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**EFFECT OF ISOTHERMAL AGING TO THE
INTERMETALLIC COMPOUND (IMC) GROWTH OF Sn-
0.7Cu-1.0Si₃N₄ COMPOSITE SOLDER ON COPPER
SUBSTRATE**

by

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LIST OF ABBREVIATIONS

AE	Activation Energy
Al	Aluminium
Al ₂ O ₃	Aluminium oxide
Ag	Argentum
Bi	Bismuth
Cr	Chromium
Cu	Copper
EDS	Energy Dispersive Spectroscopy
IMC	Intermetallic Compound
RE	Rare Earth
SAC	Sn-Ag-Cu solder
SEM	Scanning Electron Microscope
SiO ₂	Silicon oxide
Si ₃ N ₄	Silicon nitride
Sn	Stannum (Tin)
TiO ₂	Titanium oxide
XRD	X-ray Diffraction
Zn	Zink

LIST OF SYMBOLS

α	Alpha Phase
β	Beta Phase
γ	Gamma Phase
kJ/mol	Kilo Joule per Mol
μm	Micron meter

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Effect of Isothermal Aging to the Intermetallic Compound (IMC) Growth of Sn-0.7Cu-1.0 wt.%Si₃N₄ Composite Solder on Copper Substrate

ABSTRACT

The excessive growth of intermetallic compound (IMC) layer on solder joints has become a challenging to the electronic packaging industry. Excessive IMC growth is a barrier for the reliability of solder joints and the suppression of IMC is a target for most solder manufacturers. Thus, research study of IMC formations and growth are always open for discussion and development. A new Sn-0.7Cu-Si₃N₄ composite solder has been fabricated in order to overcome the above mentioned problem. In this study, isothermal aging test on the new SnCu-Si₃N₄ composite solder were carried out at 50°C, 75°C, 100°C, 125°C and 150°C for 24, 240, 480, 600 and 720 hours. The same methodology was carried out to monolithic Sn-0.7Cu solder for comparison. SEM-EDS and XRD were used for elemental and compound analysis respectively. It is seen that Cu₃Sn and Cu₆Sn₅ IMCs have remained as the IMC phases in both solders. From observation, Si₃N₄ addition has not created any other phases after soldering process. After isothermal aging, the intermetallic thickness has increased with increasing of temperature and time while the morphology has changed from scallop to planar type. However, the IMC growth rate for composite solder is low compared to Sn-0.7Cu solder. The diffusion kinetics for Sn-0.7Cu-Si₃N₄ is lower than monolithic Sn-0.7Cu solder which had resulted with larger activation energy value. The activation energy value for the solder composite is 15.5kJ/mol while for Sn-0.7Cu solder is 14.7kJ/mol. For mechanical testing, shear test was carried out by using Instron machine. The overall shear strength of Sn-0.7Cu-Si₃N₄ is higher than Sn-0.7Cu solder. Therefore, the presence of silicon nitride as reinforcement is believed has suppressed IMC formation and has improved the strength of Sn-0.7Cu-Si₃N₄ composite solder.

Kesan Penuaan Iso-terma kepada Pertumbuhan Sebatian Antaralogam (IMC) Sn-0.7Cu-1.0 wt.%Si₃N₄ Pateri Komposit Pada Substrat Kuprum

