

DEVELOPMENT OF SiC REINFORCED  
SnCu AND SAC BASED LEAD-FREE SOLDER  
COMPOSITE VIA POWDER METALLURGY ROUTE

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SnCu AND SAC BASED LEAD-FREE  
SOLDER COMPOSITE VIA POWDER  
METALLURGY ROUTE**

by

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## APPROVAL AND DECLARATION SHEET

This thesis titled Development of SiC Reinforced SnCu and SAC Based Lead-Free Solder Composite Via Powder Metallurgy Route was prepared and submitted by Zawawi Bin Mahim (matrix number: 1330410988) and has been found satisfactory in terms of scope, quality and presentation as partial fulfilment of the requirement for the award of degree of Master of Science (Materials Engineering) in University Malaysia Perlis (UniMAP)

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## LIST OF ABBREVIATIONS

Al <sub>2</sub> O <sub>3</sub>	Alumina
Bi	Bismuth
Cd	Cadmium
CTE	Coefficient of Thermal Expansion
EDX/EDS	Energy Dispersive X-ray
EU	European Union
Au	Gold
HCl	Hydrochloric acid
IC	Integrated Circuit
IMC	Intermetallic Compound
In	Indium
NEMI	National Electronics Manufacturing Initiative
Ni	Nickel
N	Nitrogen
HNO <sub>3</sub>	Nitric acid
OM	Optical Microscope
Pb	Lead
PCB	Printed Circuit Board
PM	Powder Metallurgy
RoHS	Restriction of the Use of certain Hazardous Substances in Electrical and Electronic Equipment
Ag	Silver
SAC	Sn-Ag-Cu
SMT	Surface Mount Technology
TD	Theoretical Density
TiB <sub>2</sub>	Titanium dibromide
WEEE	Waste Electrical and Electronic Equipment
XRD	X-ray Diffraction
ZrO <sub>2</sub>	Zirconia

## LIST OF SYMBOLS

%	Percent
°C	Degree Celsius
°C/min	Degree Celsius per minute
°C/s	Degree Celsius per second
$\mu\Omega.m$	Micro ohm meter
A	Area
$g/cm^3$	Gram per centimeter cubic
GPa	Giga Pascal
HV	Hardness Vickers
L	Length from contact to contact
MPa	Mega Pascal
N	Newton
R	Resistance
rpm	Rotation per minute
s	Second
V	Voltage
wt%	Weight percent
$\theta$	Angle
$\rho$	Density
$v$	Volume fraction

