

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

**NABHAN BIN ISMIL**

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**Faculty of Mechanical Engineering  
University Malaysia Pahang**

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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  $\text{Cu}_6\text{Sn}_5$  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**

$\mu$	-	Micro
SEM	-	Scanning electron microscopy
EDX	-	Energy dispersive X-ray
IMC	-	Intermetalli compound
Sn	-	Stannum
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.

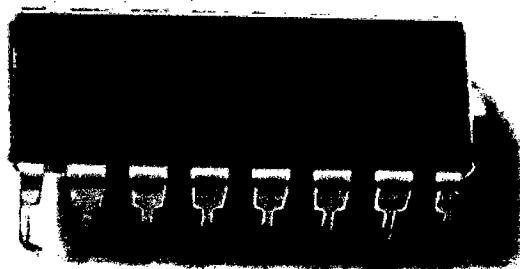
Joining technology or soldering process in electronics industry is a general scope in this project. Based on the project title, Sn-0.7Cu solder material is the main focus. However, this chapter also describe about tin-lead solder that has been used earlier than lead-free solder. Aging, which is a heat 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**

<b>Tin (wt. %)</b>	<b>Tensile Strength (MPa)</b>	<b>Shear Strength (MPa)</b>	<b>Elongation (%)</b>	<b>Elastic Modulus (GPa)</b>	<b>Izod Impact Strength (J)</b>	<b>Stress to produce 0.01%/day creep rate (kPa)</b>
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

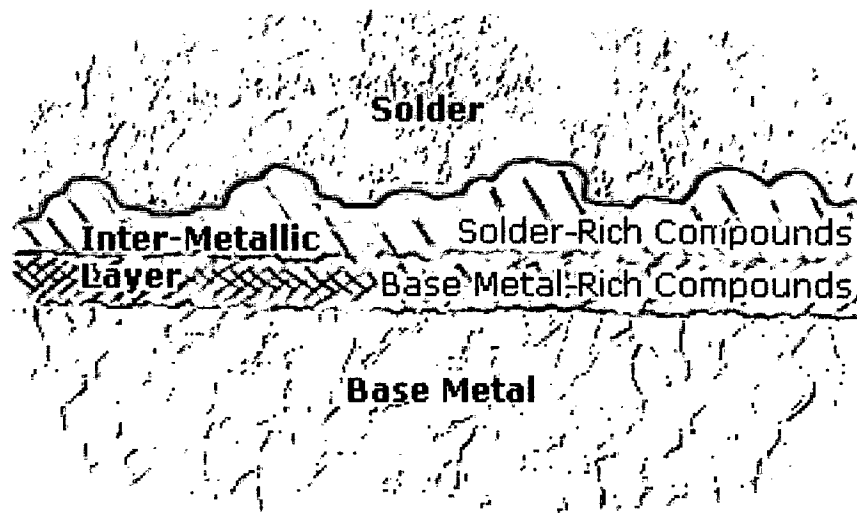
temperature for hours to allow precipitation to take place. This time delay is called aging.

## 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

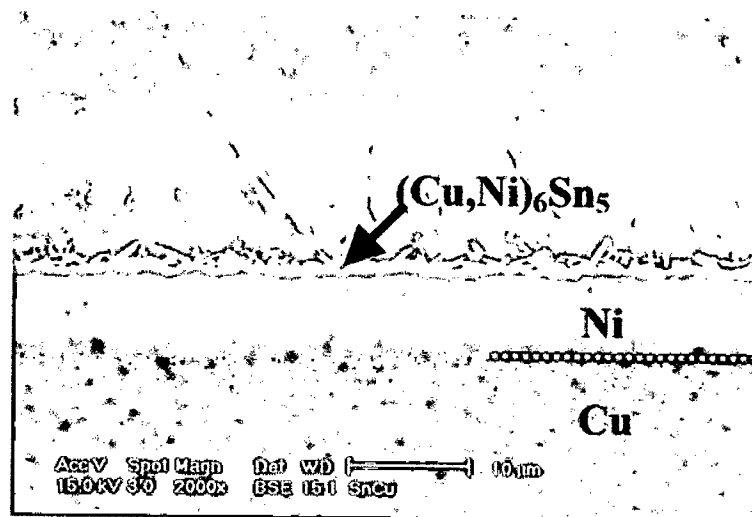
Within each inter-metallic layer, there are actually a number of different compounds formed by the solder materials and the base metal. These compounds are typically quite brittle and will adversely affect the integrity of the solder joint. As the joint is subject to stress, thermal cycles, vibration, 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.

