UNIVERSITI PUTRA MALAYSIA

INTERMETALLICS EVOLUTION AND SHEAR STRENGTH OF CARBON NANOTUBES-REINFORCED Sn-5Sb SOLDER ON COPPER BOARD

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FK 2015 14

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Thesis Submitted to the School of Graduate Studies,

Universiti Putra Malaysia, in Fulfillment of the

Requirements for the Degree of

Master of Science

June 2015

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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June 2015

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Critical evaluation of interconnection materials developed from composite solder materials and a comprehensive comparison of the joints with the host solder alloy are cardinal to the quest of divulging a more decent and dependable alternative to the existing solder candidate. In this study, the effects of multi-walled carbon nanotubes (MWCNTs) addition on the physical properties, intermetallic compound formation and shear strength property were explored. Plain Sn-5Sb solder system and carbon nanotubes reinforced solder systems (Sn-5Sb-xCNT; x=0.01, 0.05 & 0.1wt.%) were developed through the powder metallurgy route. In order to characterize the solder joint microstructure and intermetallic compound growth in the samples, plain and composite solders were reflowed on the copper (Cu) substrate at a peak temperature of 270°C. Similarly, the single-lap solder joint system was fabricated and used to evaluate the shear strength of all solder samples using the same reflow temperature as in the case of the solder/Cu joint. After the reflow process, isothermal aging study was conducted on the solder/Cu joints and the single-lap solder joint system at 170°C for time intervals of 500h, 1000h and 1500h. The melting temperature results indicated that the presence of carbon nanotubes addition to the solder matrix lead to a marginal decrease in the peak temperature of the composite solders due to high surface free energy of MWCNTs in the solder matrix with the 0.1wt.% CNTs solder reinforcement having a maximum drop of 2.72°C in melting temperature as compared to the plain solder counterpart. More so, it was evident from the study that the wetting angle decreased with increasing CNTs addition. The Sn-5Sb-0.05CNT solder joint exhibited a 14% reduction in the wetting angle result than that of the plain solder joint whereas, increasing the CNTs content up to 0.1wt.% resulted in a marginal

leap of the wetting angle. Microstructural analysis of all samples using the combined study of the SEM and EDX analysis confirms the formation of a dark dendritic shaped Cu_6Sn_5 intermetallic compound (IMC) phase floating in the solder bump which coarsens as aging time increased. Besides, light grey precipitates were spotted within the β-Sn matrix. A sparse dispersion of the precipitates was observed in the composite solder joints as against the plain solder joints for all subjected conditions. From the IMC thickness data retrieved, a minimal retardation in the total intermetallic compound layer growth was observed in the composite solder joints with respect to the plain solder joint for the reflow samples, where IMC thicknesses of 2.60μm and 3.04μm were recorded for the Sn-5Sb-0.05CNT and Sn-5Sb solder joints respectively. Meanwhile, IMC layer growth in the aged samples was observed to have being remarkably suppressed with the Sn-5Sb-0.05CNT solder joint having a value of 11.99μm for 1500h aging condition which indicates 2.64μm reduction in the value of the plain solder joint counterpart as well as the inhibiting role played by the CNTs in prohibiting the diffusion of Sn atoms needed for the interfacial IMC layer growth. Similarly, owing to the excellent mechanical properties and the level of IMC layer retardation shown by the composite solder joints, the 0.01wt.% CNTs solder reinforcement gave the highest shear strength than every other single-lap solder joints across board.

Abstrak tesis yang dibentangkan kepada Senat Universiti Putra Malaysia bagi memenuhi keperluan ijazah Sarjana Sains

EVOLUSI SEBATIAN ANTARA LOGAM DAN KEKUATAN RICIH LOGAM PATERI Sn-5Sb DIPERKUAT DENGAN NANOTIUB KARBON DI ATAS KEPINGAN KUPRUM