# PEMBANGUNAN KOMPOSIT PATERI BEBAS-PLUMBUM BERASASKAN SnCu & SAC DIPERKUAT SiC MELALUI KAEDAH METALURGI SERBUK

## ABSTRAK

Pada masa kini, pateri komposit menarik perhatian para penyelidik disebabkan oleh peningkatan yang memberangsangkan dalam sifat-sifat fizikal dan mekanikal untuk pateri bebas plumbum. Kajian ini dijalankan untuk mengkaji kesan partikel SiC terhadap perubahan mikrostruktur, sifat-sifat fizikal dan mekanikal berasaskan pateri aloi SAC dan SnCu. Kedua-dua pateri SAC dan SnCu komposit telah disintesis melalui kaedah metalurgi serbuk (PM), yang terdiri daripada beberapa proses seperti pengadunan mekanikal, pemadatan dan pensinteran. Sebanyak lima peratusan berat partikel SiC yang berbeza; 0.00, 0.25, 0.50, 0.75 dan 1.00 telah dicampur secara mekanikal ke dalam SnCu dan SAC pateri bebas-plumbum. Terdapat dua fasa yang terlibat. Fasa pertama adalah siasatan terhadap ciri-ciri pateri komposit dan fasa kedua adalah siasatan terhadap pateri komposit sebagai pateri penghubung. Keputusan kebolehasahan menunjukkan sudut sentuh yang paling kecil untuk SnCu-SiC adalah pada 18.60 sebanyak 0.75 wt.% SiC. Manakala sudut sentuh terkecil bagi SAC-SiC pula ialah 20.00 darjah sebanyak 1.00 wt.% SiC. Nilai ketumpatan bagi komposit SnCu-SiC dan SAC-SiC adalah  $7.162 \text{ g/cm}^3$  dan  $7.274 \text{ g/cm}^3$  apabila jumlah SiC yang sama iaitu sebanyak 0.75 wt.% ditambah ke dalam pateri masing-masing. Kekerasan bagi SnCu-SiC mencapai nilai maksimum 13.0 HV pada jumlah SiC sebanyak 0.75 wt.% manakala SAC-SiC mencapai nilai maksimumnya 18.6 HV apabila sebanyak 1.00 wt.% SiC ditambah. Nilai maksimum kekuatan ricih bagi SnCu-SiC ialah 14.95 MPa dengan penambahan sebanyak 0.50 wt.% SiC. Bagi SAC-SiC pula, nilai maksimum kekuatan ricihnya telah mencapai 17.79 MPa dengan penambahan sebanyak 0.50 wt.% SiC. Selepas itu, nilai kekuatan ricih merosot apabila melebihi 0.75 wt.% SiC. Kerintangan elektrik ( $\rho$ ) bagi SnCu-SiC dan SAC-SiC pateri bebas-plumbum komposit dikaji menggunakan mesin siasatan empat-titik. Keputusan menunjukkan nilai kerintangan hanya sedikit terjejas dengan penambahan zarah SiC di dalam SnCu-SiC dan SAC-SiC komposit pateri bebas-plumbum. Nilai kerintangan elektrik bagi SnCu-SiC dan SAC-SiC pateri komposit menurun apabila partikel SiC sebanyak 0.75 wt. % dan 1.00 wt. % ditambah yang mana nilainya masing-masing adalah  $1.18 \mu\Omega\text{.cm}$ ,  $1.12 \mu\Omega\text{.cm}$ ,  $0.88 \mu\Omega\text{.cm}$  dan  $0.81 \mu\Omega\text{.cm}$ . Secara keseluruhannya, keputusan yang dinyatakan di dalam kajian ini menunjukkan bahawa penambahan zarah SiC ke dalam SnCu dan SAC pateri bebas-plumbum telah meningkatkan sifat-sifat fizikal dan mekanikal.

# DEVELOPMENT OF SiC REINFORCED SnCu & SAC BASED LEAD-FREE SOLDER COMPOSITE VIA POWDER METALLURGY ROUTE

## ABSTRACT

Nowadays, composite solder has gain researcher's attention due to its promising improvement in physical and mechanical properties for lead free solder. This study was carried out to investigate the effect of SiC particle on microstructure evolution, physical and mechanical properties of SAC and SnCu based solder alloys. Both SAC and SnCu composite solders were synthesized by powder metallurgy method (PM), which consists of several processes such as mechanical blending, compaction and sintering. Five different weight percentages of SiC particles; 0.00, 0.25, 0.50, 0.75 and 1.00 were mechanically blended with SAC and SnCu lead-free solder. There were two phases involved. The first phase was investigation on the bulk composite solder properties and the second phase was investigation on the as-soldered composite solder connection. The wettability result showed the smallest contact angle for SnCu-SiC which is 18.60 degrees with 0.75 wt.% of SiC. While the smallest contact angle for SAC-SiC which is 20.00 degrees with 1.00 wt.% SiC. The optimum density value for SnCu-SiC and SAC-SiC were 7.162 g/cm<sup>3</sup> and 7.274 g/cm<sup>3</sup> respectively when 0.75 wt.% of SiC. The hardness values for SnCu-SiC samples was 13.0 HV starting at 0.75 wt.% to 1.00 wt.% of SiC addition while SAC-SiC reach the maximum value 18.6 HV at 1.00 wt.% of SiC addition. The shear strength for SnCu-SiC reach 14.95 MPa as an optimum value with addition 0.50 wt.% of SiC. After that, the value decreased until the addition 1.00 wt.% of SiC. The optimum value of SAC-SiC shear strength, 17.79 MPa was reached with the addition 0.50 wt.% of SiC. After that the value was decreased when the SiC addition has exceeded 0.75 wt.%. The electrical resistivity ( $\rho$ ) of bulk SnCu-SiC and SAC-SiC composite lead-free solders was measured with a four-point probe. The result showed there only slightly affected by the addition of SiC for SnCu-SiC and SAC-SiC composite lead-free solders. The value of electrical resistivity for SnCu-SiC and SAC-SiC composite solder are decreasing when 0.75 wt. % and 1.00 wt. % of SiC particles were added which is their value is 1.18  $\mu\Omega$ .cm, 1.12  $\mu\Omega$ .cm, 0.88  $\mu\Omega$ .cm and 0.81  $\mu\Omega$ .cm respectively. As overall, the result detailed in this work indicate that the addition of SiC particles into SnCu and SAC based lead-free solder able to enhance their physical and mechanical properties.