ABSTRAK

Pertumbuhan secara agresif lapisan sebatian antaralogam (IMC) pada sambungan pateri telah menjadi cabaran besar dalam industri pembungkusan elektronik. Pertumbuhan IMC secara agresif menjadi penghalang kepada ketahanan sambungan pateri dan menyebabkan kebanyakan pembuat pateri menasarkannya untuk mengurangkan pertumbuhan agresif IMC tersebut. Oleh sebab itu, kajian penyelidikan mengenai pembentukan dan perkembangan IMC sentiasa terbuka untuk perbincangan dan pembaharuan. Pateri komposit baru Sn-0.7Cu-Si₃N₄ telah dibangunkan untuk mengatasi masalah di atas. Ujian penuaan iso-terma terhadap pateri komposit Sn-0.7Cu-Si₃N₄ pada 50°C, 75°C, 100°C, 125°C dan 150°C selama 24, 240, 480, 600 and 720 jam. Kaedah yang sama juga telah dijalankan kepada monolitik pateri Sn-0.7Cu untuk analisis perbandingan. SEM-EDS dan XRD telah digunakan bagi menganalisis unsur dan sebatian pateri komposit. Analisis mendapati, IMC, Cu₃Sn dan Cu₆Sn₅ telah kekal wujud untuk kedua-dua pateri. Bagi pateri komposit ia menunjukkan bahawa penambahan Si₃N₄ tidak mewujudkan fasa-fasa yang lain selepas proses pematerian. Selepas ujian iso-terma, ketebalan IMC telah meningkat dengan peningkatan suhu penuaan dan masa, manakala morfologi IMC berubah daripada bentuk kekapis kepada bentuk mendatar. Walaubagaimanapun, kadar pertumbuhan IMC bagi pateri komposit adalah lebih rendah berbanding pateri Sn-0.7Cu. Kinetik resapan untuk Sn-0.7Cu-Si₃N₄ adalah lebih rendah berbanding pateri Sn-0.7Cu yang menyebabkan nilai tenaga pengaktifannya menjadi lebih besar. Nilai tenaga pengaktifan bagi pateri komposit adalah 15.5kJ/mol manakala bagi pateri Sn-0.7Cu adalah 14.7kJ/mol. Bagi ujian mekanik, ujian ricih telah dijalankan dengan menggunakan mesin Instron. Kekuatan keseluruhan Sn-0.7Cu-Si₃N₄ adalah lebih tinggi daripada pateri Sn-0.7Cu. Oleh itu, kehadiran silikon nitrida sebagai tetulang dipercayai telah menghadkan pembentukan IMC dan telah meningkatkan kekuatan ricih Sn-0.7Cu-Si₃N₄ pateri komposit.

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Soldering is the metallurgical parts for the process of joining method that uses conventionally lead-tin solder as a filler metal. The solder joint plays a crucial role in interconnection and performs thermal, mechanical and electrical functions (Pang et al., 2001). Tin-lead eutectic solders have been widely used due to good properties such as mechanical, fatigue, creep properties, low melting point (Chellaih et al., 2007; Satyanarayan & Prabhu, 2011).

Formerly nearly all solders contained lead, but environmental concerns have increasingly dictated the restriction of lead solder alloys for electronics and plumbing purposes. The strict legislation to ban the using of lead in electronic applications because of environmental concerns has shown intense of development to replace lead containing solders in recent years (Haseeb & Leng, 2011; Satyanarayan & Prabhu, 2011). However, the performance and reliability of lead-free solders in industrial nowadays are not suitable for many applications (Haseeb & Leng, 2011).

There's a few most promising candidates for lead-free solder alloy that been used in electronic packaging industry which to produce high reliability and good performance of solder (J. Chen et al., 2011). National Electronics Manufacturing Initiative (NEMI) and Nortel Network recommended the Sn-0.7Cu as first priority and Sn-3.5Ag as second priority to replace Sn-Pb solder alloy for wave soldering

applications (Rizvi et al., 2007a; Satyanarayan & Prabhu, 2011). Furthermore, the Sn-0.7Cu is the cheapest lead-free solder, which have cost only 30% higher than conventional Sn-Pb solder (Rizvi et al., 2007a; Satyanarayan & Prabhu, 2011). However, Sn-0.7Cu has lower mechanical properties compared to the Sn-37Pb and other lead-free solder (Rizvi et al., 2007a; Satyanarayan & Prabhu, 2011).