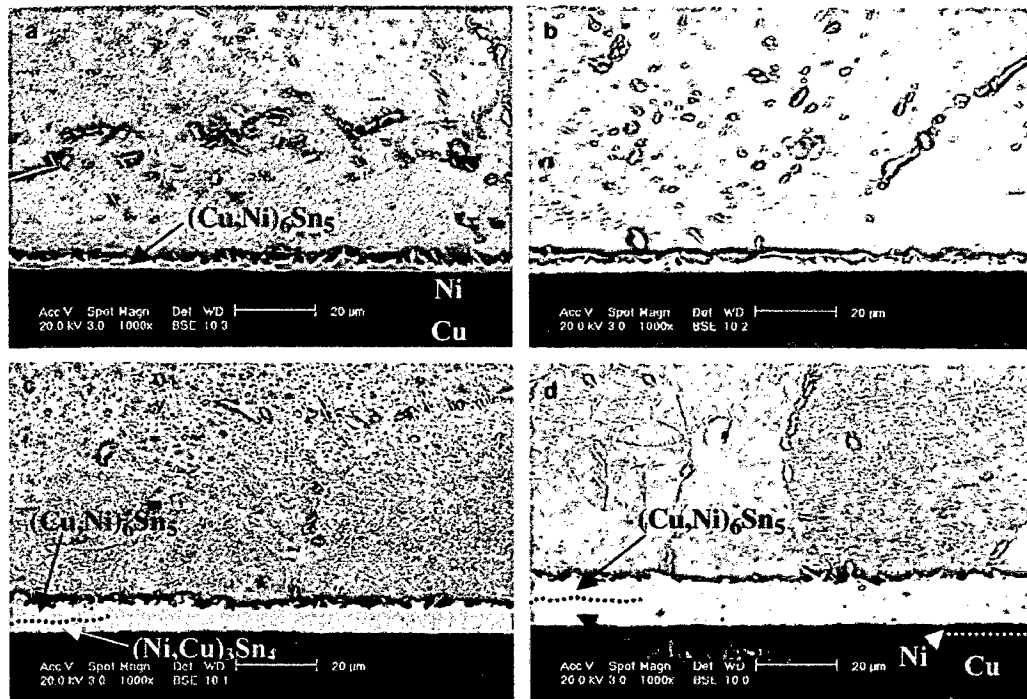
### 2.7.1 Interfacial Reaction and Mechanical Properties of Eutectic Sn-0.7Cu/Ni BGA Solder Joints during Isothermal Long-term Aging

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

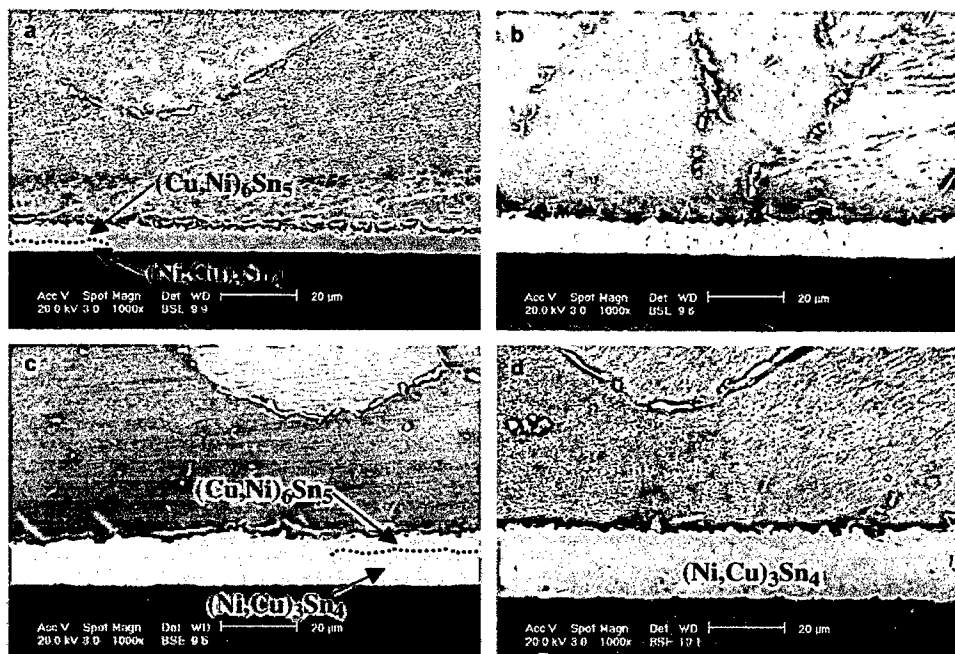
After aging at 185°C for 3 days, two intermetallic compound  $(\text{Cu, Ni})_6\text{Sn}_5$  and  $(\text{Ni, Cu})_3\text{Sn}_4$  were observed. These intermetallic compounds also growth at the interface after aging at 200°C for 1 day. Due to the restriction of supply of Cu atoms, the growth of  $(\text{Ni, Cu})_3\text{Sn}_4$  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  $\text{Cu}_3\text{Sn}$  intermetallic compound layer was formed underneath  $(\text{Ni}, \text{Cu})_3\text{Sn}_4$  intermetallic compound layer.