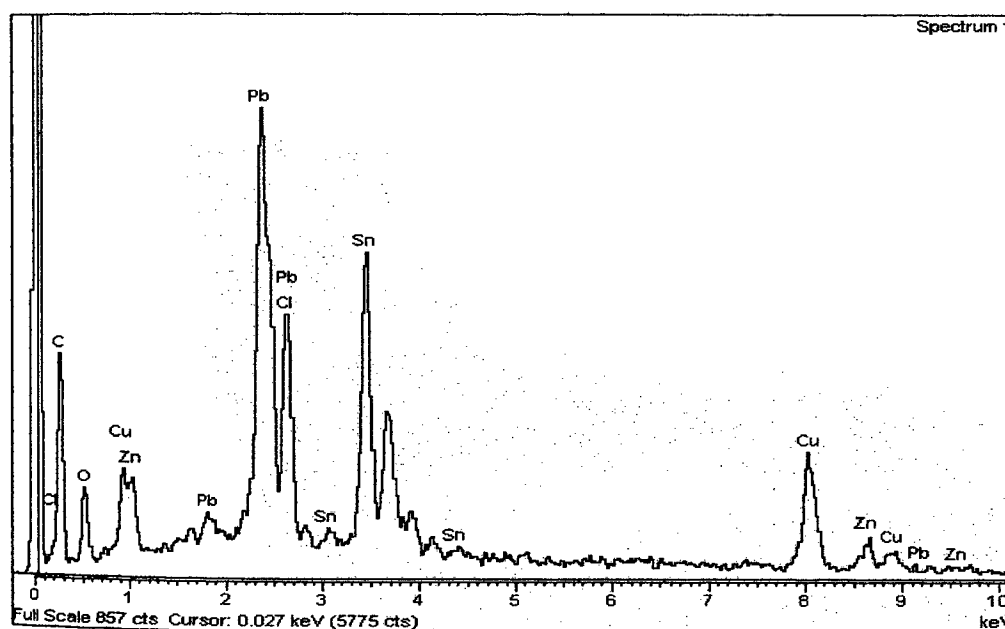


Figure 4.6: EDX analysis of IMCs layer after aging for 5 hours at 100°C

4.2.2.3 Aging for 1 hour at 200°C

Aging is performed for 1 hour at 200°C for the fourth sample. The SEM micrograph for the sample is seen in figure 4.7. At this stage intermetallic compound layer is in uniform layer. Since the melting point for Sn-Pb solder is 183°C, the solder liquidize in the furnace during soldering. This is the reason why the solder in the micrograph looks not really in solid shape.

