UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION OF MULTI-WALL CARBON NANOTUBE/MULTILAYER GRAPHENE FIBRE-REINFORCED SOLDER ALLOY COMPOSITES

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By

NIMA GHAMARIAN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

Dedicated to my family for their love, support and encouragement

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

CHARACTERIZATION OF MULTI-WALL CARBON NANOTUBE/MULTI-LAYER GRAPHENE FIBRE-REINFORCED SOLDER ALLOY COMPOSITES

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Chairman : Associate Professor Azmah Hanim Mohamed Ariff, PhD
Faculty : Engineering

In this study, the metallurgical effect of multi-wall carbon nanotube (MWCNT) and multi-layer graphene (MLG) on the eutectic Bi-Ag alloy were explored. Plain Bi-2.5Ag and its reinforced solder systems (Bi-2.5Ag + xMWCNT/ xMLG, x= 0.01, 0.03, 0.05, 0.07 and 0.1 wt%) were investigated through melting temperature, electrical conductivity, corrosion behavior, wettability and mechanical strength. The composite samples were produced following the powder metallurgy method. The results presented for mechanical alloying demonstrate that this method was a suitable technique for dispersing MWCNT and MLG in Bi-Ag powders. Overall, MWCNT and MLG increased the melting point. The maximum melting point recorded when 0.1 wt% of nano particles was in the matrix as 272.02 °C and 269.62 °C for MWCNT and MLG, respectively. It was found that for both reinforced nano particles in the matrix, the electrical resistivity decreased; while, the effect of MWCNT on the electrical resistivity of the solder matrix was more than MLG. The maximum decrease of resistivity was observed for the sample with 0.1 wt% of nano particles which is 1.89 (μΩ.cm) for MWCNT and 2.53 (μΩ.cm) for MLG. The result showed that MWCNT and MLG improved the Bi-2.5Ag wettability. However, it was observed that MLG decreases the wetting angle more than MWCNT. The corrosion behavior of Bi-2.5Ag and its composite samples with MWCNT and MLG was investigated by an electrochemical technique. The results from Tafel plot curves which were run in three different acidic electrolytes illustrated that the corrosion rate for all the composite samples increased. Furthermore, it was deduced that the corrosion rate and passivation were the functions of the electrical conductivity of the sample, the electrical conductivity of electrolyte and the number of H^+ in the corrosive electrolyte. Adding MWCNT and MLG to the Bi-2.5Ag improved the shear strength. However, it was observed that the shear stress of Bi-2.5Ag by adding MWCNT and MLG nano particles just improved to some particular weight percentages of nano particles in the

matrix which were 0.07 wt% for MWCNT and 0.05 wt% for MLG. In a nutshell, adding MWCNT and MLG nano particles to Bi-2.5Ag solder matrix improved the melting behavior, electrical conductivity, wettability and shear strength but negatively impact the corrosion behavior.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENCIRIAN KARBON NANOTIUB BERBILANG DINDING/GRAFIN BERBILANG LAPIS SERAT MEMPERKUKUH ALOI PATERI KOMPOSIT

Oleh

NIMA GHAMARIAN

September 2017

Pengerusi : Profesor Madya Azmah Hanim binti Mohamed Ariff, PhD
Fakulti : Kejuruteraan