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Research

The oldest metallurgical technology used to join two or more metal parts is metal soldering. This technique is widely applied in electronic packaging industry until nowadays. It is heated to its melting point and used to join other metals and having a melting point below that of the work piece (Kim et al., 2009). Solder as a joining material, it is tasked with provides electrical, thermal and mechanical continuity in electronics assemblies. The important things for the solder are its performance and quality in a solder joint which is essential in determining the functionality and life period of the assembly (Peng et al., 2011).

Lead-containing (Pb-containing) chemicals have been recognized as poisons for a long time. However, metallic lead and its alloys have been used widely around the world for generations without much concern. Due to recent discovery that small amount of lead in human body can cause severe medical issues, health concern about the use of metallic lead and its alloy in soldering began to circulate (Manko, 2001). For the health and environment concern, which is inherently toxic, have led to the banning of Pb usage in electronic manufacturing by US, Japan and countries in the European Union. These

leads to the development of Pb-free solder and increase in research activities of this field of study (Peng & Shen et al., 2011).

It has long been established that tin-based solders, including SnAg, SnAgCu, and Sn-0.7Cu, were the most important solders for electronic applications in industries (Laurila et al., 2005). Many researchers still study the selection for Pb-free alternatives. SAC have attracted considerable attention as leading alloy Pb-free solder because of its greater mechanical properties (Bath, Handwerker & Bradley, 2000). Meanwhile, another solder alloy Pb-free that widely used in industry is SnCu. One of the main attraction for this solder alloy have been choose is cost. For both alloy solder, the minor addition of ceramic element to develop composite lead-free solder were studied by researchers in this field. The purposes to this study is to improve the properties of the both composite lead-free solder.

## **1.2 Problem Statement**

Lead-free solder has been subject of intense research activity over the past decade, but it is hard to meet the characterization and mechanical properties of Sn-Pb solder. Sn-Ag-Cu and Sn-Cu family of alloys were the most preferred and promising among all the Pb-free solder alloys proposed. Much publicity had been received by the eutectic Sn-3.0Ag-0.5Cu and Sn-0.7Cu solidifying their position as the most promising successor to Pb alloys solder. The previous researchers have introduced two common fabrication techniques for lead-free solder development which is; powder metallurgy (PM) method and casting method. There was problem occur to fabricate the composite solder by casting method due its needs to deal with small size of non-metal reinforcement powder and temperature controlling issues. PM method to fabricate the

composite solder lower cost and energy efficient compare to casting method (Mohd Salleh et al., 2013).

Generally, it was acknowledging that Sn-0.7Cu solder have problem to wet through hole properly due to poor wettability (Rizvi et al., 2006). To obtain excellent bonding between substrate and solder, the formation of interfacial intermetallic layer is needed. The brittleness and excessive thickness of the Sn-0.7Cu IMC layer were causes deteriorate of the mechanical and electrical properties of the joint. Sn-3.0Ag-0.5Cu solder alloy was chosen due to their melting point which is relatively low and closer to Sn-Pb. However, formation of large  $Ag_3Sn$  IMC within the bulk solder matrix due to the high amount of Ag is known to easily initiate cracks.  $Ag_3Sn$  IMC which is brittle in nature can cause degradation in term of hardness performance and commonly will causes failures in portable electronic devices that used the solder (Shnawah et al., 2012).

In this research, SiC particles were chosen as reinforcement for SnCu and SAC solders due to its light weight, and ease of handling. The additional of SiC particles is believed be able to improve the microstructural, physical, electrical, mechanical properties, solder joint and IMC formation issues. The amount of SiC particles play a role in affecting the properties in composite solder. In this study, the diversity of SiC particles as reinforcement to the SnCu and SAC based solder were collected and compared.