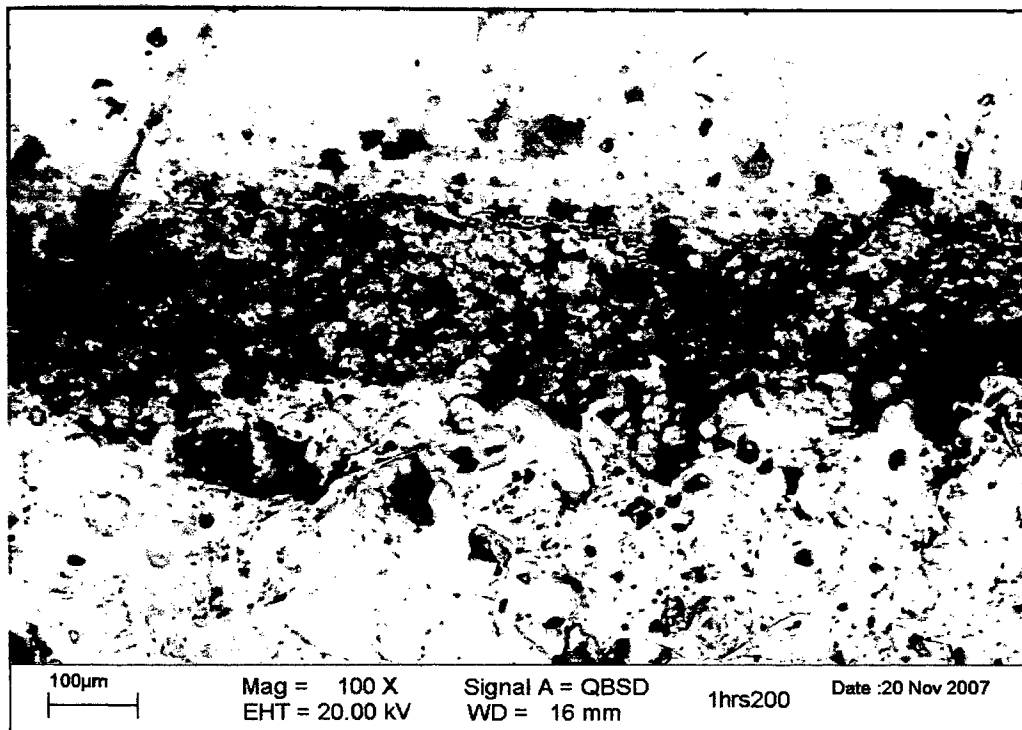


Figure 4.7: SEM micrograph of IMCs layer after aging for 1 hour at 200°C

Base on the EDX analysis for Sn and Cu elements, there are a little percentage of them at IMCs layer. Pb atom is slightly higher than Sn and Cu atoms in percentage. This is because the liquid solder affect the formation of IMCs layer at the interface. The table for EDX analysis is as below.

Table 4.4: EDX element analysis of IMCs layer for aging for 1 hour at 200°C at interface

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	18.06	0.4437	39.84	1.57	80.12
O K	1.53	0.4275	3.50	0.74	5.29
Na K	0.63	0.7473	0.82	0.23	0.86
Cl K	5.95	0.6394	9.11	0.46	6.20
Cu K	6.00	0.8934	6.57	0.61	2.50
Sn L	2.86	0.6944	4.04	0.45	0.82
Pb M	34.51	0.9349	36.13	1.26	4.21
Totals			100.00		

The graph below shows the presence of elements of IMCs layer for aging for 1 hour at 200° at interface.

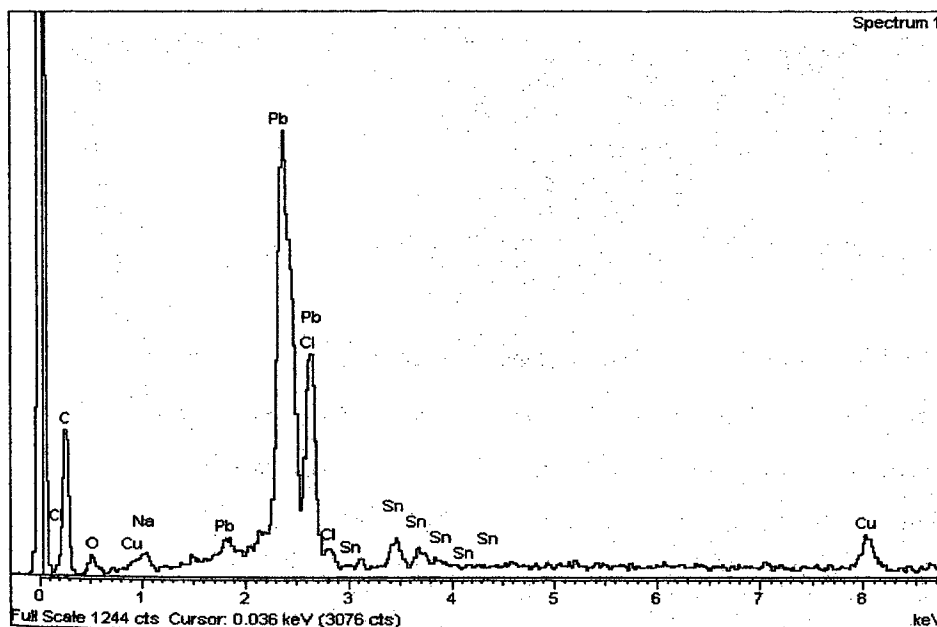


Figure 4.8: EDX analysis of IMCs layer after aging for 1 hour at 200°C

4.2.2.4 Aging for 5 hours at 200°C

Aging is performed for 5 hours at 200°C for the last sample. The SEM micrograph for the sample is seen in figure 4.9. Since the melting point for Sn-Pb solder is 183°C, the solder liquidize in the furnace during soldering. Thus, the formation of Cu_6Sn_5 intermetallic compound is not found.