Yang demikian, dalam kajian ini, kesan metalurgi karbon nanotiub berbilang dinding (MWCNT) dan grafin pelbagai lapisan (MLG) dalam eutektik Bi-Ag telah diterokai. Bi-2.5Ag yang diperkukuh dengan pateri sistem (Bi-2.5Ag + xMWCNT / xMLG, x = 0.01, 0.03, 0.05, 0.07 dan 0.1 wt%) telah dikaji melalui suhu lebur, pengaliran elektrik, tingkah-laku hakisan, keboleh basahan dan kekuatan mekanikal. Sampel komposit dihasilkan menggunakan kaedah metalurgi serbuk. Keputusan yang diperolehi untuk pengaloian mekanikal menunjukkan bahawa kaedah ini adalah satu teknik yang sesuai untuk menyebarkan MWCNT dan MLG dalam serbuk Bi-Ag. Secara keseluruhannya, MWCNT dan MLG telah meningkatkan suhu takat lebur bahan. Takat lebur maksimum direkodkan apabila 0.1 wt % zarah nano berada di dalam matriks iaitu 272.02 °C dan 269.62 °C untuk MWCNT dan MLG. Didapati bahawa untuk kedua-dua zarah nano diperkuat matriks, ketahanan elektriknya berkurangan; manakala, kesan MWCNT pada ketahanan elektrik pateri adalah melebihi daripada MLG. Penurunan maksimum ketahanan diperhatikan bagi sampel dengan 0.1 wt% zarah nano iaitu 1.89 (μΩ.cm) MWCNT dan 2.53 (μΩ.cm) bagi MLG. Keputusan menunjukkan bahawa MWCNT dan MLG menambah baik keboleh-basahan Bi-2.5Ag. Walau bagaimanapun, telah diperhatikan bahawa MLG lebih mengurangkan sudut basahan berbanding MWCNT. Kelakuan hakisan Bi-2.5Ag dan sampel komposit dengan MWCNT dan MLG dikaji melalui teknik elektrokimia. Hasil daripada plot Tafel, lengkung yang dijalankan dalam tiga elektrolit yang berbeza menggambarkan bahawa kadar hakisan bagi semua sampel komposit meningkat. Selain itu, dapat disimpulkan bahawa kadar kakisan dan pasivasi adalah bergantung kepada konduktiviti sampel, konduktiviti elektrik elektrolit dan nombor H^+ di dalam elektrolit mengakis. Penambahan MWCNT dan MLG bagi Bi-2.5Ag meningkatkan kekuatan ricih. Walau bagaimanapun, adalah diperhatikan bahawa tegasan ricih Bi-2.5Ag dengan penambahan MWCNT dan MLG zarah nano hanya menambah baik sehingga peratusan berat zarah nano dalam matriks mencapai 0.07 wt% bagi MWCNT

dan 0.05 wt% untuk MLG. Secara ringkas, penambahan zarah nano MWCNT dan MLG ke Bi-2.5Ag matrix pateri telah menambah baik tingkah laku peleburan, pengaliran elektrik, keboleh basahan dan kekuatan ricih, tetapi memberi impak negatif terhadap tingkah laku hakisan bahan.

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I certify that a Thesis Examination Committee has met on 28 September 2017 to conduct the final examination of Nima Ghamarian on her thesis entitled "Characterization of Multi-Wall Carbon Nanotube/Multi-Layer Graphene Fibre-Reinforced Solder Alloy Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

Δl	Displacement
ΔR	Differential resistance
$2\theta^{\circ}$	2 Theta degree
A	Ampere
AFM	Atomic Force Microscopy
Ag	Aurum (Silver)
Au	Argentum (Gold)
BC	Before Christ
BET	Brunauer-Emmer-Taller
BGA	Ball grid array
Bi	Bismuth
C	Coulomb
C.R.	Corrosion rate
CE	Counter electrode
CNT	Carbon nano tube
Co	Cobalt
CTE	Thermal expansion coefficient
Cu	Copper
CV	Cyclic voltammetric
DC	Direct current circuit
DSC	Differential Scanning Calorimeter
E_{corr}	Corrosion potential
EDX	Energy Dispersive X-ray
E_g	Band gap energy