Oleh

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Penilaian kritikal terhadap sambung tara dipertingkatkan dengan penggunaan bahan pateri komposit dan perbandingan secara meluas sambungan menggunakan bahan asas pateri aloi adalah penting dalam penyelidikan ini bagi mencari alternatif lain kepada bahan pateri sedia ada. Dalam kajian ini, kesan pertambahan nanotiub karbon berbilang-dinding (MWCNTs) terhadap ciri fizikal, logam antara muka dan kekuatan daya ricih telah dikaji. Sistem pateri biasa Sn-5Sb dan sistem pateri diperkuat dengan nanotiub karbon (Sn-5Sb-xCNT; x=0.01, 0.05 & 0.1wt. %) telah dihasilkan melalui kaedah metalurgi serbuk. Bagi pencirian mikrostruktur sambungan pateri dan pertumbuhan sebatian antara logam dalam sampel, pateri biasa dan komposit telah di cairkan diatas kepingan tembaga (Cu) substrat pada suhu puncak 270°C. Begitu juga dengan sistem sambungan pateri satu-lapisan yang telah dihasilkan dan digunakan untuk menilai kekuatan ricih pada semua sampel juga menggunakan suhu pencairan yang sama seperti dalam kes sambunagan pateri/Cu. Selepas process pencairan, kajian penuaan suhu setara telah dijalankan ke atas sistem sambungan pateri/Cu dan sambungan pateri satu-lapisan pada 170°C untuk selang masa 500 jam, 1000 jam dan 1500 jam. Maklumat yang diperolehi daripada suhu lebur menunjukkan bahawa pertambahan nanotiub karbon di dalam matrik pateri menyumbang kepada penurunan suhu maksimum pateri komposit kerana MWCNTs mempunyai tenaga bebas yang tinggi di dalam matrik pateri. Setelah diperkuat dengan 0.1wt.% CNTs, pateri komposit mengalami penurunan suhu maksimum

sebanyak 2.72°C untuk titik leburnya berbanding dengan pateri biasa. Tambahan pula, kajian ini jelas menunjukkan bahawa sudut pembasahan juga menurun dengan pertambahan CNTs. Sambungan pateri yang diperkuat dengan 0.05wt.% CNTs mengalami penurunan sebanyak 14% untuk sudut pembasahan berbanding dengan pateri biasa dan sementara itu, pertambahan CNTs sehingga 0.1wt.% menyebabkan sudut pembasahan meningkat. Analisis mikrostruktur terhadap kesemua sampel dengan menggunakan SEM dan EDX, mengesahkan terdapat pembentukan fasa sebatian antara logam (IMC) Cu_6Sn_5 berbentuk dendrit gelap yang kasar diatas permukaan pateri disebabkan oleh masa penuaan yang meningkat. Selain itu, mendakan kelabu bercahaya juga turut kelihatan disekitar matrik β-Sn. Diperhatikan juga, penyebaran mendakan kurang berlaku dalam sambungan pateri komposit berbanding dengan sambungan pateri biasa untuk kesemua keadaan. Data ketebalan IMC yang diambil menunjukkan, pertumbuhan lapisan sebatian antara logam bagi sambungan pateri komposit adalah minimum berbanding dengan sambungan pateri biasa bagi sampel setelah di cairkan kali pertama, dimana bagi sambungan pateri Sn -5Sb-0.05CNT dan Sn-5Sb masing-masing telah mencatatkan ketebalan IMC 2.60μm dan 3.04μm. Sementara itu, pertumbuhan lapisan IMC bagi sambungan pateri Sn-5Sb-0.05CNT juga diperhatikan mengalami perubahan yang ketara dengan ketebalan 11.99 μm bagi keadaan penuaan selama 1500 jam, ianya menunjukkan pengurangan sebanyak 2.64 μm daripada sambungan pateri biasa. Hal ini disebabkan oleh CNTs yang menghalang pemindahan atom Sn yang diperlukan untuk pertumbuhan lapisan antara muka. Oleh kerana ciri-ciri mekanikal dan tahap pembantutan lapisan IMC yang ditunjukkan oleh sambungan pateri komposit adalah tinggi, pateri yang diperkuat dengan 0.01wt.% CNTs memberikan kekutan daya ricih yang paling tinggi berbanding sambungan pateri satu-lapisan yang lain secara keseluruhannya.

ACKNOWLEDGEMENT

My heartfelt gratitude goes to my parents, Prof & Mrs Dele-Afolabi for their resolute support, financial backing and the dedicatory prayers bestowed on me throughout the course of this work.

The immense effort, constructive comments and profound guidance received from the chairman of my supervisory committee, Dr Azmah Hanim Mohamed Ariff are heartily treasured.

I also wish to express my appreciation to the members of my supervisory committee, Dr Norkhairunnisa Mazlan, Dr Hamdan Mohamed and Dr Suraya Mohd Tahir for their reformative suggestions and supervision.

Special thanks to Dr Dele-Afolabi Folarin and Dele-Afolabi Adeola for their love, encouragement and support in making this academic pursuit a reality.

The excellent advice from friends and family members are equally acknowledged.