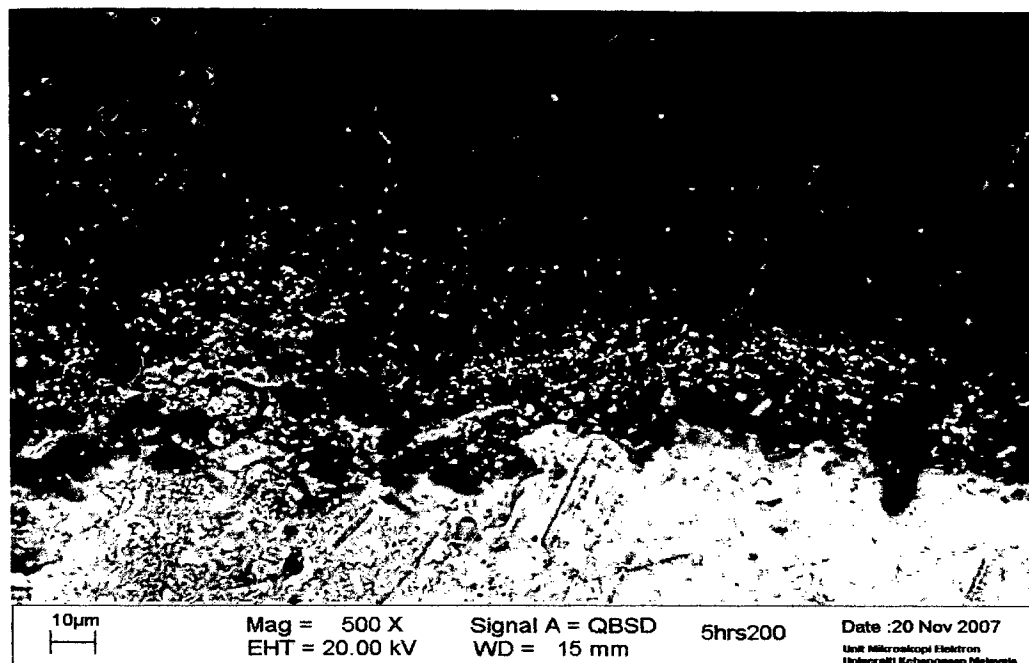


Figure 4.9: SEM micrograph of IMCs layer after aging for 5 hours at 200°C

Table 4.5 shows EDX analysis for sample aged for 5 hours at 200°C at IMCs layer. The percentages of Sn and Cu elements are higher than Pb. The high aging time and temperature causing the formation of IMCs layer faster than sample aged for 1 hour at 200°C. However, the Cu_6Sn_5 intermetallic compound is still not found.

Table 4.5: EDX element analysis of IMCs layer for aging for 5 hours at 200°C at interface

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	6.06	0.4119	16.76	1.69	53.40
O K	1.62	0.4607	4.01	0.84	9.60
Cl K	4.38	0.7097	7.03	0.39	7.59
Cu K	25.36	0.9511	30.37	1.10	18.29
Sn L	17.01	0.7847	24.70	0.95	7.96
Pb M	12.61	0.8387	17.13	1.05	3.16
Totals			100.00		

The graph below shows the presence of elements of IMCs layer for aging for 1 hour at 200° at interface.

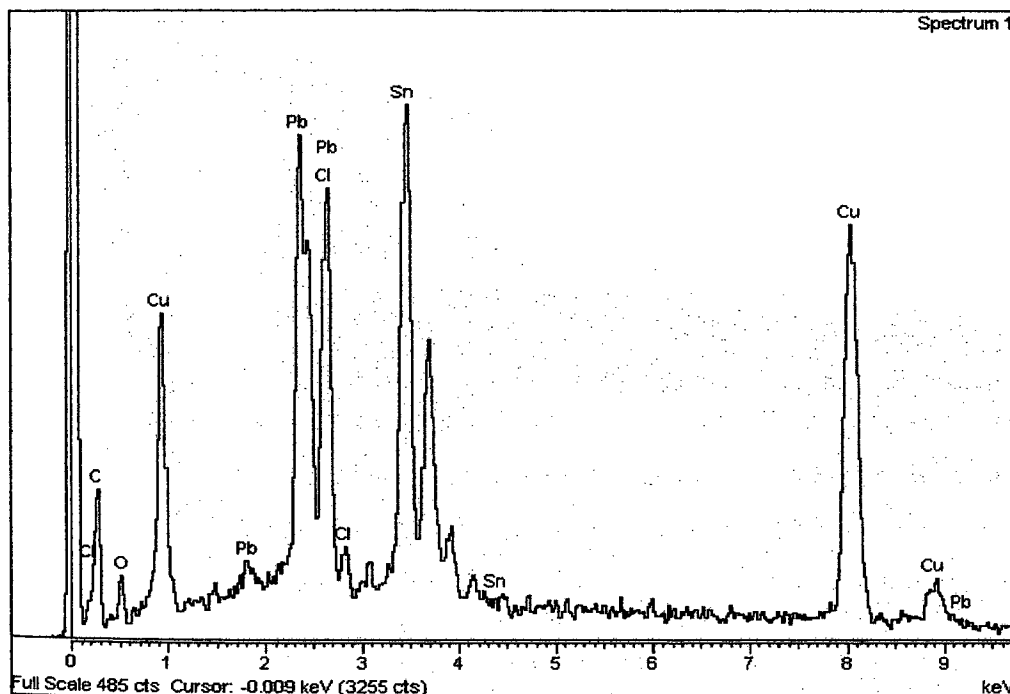


Figure 4.10: EDX analysis of IMCs layer after aging for 5 hours at 200°C

4.3 Discussion

The interfacial reaction between Sn-0.7Cu solder material and Cu base metal resulted in formation of Cu_6Sn_5 intermetallic compound layer according to (7). The thickness of the IMC layer is increased with increasing reaction time.