EMF	Electromotive force series
E_{pp}	Passivation potential
EPR	European Recycling Platform
EW	Equivalent weight
F	Faraday constant
FESEM	Field Emission Scanning Electron Microscope
ɣ	Shear strain
TEM	Transmission Electron Microscope
i_{cc}	Critical current
i_{corr}	Corrosion current
IMC	Intermetallic compound
i_p	Passive current
IPP	Integrated product policy
K	Kelvin
MCV	Maximum concentration value
MLG	Multi layer graphene
MWCNT	Multi wall carbon nano tube
ɳ	Over-potential
Ni	Nickel
Pb	Lead
PBBs	Polybrominated biphenyls
PBDEs	Polybrominated diphenyl ethers
PCB	Printed circuit board
PWB	Printed wiring board
Q	Electric charge

R	Resistance
RE	Reference electrode
RoHS	Restriction of Hazardous Substances
s	Second
SAC	SnAgCu (Tin-silver-copper)
Sb	Antimony
SCE	Standard Calomel Electrode
SLG	Single layer graphene
Sn	Tin
SWCNT	Single wall carbon nano tube
T	Temperature
t	Thickness
T	Shear stress
T_c	Critical temperature
um	Micrometer
UTM	Universal Testing Machine
UV	Ultra Violet
WE	Working electrode
wt%	Weight percentage
X	Magnification
XRD	X-ray Diffraction
Zn	Zinc
θ	Wetting angle
p	Electrical resistivity
ρ	Density

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The tendency in utilizing electronic products will be in wireless, portable, and handheld features. This fact drives the industrial growth in the microelectronic technologies. The rapid growth, demands for new materials more than ever (Berni et al., 2016; Sattler, 2016). Solder alloys and conductive adhesives components have been used by electronic manufacturers to provide electrical interconnection between electrical segments and the printed wiring board (PWB)(Dunn, 2015). Lead-Tin (Pb-Sn) alloys are a promising interconnection material. Lead (Pb) has been used since the 1940s in view of its proven reliability with eutectic phase when chemically mixed with tin (Sn) (Plumbridge, 1996).

According to mass production of electronic gadgets, it is impossible to turn a blind eye on the disposal of these products and abandon them in the environment (Kang and Schoenung, 2005; Rydh and Sun, 2005). Solder alloys and conductive adhesives contain a high level of Pb (Li et al., 2005). Pb and its alloys as a heavy metal under certain conditions are harmful to human and environment; therefore some countries impose a law to limit the usage of Pb in the industries. For instance, Canada bans Pb in petrol in 1990 (Bollhöfer and Rosman, 2001), U.S.A in 1996 (Kovarik, 2005), and in 2002 it is banned in the European Union (Bollhöfer and Rosman, 2001).

European Union Parliament and Council resolution, in July 2006, restrict the use of Hazardous Substance (RoHS) in electrical products to avoid Pb in manufacturing. Furthermore, in January 2005, Japan as the paramount electronic gadget producers admitted widening the use of lead free solders instead of lead solders (Hwang, 2004a). The limitation of using Pb in electronic industries carves a path for the scholars to look for lead free alloys to suit their application as high and low temperature solders (Frear and Vianco, 1994).

In the recent times, heat generation and thermal conductivity in electronic device which were used in aerospace, automotive, militaries, nuclear power facilities, oil industry and monitors initiate the need for high temperature lead free solder (Chidambaram et al., 2011; Kang and Sarkhel, 1994; Suganuma, 2001; Suganuma et al., 2009). Some elements such as gold (Au), zinc (Zn), bismuth (Bi) and tin (Sn) have been suggested to replaced Pb as lead free soldering alloys in high temperature applications (Evans, 2007).

Among all suggested high temperature lead free solder alloys, Bismuth-Silver Alloys (Bi-Ag) provides better features for different applications (Shimoda et al., 2012). It can be concluded from the Bi-Ag phase diagram that a binary system contains only one eutectic reaction, which occurs at 97.5% Bi and at a temperature around 262.5 °C. There are no intermetallic parts, and the low solubility of silver in bismuth precludes solid-solution establishment. The presence of the silver element in the microstructure makes Bi-Ag alloys mechanically stronger and more ductile than pure bismuth (Nahavandi, 2014).