Declaration by graduate student

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LIST OF SYMBOLS AND ABBREVIATIONS

As	As Soldered
h	Hours
°C	Degree Celsius
N	Newton
μm	Micrometer
nm	Nanometer
mm	Millimeter
wt.%	Weight Percent
MPa	Mega Pascal
OM	Optical Microscopy
SEM	Scanning Electron Microscopy
FESEM	Field Emission Scanning Electron Microscope
DSC	Differential Scanning Calorimetry
UTM	Universal Testing Machine
PM	Powder Metallurgy
EDX	Energy Dispersive X-ray
IMC	Intermetallic Compound
CNTs	Carbon Nanotubes

MWCNTs	Multi Walled Carbon Nanotubes
SWCNTs	Single Walled Carbon Nanotubes
FCNT	Functionalized Carbon Nanotubes
RoHS	Restriction of Hazardous Substance
WEEE	Waste of Electrical and Equipment
JEITA	Japan Institute of Electronics Industry Technology Association
IPC	Institute of Interconnecting and Packaging Electrical Circuits
YS	Yield Strength
UTS	Ultimate Tensile Strength
USS	Ultimate Shear Strength
ASTM	American Society for Testing and Materials
RMA	Rosin Mildly Active Flux
CTE	Coefficient of Thermal Expansion
Cu	Copper
Sn	Tin
Sb	Antimony
Au	Gold
O	Oxygen
C	Carbon
SAC	Sn-Ag-Cu (Tin-Silver-Copper)

CHAPTER ONE

INTRODUCTION

1.1 Background of Research

Solder joints for electronic packaging serves as a connecting medium between the electrical components and the circuit board. However, characterization of the intermetallic compound formed during soldering process is highly essential in predicting investigations on lead-free solders that the intermetallic compound layers (IMCs) might serve as failure initiation point in interconnection joints due to their brittle nature. For these reasons, suggestions have been raised to suppress the excessive growth of this interfacial IMC layers (Kantarcioglu and kalay, 2014). Meanwhile, the trend of more sophisticated, efficient and miniaturized electrical systems has led to the shrinkage in the interconnection joints of electrical components thereby posing several challenges such as solder joint reliability to the development of lead-free solders as well as the electronic packaging industries (Tang et al., 2014). Similarly, the consideration of a solder as a potential substitute for the conventional Sn-Pb solders implies that such solder must closely possess the properties and dependable reliability attribute of the binary Sn-Pb solder.

In recent times, investigations have shown that the addition of alloying elements often bring improvement in the properties of lead-free solders and appropriate selection of reinforcement materials could desirably introduce a more homogenous or non-coarsening particles (Yang et al., 2014 and Gancarz et al., 2014). In light of the aforementioned facts, carbon nanotubes (CNTs), a typical form of material which is gaining momentum in scientific explorations was introduced as the reinforcement material in the current study.

For the purpose of this study, the Sn-5Sb reinforced with varying weight (i.e. 0.01, 0.05 and 0.1wt. %) multi walled carbon nanotubes (MWCNTs) will be fabricated through the powder metallurgy technique consisting of blending and compaction. Hence, the compacted composite Sn-5Sb-xMWCNT will be reflowed in the oven and thereafter subjected to the aging process after which microstructural and shear strength analysis will be explored for proper documentation of the intermetallics evolution and mechanical properties of the fabricated composite solder.

1.2 Problem Statement

Technological progression has been a great tool in global development and advancement. The electronics and micro-system industries are not left behind in this as more powerful, efficient and miniaturized electrical gadgets have been produced in recent times. Owing to this great advancement in technology, the input/output terminals in electronic packaging have greatly increased resulting to a proportional increase in solder interconnection joints of electronic components (Kumar et al, 2008).

In time past, the eutectic alloys of 60Sn-40Pb and 63Sn-37Pb solder alloy with a eutectic melting temperature of 183°C have been widely used and they have long yielded so many benefits in the course of electronic/component packaging due to factors such as good reliability, low cost and good wettability on copper (Cu) substrate. However, the advent of green technology has led to the enactment of legislations restricting the usage of the customarily used leaded solders and encouraging the utilization of environmental friendly materials.

Solder alloys to be used in densely packed electronic devices must possess such properties that will make them stand the test of time and withstand adverse effects associated with high homologous temperature developed throughout the service life of such device. Similarly, for the production of good and dependable interconnects, the intermetallic compound (IMC) stratum formed between the solder alloy and substrate must possess desirable characteristics. Naturally, the brittle nature of IMC increases as the layer thickness increases thereby enhancing the propensity to generate defects such as cracks or coarseness of microstructural grains. Unfortunately, none of the lead free solders available has proven to be suitable to replace the high lead based solders due to setbacks resulting from unfavorable intermetallics growth, physical and mechanical properties. Hence, industries are in need of solder materials with enhanced electrical and mechanical properties that can meet up with the present demands of the end users.