### **1.3 Objectives**

The objectives for this research are:

- i. To characterize and compare the effect of SiC reinforcement particles on melting temperature, electrical, mechanical properties and density of SnCu and SAC lead-free composite solder.

- ii. To investigate the effect of SiC reinforcement particles on the microstructure, intermetallic compound (IMC) formation and wettability of lead-free composite solder joint.

#### **1.4 Scope of Study**

SnCu and SAC are two based solder that were used in this study. The reinforcement for both is SiC with the same percentage; 0.25 wt.%, 0.50 wt.%, 0.75 wt.% and 1.00 wt.%. This composite solder was prepared by using powder metallurgy technique. The purposes of this study are to investigate mechanical properties and physical properties of the produced samples. Furthermore, to determine the other characteristic for both composite solder, 4-point probe, XRD, DSC and SEM also were tested to the samples. Wettability for comparison can be observe by using SEM.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Metallurgical Joining Technique

Soldering can be technically defining as a metallurgical joining technique in which uses a filler metal with melting point below 315 °C as the solder. Soldering requires neither diffusion nor intermetallic compound growth with the base metal achieve bonding but the wettability of the solder material for the bonding formation. Metallurgical bonds contain bond between metal and metal only. In general, soldering is a technique to bond metal to metal in which ensure the presence of metallic continuity between the two metals.

The other method metallurgical joining methods are brazing, welding, and some of the more recent developments that are combination of these, for example, diffusion bonding and thermal-compression bonding. The limit of 315 °C was set arbitrarily, and many people consider 425 °C the cut off point for soldering. Manko (2001) prefer to place solder alloys in the 315 °C – 425 °C in a subgroup of “hard solders” while the lower-temperature alloys would be “soft solders”. In comparison with soldering, brazing is defined as follows. Brazing is a metallurgical joining method using a filler metal that melt over 425 °C and that relies on wetting as well as diffusion for the bond

strength. Under the same conditions, welding could be defined as follows. Welding is a metallurgical joining process that relies on the diffusion of the base metals with or without the filler metal for the joining formation.

## **2.2 Solders as Interconnect Materials**

Since many decades ago until now, solder was act as interconnect between electronic component and printed circuit board (PCB). The process of joining the metal parts using the molten solder materials is called soldering process. Electronic packaging can be basically referred to the structures in which an integrated circuit (IC) chip is packaged in a way that it can efficiently connect with the larger environment and run as one system in an electronic device. Electronic packaging can also have called as integrated circuit packaging which is, microelectronic packaging relates to the development of smaller electronic designs and components in an IC package. In the manufacturing process of electronic products, there are three “Packaging Levels” that involve solder interconnect materials (Greig, 2007).

Figure 2.1 shows the typical hierarchy of electronic packaging assembly processes. The hierarchy was divided into four levels (Wang et al., 2009). The first level is the part between an IC die to a chip carrier. On the second level, refers to the interconnection of multichip through a substrate interconnect on the PCB or can also be referred as PWA (printed wiring assembly). The interconnection technologies in the second level can be either through hole or surface mount technologies (SMT). Cu has been widely used as conductor traces on the PCB in electronic packaging assemblies. The Cu traces work as the communication paths between different IC chips and another component. The third level involves the mounting of many PCBs on a motherboard

through sockets or connectors. Finally, the whole system is complete when motherboards relate to each other containing of several subassemblies