During soldering and aging of the lead-free solder on Cu pads, interfacial transformation occurs at the solder/Cu interfaces and in the solder matrix that evolves Cu-Sn intermetallic compound known as Cu_3Sn and Cu_6Sn_5 intermetallic phase (Rizvi et al., 2007a; Satyanarayan & Prabhu, 2011). This intermetallic layer is good for wetting, but growth of excessive intermetallic layer weakens the strength of solder joints due to brittleness and weakness (Rizvi et al., 2007a; Yoon & Jung, 2006). Besides that, it is important to understand and control the factors of growth rate of the intermetallic compound lead-free solders (Yoon & Jung, 2006).

The phenomenon of excessive IMC growth of lead-free solder has triggered the idea to add reinforcement to eutectic solder to become composite solder. Generally, the addition either metal or ceramic as reinforcement in the lead-free solders are functions as diffusion barrier layer to retard the rapid interfacial reaction between solder and Cu substrate and improve the wettability and mechanical properties of solder (Haseeb & Leng, 2011; M. A. A. Mohd Salleh et al., 2011; Rizvi et al., 2007a). According to Tsao, (2011), addition non-reacting and non-coarsening reinforcement in solder material reduced the average size and spacing of intermetallic particles. The refined sized and spacing of intermetallic particles will slower the growth rate of IMC at interface between solder and substrate (Tsao, 2011). The composites solder means the solder materials composed two or more different elements which include metals and non-metallic materials.

Accordingly, composite solder have good creep and thermo-mechanical fatigue resistance in solder joints (Shen & Chan, 2009). The development of lead-free composite solder in electronic industries which is to replace lead solder has been studied. In this research, the usage of 1.0% micron-sized silicon nitride (Si_3N_4) reinforcement in Sn-0.7Cu base solder is a new material development.

1.2 Problem Statement

Industrial of electronic packaging have moved forward to smaller solder joints and fine-pitch interconnection. The ultrafine solder joint packaged in narrow space will lead to a high homologous temperature during service. This phenomenon may lead to IMC overgrowth and coarsening microstructure. The formation and the subsequent growth of the intermetallic is a major issue in soldering. The excessive growth formation of intermetallic compound will reduce the reliability of electronic devices and detrimentally affects its mechanical properties such as reduce the shear strength of solder joints.

Normal operating temperature of electronic devices is in between 50°C to 80°C and maximum operating temperature is about 125°C and thus it is important to understand the performance of the solder material at the opening procedure especially in understanding the intermetallic growth of solder.

The study of interfacial intermetallic growth in isothermal aging is very important since aging process mimics the real application of solder materials in electronic packaging. With a newly development Sn-0.7Cu- Si_3N_4 composite solder, it is important to understand its intermetallic growth after isothermal aged and its effect to the solder joint performance.

1.3 Objective

The objectives of this research in detail consist of :

1. To synthesis a novel composite solder material by using Sn-0.7Cu solder system with the addition of 1.0 in weight percent (wt.%) Si_3N_4 .
2. To study and analyze the growth kinetic of intermetallic (IMC) formation in Sn-0.7Cu- Si_3N_4 composite solder on Cu substrate after isothermal aging.
3. To study and analyze the effect of thermal aging process to the shear strength of Sn-0.7Cu- Si_3N_4 composite solder on Cu substrate.

1.4 Scope of study

In recent years, a lot of studies have indicates the properties and intermetallic formation of various composite solders. In this research work, the new developments of composite solder have been studied by using Sn-0.7Cu solder with silicon nitride (Si_3N_4) as the reinforcement. The solder will be fabricated by using powder metallurgy method. Sn-0.7Cu- Si_3N_4 have been reflowed on printed circuit board (PCB) and followed by aging process at various aging temperature and time. All samples will undergo the standard metallographic process for microstructural observation and growth rate analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Soldering

Soldering is a process which involves the bonding mechanism or interconnection between metal substrate and solders alloy by using reflows or wave process. This interconnection will acquire the formation interfacial intermetallic compound (IMC) between solder and metal substrate. Soldering also differs from brazing since the filler metal for brazing melts at a higher temperature; more than 450°C, but the metal substrate does not melt.