The reliability and applicability of Bi-Ag solder alloy in industries need further investigations considering their advantages (e.g., good electrical conductivity) and disadvantages (e.g., brittleness) (Song et al., 2006, 2007b). Thus, the investigations to improve the physical and mechanical properties of these alloys is ongoing (Spinelli et al., 2014).

Studies have shown that adding alloying elements frequently improves the properties of alloy (Allaoui et al., 2002; Avner, 1964). Improvement in the characteristics of lead free solders by proper selection of the reinforcement materials could advantageously produce better alloy due to its physical and mechanical properties (Gancarz et al., 2014; Yang et al., 2014). In consideration of the aforementioned facts, multi wall carbon nanotube (MWCNT) and multi-layer graphene (MLG) were selected as the reinforcement materials in the current study according to their good electrical conductivity and mechanical strength.

In the nutshell, the eutectic Bi-Ag lead-free solder reinforced with varying weight (0.01, 0.03, 0.05, 0.07 and 0.1 wt%) of multi wall carbon nanotube (MWCNT) and multi-layer graphene (MLG) were analyzed in order to fabricate the composite solder. Powder metallurgy technique was used for mixing and compaction of the elements. Then, the pellets were melted in the furnace on copper (Cu) plates. Physical, shear strength, corrosion behavior and electrical conductivity effects of MWCNT and MLG on lead free eutectic Bi-Ag solder alloy were studied.

1.2 Problem Statement

It is crystal clear that; the wide use of the electronic equipment's. Nowadays, lead (Pb) is a commonly used element in many components of the electronic industry especially as solder alloys (Pb-Sn) (Tribula et al., 1989). Pb in solder materials is used in coatings soldered on printed circuit boards, pins and ends of boards (Lee et al., 2001; Zeng and Tu, 2002).

Due to the fact that Pb is in the class of top seventeen chemical poisoning elements (heavy elements), it leaves no doubt about its harmful effect on human health. Its accumulation in the human body causes disorders in the nervous system, reproductive systems, the disturbance in neurological and physical development, anemia and hypertension (Duruibe et al., 2007).

Prohibiting the use of solder containing Pb in many industries was applied from 2006 in United States of America (USA). Regarding this fact, a new solder alloy, Bi-Ag, as one of the lead-free solder alloys has been considered as a promising replacement for high temperature solder applications (Song et al., 2007b).

But Bi-Ag alloys are still not vastly used in industries. It has some disadvantages such as brittleness, low electrical conductivity and low shear modulus. Tackling this problem and improving the binary phase alloys especially its eutectic has caught the attention of scholars (Song et al., 2007a).

It has been suggested by the researchers that adding one or more elements can be helpful to improve the properties of the alloy (Efzan and Singh, 2014). Due to this fact, the metallurgical effect of multi wall carbon nanotube (MWCNT) and multi-layer graphene (MLG) on eutectic Bi-Ag alloy was studied and proposed in this research.

1.3 Significance of Study

Reliability of interconnection joints is a major concern in the improvement of lead-free solders. The investigation in this subject area must first prioritize the existing trend in the electronic packaging industries. The current choices for replacements of lead alloy system are based on Ag, Bi, Zn and Sn alloy systems. So far, some preference has been placed on the Bi-Ag solder material as a promising candidate but it is not a matured material yet with the required reliability and optimized properties to be use in the industry.

In this research, based on the desirable properties shown by the Bi-Ag binary alloy, an attempt is made to investigate the alternative of nano-composite Bi-2.5Ag for high-temperature application. Therefore, this thesis critically examines and documents the detailed information required to raise the consciousness of researches and electronic industries towards a revamped Bi-Ag lead-free solder, through the incorporation of carbon nanotubes and graphene.