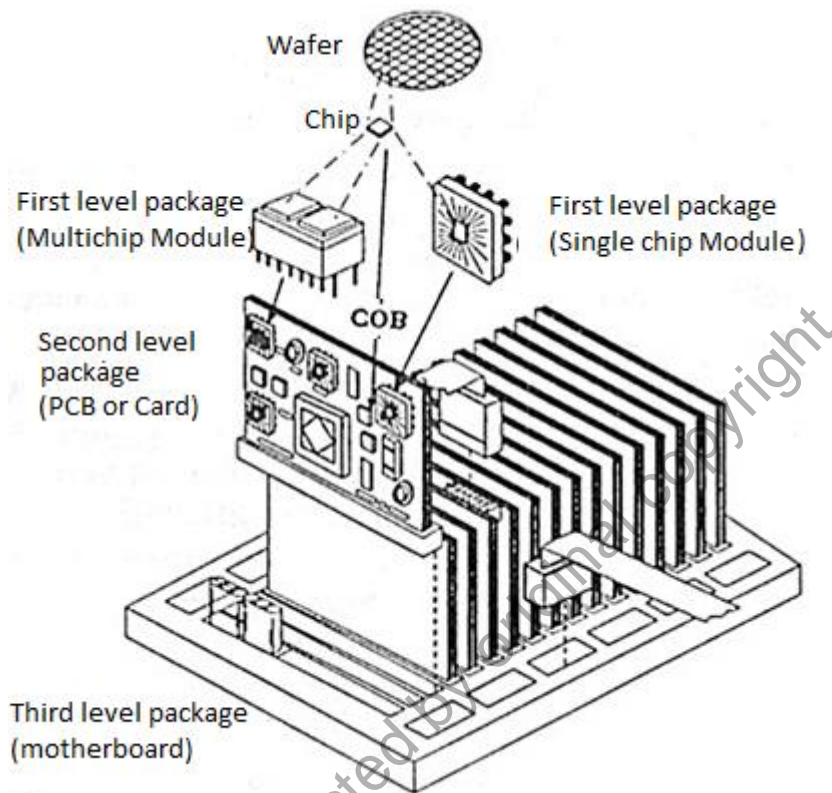


Figure 2.1: The level hierarchy of electronic packaging in electronic products. (Wang & Liu, 2009).

### 2.3 Solder Alloys

The earlier solders were based on alloys of tin while the term of solder is adaption from Old French; soudure which means to make solid. Since 2001 as shown in Table 2.1, either researchers use various type of solder in their research to observe and analyses the solder characteristic. Recently, Sn-Ag, Sn-Ag-Cu, Sn-Zn and Sn-Cu is the common solder used in electronic packaging industrial which to replaced Sn-Pb solder. However, none of them has achieved compare with lead solders (Mohd Salleh et al., 2011)

From table 2.1, the SnCu is not fully investigated by other researchers. It shows that other researchers mostly investigate SAC solder as the restriction of Sn-Pb solder in electronic packaging industrial, the study of this solder has been reduced during year 2010. The SAC solder has greater physical and chemical characteristics compared to SnCu. Apart from that, the wetting speed of SnCu based solder is slower compared to SAC solders (Peter Biocca, 2009). In this study, the SnCu and SAC were chosen and added by ceramic reinforcement to observe the properties evolution.

Table 2.1: Solder uses of another researcher since 2001

Solder	Year	Solder Fabrication	Reference
Sn-9Zn	2010	Casting	(Hu, Hu et al. 2010)
Sn-9Zn-Cr	2010	Casting	(Hu, Hu et al. 2010)
Sn-0.7Cu-Si <sub>3</sub> N <sub>4</sub>	2011	Powder Metallurgy	(Mohd Salleh, Al. et al. 2011)
Sn-0.7Cu-TiO <sub>2</sub>	2011	Mechanical Alloying	(Tsao 2011)
Sn-9Zn-Al <sub>2</sub> O <sub>3</sub>	2010	Mechanical Alloying	(Fouzder, Gain et al. 2010)
Sn-0.3Ag-0.7Cu-In	2010	Casting	(Kanlayasiri and Ariga 2010)
Sn-Ag-Cu-1.0ZrO <sub>2</sub>	2011	Mechanical Alloying	(Gain, Fouzder et al. 2011)
Sn-3.5Ag-0.7Cu	2006	Powder Metallurgy	(Nai, Wei et al. 2006)
Sn-Ag-Cu-Ni	2009	Mechanical Alloying	(Liu, Yao et al. 2009)
Sn-37Pb	2009	Casting	(Zou and Zhang 2009)
Sn-4Ag	2009	Casting	(Zou and Zhang 2009)

### 2.3.1 Sn-lead Solder

Lead (Pb) was identified as toxicity element that can harm human health and environmental. Hence, Pb, legislation was establish in regard to restrict the uses of lead

in electronic industries due to safety and health concern ( Kotadia et al., 2014). When used in hand soldering and tinning operations, the risk of lead exposure was significantly higher due to lead being relatively non-volatile at normal soldering temperature (Allenby & Ciccarelli, 1993). Among the major effect that lead can cause to health are respiratory system disorder and delay in neurological nerve relay (Monsalve, 1984).