Interconnections of solder and substrate will perform in several major functions which consists of electrical connection, mechanical support and for thermal management purposes. For electrical, they serve the path of electrical connection from first level interconnection to the second level interconnection in electronic circuit. Meanwhile, they also provided the mechanical support for various electronic chips which are connected. Therefore, when the power in electronic chips increases, the intermetallic of solder on chips also serves the functions as heat dissipation. The successful functioning of electronic devices depends on reliable intermetallic formations provided by tiny IMC formation and lower IMC growth kinetics with under various conditions (Shangguan, 2005).

2.1.1 Reflow soldering

Reflow soldering process for joining solder alloy to the substrate mostly using convection reflow ovens which capable achieving higher temperature and suitable for lead-free soldering process (Jasbir Bath, 2007). Jasbir (2007) also mention that the accurate reflow profile for lead-free soldering is very important due to precision soldering process. The reflow profile for Sn-0.7Cu solder can be seen in Figure 2.1.

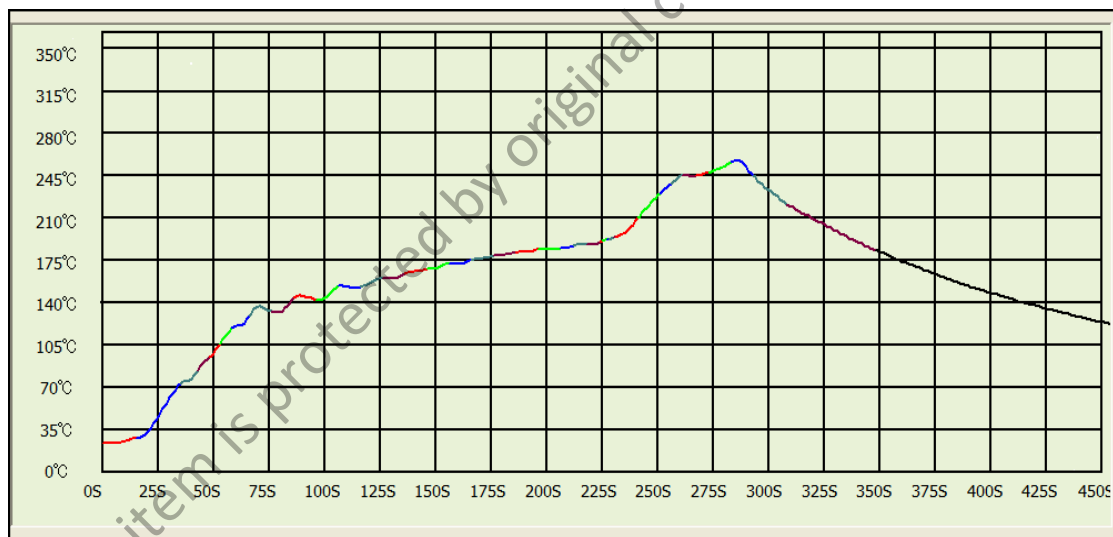


Figure 2.1: Reflow profile for Sn-0.7Cu solder provided by Qualitek

According to Shangguan (2005), a peak temperature in reflow profile is the most important key parameter due to the solder interacts with substrate during melting, wetting and flows.

2.2 Solder alloys

Solders are defined as fusible metal alloys with melting points below 450°C used joint metallic parts. The earliest solders were based on alloys of tin while the term

of solder is adaption from Old French; soudure which means to make solid (Humpston & Jacobson). Since 2001 as shown in Table 2.1, other researchers use various type of solder in their study to observe and analyze the solder properties. Recently, Sn-Ag, Sn-Ag-Cu, Sn-Zn and Sn-Cu is the common solder used in electronic packaging industrial which to replace Sn-Pb solder, but none of them achieved better properties than lead solders (M. A. A. et al., 2011).