According to (6), the Cu_6Sn_5 IMC was identified in the as-soldered interface. The morphology change of the intermetallic compound is depending on the time and temperature of aging. Aging, which is a heat treatment process, is relying on time and temperature.

The changes also depend on the composition of the solder used and the substrate as well. Basically, the thickness of intermetallic compound gradually increases as the aging time increases. The growth of intermetallic compound is faster with higher aging temperature.

Basically, the formation of Cu_6Sn_5 IMCs layer is found at sample aged for 1 hour at 100°C and sample aged for 5 hours at 100°C . The formation of Cu_6Sn_5 IMCs layer is confirmed by EDX analysis. EDX analysis is to determine the composition and percentage of the elements at the microstructure. From the experiment, the thickness of IMCs layer is increased with increasing reaction time and temperature. The higher aging time and temperature, the growth of IMCs layer will become faster.

There are some errors that occur during the experiment that affect the results. Although the Cu metals were polished before soldering, there are some other elements existed on the microstructure instead of Sn and Cu elements. In addition, the condition during soldering also affects the formation of IMCs layer which will affect the integrity of the solder joint.

Other reasons that prohibit on getting desired results are:

- i. The working conditions:

Working place must be clean to ensure the cleanliness of the samples because solder can not work properly under dirty conditions.

- ii. **Soldering process:**
Soldering process must be done according to the rules to ensure good solderability. Bad solderability will defect joint integrity due to the natural weakness of the IMCs layer.
- iii. **Aging time and temperature:**
These two parameters must be set at the right value to obtain desired result and adequate IMCs layer.
- iv. **Polishing:**
Samples are required to be polished before proceed with SEM analysis. This is a must to look at the microstructure clearly. Polishing is to remove dirt and carbon that exist during aging.
- v. **Lack in expertise:**
This project is a new project proposed by this university. None of the staff; technicians and lecturers has an experience on this subject but only a vast knowledge in the subject. Many aspects likely are lack in connecting the theory and practical applications.
- vi. **Lack of equipment:**
As this university is still new and still in the stage of developing, many equipment is not available. The availability of the machine also reflects to the project progress.

CHAPTER 5

CONCLUSION

5.1 Introduction

This chapter presents the conclusion of the Final Year Project after conducted the experiment. Since tin-lead solder is a toxic and harmful to both environment and human health, lead-free solder is used instead of tin-lead solder in electronic industry. Recently, many researchers have found the promising lead-free solder to replace the conventional tin-lead solder. One of them is Sn-0.7Cu lead-free solder. Solderability is important to ensure good joint strength. However, lead-free solder may produce mechanically weaker joints depending on manufacture conditions.

From the previous journal and study on the interfacial reaction between solder material and metal substrate, it is found that the IMC layer growth is depend on the type and composition of solder material and the substrate.

5.2 Conclusion

A lot of information had been gathered from the research and the studies. Soldering between solder and the substrate may produce intermetallic compound at the interface. This intermetallic compound is needed to ensure strong bonding of the solder and the substrate.

However, the excessive amount of intermetallic compound will affect the solder joint and may lead to the failure of the joint due to its nature weakness and brittleness. The morphology change of the intermetallic compound is observed at different aging time and temperature. It is important to set the right aging and time temperature resulting adequate IMC layer to get the good solderability and to ensure the solder have good strength.

From the EDX analysis, the presence of Cu_6Sn_5 IMCs layer is found at the interface layer between solder and base metal. This result is same as reported in previous journals. The thickness of IMCs layer is increased with increasing of aging time and temperature. This result also same with the previous works. As a conclusion, the objective of this project is achieved due base on the research and the experiment.

5.3 Recommendation

Base on the project progress, some recommendations are stated below:

- i. This project should be improved in the future with improvement of equipments and machines at the lab.
- ii. Since the soldering process plays an important role in electronics industry worldwide, the continuous work on this project is required.
- iii. The experiment methodology has to be improved and conducted in detail to obtain desired result.
- iv. Instead of this project title, there is much other type of solders material and base metal to be investigated.

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