Three commands under the European Parliament and the council of European Union have been initiated by the European Commission for restriction of the use of Pb in electronic applications. There are Waste Electrical And Electronic Equipment (WEEE), Restriction of Use of Hazardous Substances (RoSH) and End of Life Vehicle (ELV) directives (Satyanarayan & Prabu, 2011). The European Union's order on waste electrical and electronic equipment (WEEE) announced that after January 2006 the use of lead in the consumer electronic market was prohibited ( Tsao, 2011).

### **2.3.2 Sn-0.7Cu Solder**

Eutectic SnCu solder was used as one of the alternative to Sn-Pb solder due to its lowest cost among Pb-free solders but still cost 30 % higher than conventional Pb-based solder (Satyanarayan & Prabhu, 2011). The Sn-0.7Cu was widely 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). Figure 2.2 shows the phase diagram of Sn-Cu system.

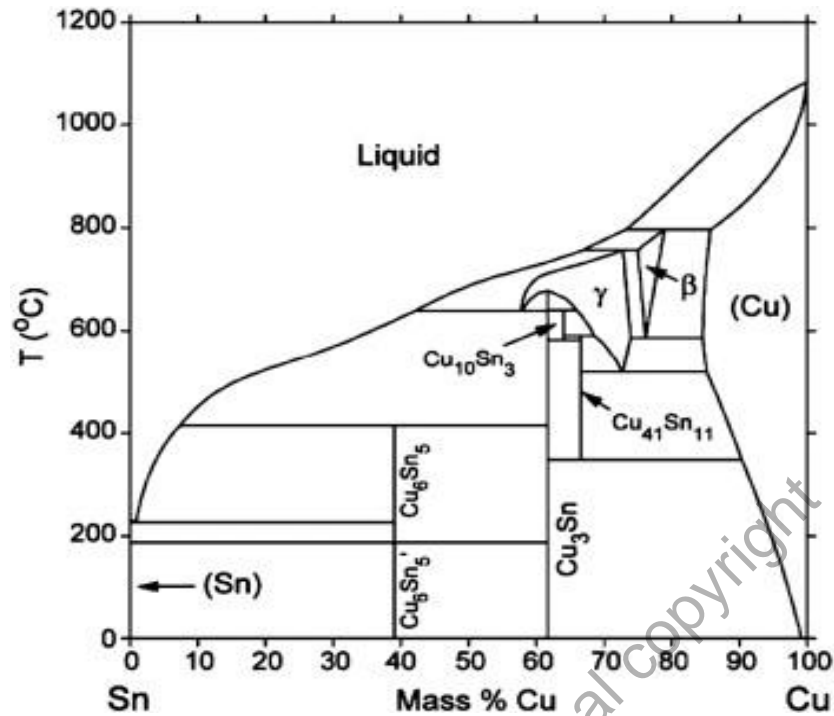


Figure 2.2: Phase diagram of Sn-Cu system (Prabhu, 2011)

### 2.3.3 Sn-Ag-Cu (SAC) solder

One of the most preferred and popular solder used surface mount technology (SMT), ball grid array (BGA) applications and chip scale package (CSP) solder interconnects is SAC alloys with high content of Ag (El-Daly et al., 2013). But due to the higher rigidity of high Ag SAC alloys compare to Sn-Pb alloys, more failure in term of drop and high impact test can be observed when high Ag SAC alloys where used as solder material (Shnawah et al., 2012). In addition, serious chip joining problem such as chip-to-package interaction (CPI) or white bump defects in a back-end-of-line (BEOL) structure frequently occur due to their joint strength. Consequently, the performance of SAC solder joints under high strain rate and large temperature range typical of drop impact situations is a major concern. To resolve this problem, SAC solders with low content of Ag or Cu were developed. Reducing Ag content of the SAC alloys, such as