According to the restriction of Sn-Pb solder in electronic packaging industry, the study of this solder material has become unpopular since 2010 where researchers changed to lead-free solder. From Table 2.1, the types of solder and synthesis method studied by other researchers showed that, the solder composite Sn-0.7Cu is not fully investigated by other researchers. It shows that other researchers are mostly interested to investigate Sn-Ag-Cu solder compared to Sn-0.7Cu composite solder.

2.2.1 Sn-0.7Cu lead-free solder

Sn-0.7Cu eutectic solder is used as alternative to replace the Sn-Pb solder because of cheapest solder among all other lead-free solders and the cost is only 30% higher than Sn-Pb solder (Satyanarayan & Prabhu, 2011; Tsao, 2011). The Sn-0.7Cu solder was used in wave soldering and considered in high temperature application such as in automotive industry. The Sn-Cu binary alloy has a eutectic composition of Sn-0.7Cu and eutectic temperature of 227°C (Satyanarayan & Prabhu, 2011) as shown in Figure 2.2.

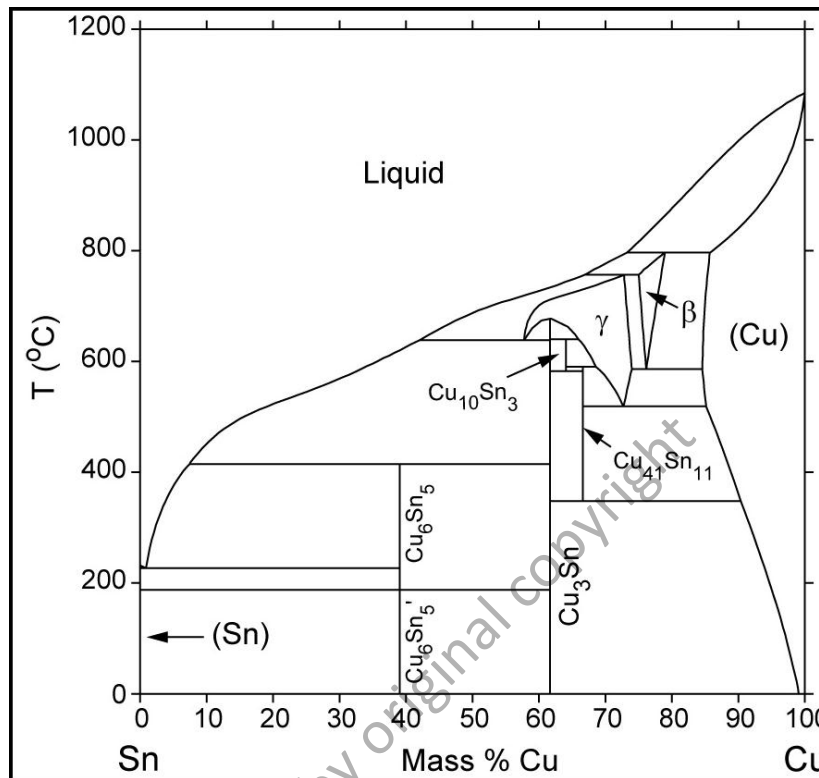


Figure 2.2: Phase diagram of Cu-Sn binary

Source: (Humpston & Jacobson)

The solid solubility of Cu in tin at eutectic temperature is only 0.006 wt.% to 0.01 wt.%. The solidification reaction will then form hollow rod of IMC Cu_6Sn_5 which precipitated in tin rich grains (Satyanarayan & Prabhu, 2011). From the phase diagram as in Figure 2.2, the stable intermetallic phases are below 300°C with existence of large Sn grains and a fine dispersion of Cu_6Sn_5 intermetallics (Satyanarayan & Prabhu, 2011).

According to El-Daly et al., the Sn-0.7Cu eutectic solder has good advantages due to low cost, blockage massive dissolution of Cu atoms on copper substrate especially during soldering process and most promising candidate to replace Sn-Pb solder (El-Daly & Hammad, 2011). According to the Table 2.1, the developments of Sn-0.7Cu solder from other researchers are very little compared to another solder. The most promising used solder by other researcher are Sn-Ag and Sn-Zn solder. The Table