In recent times, research has shown that the addition of alloying elements often improves the properties of lead-free solders. Besides, appropriate selection of reinforcement materials could desirably introduce a more homogenous or non-coarsened particles (Laurila et al, 2009). More so, investigations by Nai et al. (2009) and Niranjaini et al. (2011) have shown a significant improvement in the interfacial intermetallics compound growth and mechanical properties of CNTs reinforced solder joints.

However, despite the remarkable roles played by carbon nanotubes (CNTs) incorporation into the lead-free solder matrix, no investigation is yet to be conducted on the influence of CNTs in the Sn-5Sb solder system. Therefore, the current study attempts to offer a promising candidate in the form of a composite solder to upgrade the intrinsic properties of the conventional Sn-5Sb lead free solder as well as produce a candidate that can decently operate in a robust environment. Through the incorporation of carbon nanotubes to the solder alloy, it is expected that the exceptional physical and mechanical properties of the CNTs will go a long way in deterring the pitfalls associated with the Sn-5Sb/Cu joint.

1.3 Significance of Study

Lifetime operational capability of interconnection joints is of major concern in the evolution of lead free solders. Ongoing and future studies in this subject area must first prioritize the current trend in the electronics/microsystems industries. With the shrinkage in size of electronic gadgets, it is expected that the magnitude of the residual stresses which emanate from temperature fluctuations during the service life of the products increases.

Interestingly, the interconnection joints developed from the Sn-5Sb solder system due to its relatively high melting temperature of 245°C can serve a dual purpose in electronic packaging. First, it can be used conventionally for the step soldering module in electronic packaging which is basically required to minimize the exposure of existing soldered joints with thermally sensitive components from successive reflow process. Secondly, it can be utilized for high temperature application in order to produce reliable soldered joints that will thrive well under extreme service condition and function decently in densely packed devises.

From investigations gathered so far, more preference has been placed on the Sn-Ag-Cu (SAC) solder counterpart as the most promising candidate to replace the lead solders. Hence, other lead free solder counterparts have been subjected to neglect. Similarly, high leaded solders are still preferred over the alternative high temperature lead free solders developed as legislations are yet to be enacted to restrict the usage of this candidate.

Lastly, based on the desirable properties shown by the Sn-Sb family, the alloy tends to be suitable for step soldering technology as well as to relieve the high-lead content solders for high temperature applications. Therefore, this thesis critically examines and documents the detailed information required to raise the consciousness of researchers and electronic industries towards a revamped Sn-5Sb lead free solder, through the incorporation of carbon nanotubes.

1.4 Aim and Objectives

The objectives are:

- To investigate the aging effect on the interfacial morphology of plain Sn-5Sb solder alloy and Sn-5Sb-xMWCNT (x=0.01, 0.05 and 0.1 wt.%) composite solders.
- To study and analyze the shear strength and fractography of the plain Sn-5Sb solder alloy and Sn-5Sb-xMWCNT (x=0.01, 0.05 and 0.1 wt.%) composite solders.

1.5 Scope of Study

The scope of the work is centered on two principal properties (i.e. the intermetallics formation and shear strength analysis) which are quite crucial in analyzing the integrity of the solder joints formed. For the analysis, the soldered joints of the four samples were aged at a temperature of 170°C at various time intervals of 500 h, 1000 h and 1500 h. This procedure is applicable for both the interfacial intermetallic compound layer (IMC) and shear strength analysis. A comparison in the IMC evolution of the as reflowed and the aged samples was made for each of the solder samples followed by the growth kinetics interpretation. Similarly, all samples for the shear strength analysis were subjected to the same aging temperature and results were compared as those for the IMC analysis. Finally, a critical evaluation was made on the fractography results obtained from the SEM/EDX.

1.6 Thesis Outline

The first chapter in the thesis contains sub-sections that give a broad insight into the study as a whole. These include the background, the problem statement, and significance of study and research objectives. The scope in terms of the methods and subjected aging conditions are also included in this chapter.

A critical review of relevant investigations in the line of this study is presented in chapter two, including trends and overview of the important concepts of this work. Overviews of theories, backgrounds and versatility of lead free solders are presented in order to stimulate the sensitivity of the procedural steps embarked upon in this study towards existing standards set aside for evaluation of the interconnection joints produced during the course of this work.

An experimental documentation of the steps and procedures employed in the data collection for the different facets of this work are described in chapter three. Here, overview and discussions on the various sample preparations, equipments

utilized and microscopic techniques are presented. The procedural steps and formula adopted for the measurement of the interfacial intermetallics layer for samples subjected to various solid-state aging conditions are brought to the fore.

Chapter four contains the results obtained in the course of this study and the discussion of the data in relation with the research objectives and the established findings in the field of lead free solders.

In chapter five, the conclusion of the whole work and a summary of the results are presented, including recommendation and major findings.

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