1.4 Objectives of the Research

The objectives of the present work which are effects of multi wall carbon nanotubes and multi-layer graphene on Bi-2.5Ag lead free solder were summarized as follows:

1. To analyze the effect of MWCNT and MLG on the melting performance of Bi-2.5Ag alloys.

2. To determine the effect of MWCNT and MLG on the electrical conductivity of Bi-2.5Ag alloys at room temperature.

3. To investigate the effect of MWCNT and MLG on the corrosion behavior of Bi-2.5Ag alloys by electrochemical method.

4. To determine the effect of MWCNT and MLG on the wetting/contact angle of Bi-2.5Ag alloys on a Cu substrate.

5. To analyze the effect of MWCNT and MLG on the shear strength of Bi-2.5Ag solder using single lap-shear test method.

1.5 Scope of Study

The scope of the research is centered on five principal properties of solder alloys which are critical in analyzing the reliability of the solder joints. The effect of reinforcement of Bi-2.5Ag with MWCNT and MLG (Bi-2.5Ag + xMWCNT/xMLG, x= 0.01, 0.03, 0.05, 0.07 and 0.1 wt%) were studied according to the melting temperature, electrical conductivity, corrosion behavior, wetting angle and strengthening of solder composite.

The Bi-2.5Ag composite was produced by mechanical alloying of components which were in the powder form. Differential Scanning Calorimetry (DSC) was used to find the effect of MWCNT and MLG on melting behavior of Bi-2.5Ag. In order to report the wetting angle associated with each experiment (each weight percentage of nano particles in the solder matrix). Six angles were measured by an optical microscope (OM) and the mean value was determined. Due to the importance of electrical resistivity of solder and interconnection alloys for electronic assembly, the effect of MWCNT and MLG on Bi-2.5Ag was studied using Standard Four-Probe Technique. Corrosion behavior of reinforced samples was studied by electrochemical method in three corrosive electrolytes (HCl, HNO_3 and H_2SO_4). Furthermore, composite Bi-2.5Ag joints behavior was investigated using single lap-shear test method by 10 kN universal testing machine. In the interest of studying the single lap joints sample failure during the shear test, the fractured samples were randomly selected and investigated using an optical microscope (OM) and atomic force microscopy (AFM).

1.6 Hypotheses

The study's research hypotheses are as follow:

H1: Melting behavior of Bi-2.5Ag will be affected by MWCNT and MLG reinforcement. Some researches show significant in melting point while other researches show no effect at all or negligible difference in melting point with addition of MWCNT and MLG in lead free solders.

H2: Electrical conductivity of Bi-2.5Ag will be improved by adding reinforcement nano-particles, since MWCNT and MLG are excellent electrical conductors.

H3: Wettability of Bi-2.5Ag will be improved by adding MWCNT and MLG to the solder matrix up to a certain percentage. After a certain threshold, the wetting angle increase due to agglomeration of the reinforcements.

H4: Corrosion behavior of Bi-2.5Ag will be affected by MWCNT and MLG reinforcement. Not enough information to predict the trend of the behavior and the extent of the difference.

H5: Shear strength of Bi-2.5Ag will be improved by adding reinforcement nano-particles up to a certain percentage of reinforcement. Beyond a certain threshold value, agglomeration of MWCNT and MLG will have a negative impact on the shear strength of the solder.

1.7 Structure of the Thesis

The thesis was written into five chapters. After an introduction which describes the background of this research, problem statements, and research objective are in the first chapter. The framework structure of the remaining part of the thesis is as follow:

Chapter two gives literature review on the general knowledge within the scope of this research, materials, technologies and methods. Chapter three illustrates in detail the methodologies which were conducted in this study.

Chapter four discusses and explains the results (based on the analyzed experimental data) of the metallurgical effects of multi walled carbon nanotubes or multi-layer graphene on eutectic Bi-2.5Ag lead free solder.

Finally, chapter five summarizes and reviews all the present analyzed works and provides deductions on the observations obtained by this research. In this chapter, recommendations are also given for future research directions.

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