FABRICATION AND CHARACTERIZATION OF HYBRID MICROWAVE ASSISTED SINTERING Sn-0.7Cu + 1.0wt.% Si_3N_4 COMPOSITE SOLDER

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Fabrication and Characterization of Hybrid Microwave Assisted Sintering Sn-0.7Cu + 1.0wt.% Si_3N_4 Composite Solder

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LIST OF ABBREVIATIONS

Ag	Silver
Al_2O_3	Alumina
Au	Gold
Bi	Bismuth
Cd	Cadmium
Cr^{6+}	Hexavalent Chromium
CTE	Coefficient of Thermal Expansion
Cu	Copper
EDX	Energy Dispersive X-ray
EEE	Electrical and Electronic Equipment
EPA	Environmental Protection Agency
EU	European Union
HCl	Hydrochloric acid
Hg	Mercury
HNO_3	Nitric acid
IC	Integrated Circuit
IMC	Intermetallic Compound
In	Indium
ISM	Industrial, Scientific and Medical
ISO	International Standard Organization
MCV	Maximum Concentration Value
MMC	Metal-matrix Composite
NC	No-Clean
NEMI	National Electronics Manufacturing Initiative
Ni	Nickel
N	Nitrogen
OM	Optical Microscope
Pb	Lead
PBB	Polybrominated Biphenyls
PBDE	Polybrominated Diphenyl Ethers
PCB	Printed Circuit Board

PD	Penetration Depth
PM	Powder Metallurgy
PWA	Printed Wiring Assembly
RoHS	Restriction of the Use of certain Hazardous Substances in Electrical and Electronic Equipment
SAC	Sn-Ag-Cu
SEM	Scanning Electron Microscope
Si	Silicon
Si_3N_4	Silicon Nitride
SiC	Silicon Carbide
SMT	Surface Mount Technology
Sn	Tin
SnO_2	Tin Oxide
TiB_2	Titanium Diboride
VPS	Vapour Phase Soldering
WEEE	Waste Electrical and Electronic Equipment
XRD	X-ray Diffraction
ZrO_2	Zirconia

LIST OF SYMBOLS

T_m	Melting temperature
λ	Wavelength
ρ	Density
ν	Volume

Fabrikasi dan Pencirian Pateri Komposit Sn-0.7Cu + 1.0wt.% Si_3N_4 Disinter Secara Hibrid Melalui Bantuan Gelombang Mikro

ABSTRAK

Salah satu kaedah untuk meningkatkan ciri-ciri aloi pateri bebas plumbum yang sedia ada adalah melalui pendekatan teknologi komposit, di mana partikel-partikel seramik berteknikal tinggi boleh ditambah masuk ke dalam matriks aloi pateri. Oleh yang demekian, pateri komposit Sn-0.7Cu + 1.0wt.% Si_3N_4 telah disintesis menggunakan kaedah-kaedah metalurgi serbuk yang terdiri daripada proses pengadunan, pemadatan dan pensinteran di dalam kajian ini. Kajian ini juga telah memperkenalkan proses pensinteran secara hibrid melalui bantuan gelombang mikro yang berkebolehan untuk mensinter pateri komposit bertetulang seramik pada suhu 185 ˚C dalam masa 2 minit tanpa masa pemanasan dan perlindungan gas lengai. Dalam usaha untuk menilai keserasian pendekatan pensinteran secara hibrid melalui bantuan gelombang mikro terhadap pembangunan pateri komposit bertetulang seramik, perbandingan pemprosesan dan sifat-sifat pateri komposit Sn-0.7Cu + 1.0wt.% Si_3N_4 melalui kaedah pensinteran konvensional dan pensinteran gelombang mikro telah dijalankan dengan lebih terperinci. Suhu pensinteran yang sama iaitu 185 ˚C telah digunakan untuk kedua-dua jenis pensinteran, di mana pensinteran konvensional dilakukan di dalam tiub relau dengan kehadiran gas argon selama 2 jam. Sampel pateri monolitik Sn-0.7Cu juga telah disintesis sebagai sampel kawalan dengan cara yang sama. Pensinteran secara hibrid melalui bantuan gelombang mikro menunjukkan kelebihan dalam pemprosesan berbanding kaedah pensinteran konvensional, seperti kadar pemanasan yang tinggi, masa pensinteran yang singkat, kadar penggunaan tenaga yang kurang dan peralatan yang jauh lebih murah. Pengaruh perbezaan metodologi pensinteran pada sampel pateri komposit Sn-0.7Cu + 1.0wt.% Si_3N_4 telah dikaji berdasarkan ketumpatan, keliangan, kekerasan, mikrostruktur, kebolehbasahan dan ketebalan sebatian antara logam ke atas Cu-substrat. Didapati bahawa kaedah pensinteran secara hibrid melalui bantuan gelombang mikro mampu memadatkan kompakan pateri komposit Sn-0.7Cu + 1.0wt.% Si_3N_4 dalam masa yang lebih singkat, bagaimanapun, kaedah pensinteran konvensional di dalam kajian ini menunjukkan kepadatan dan keliangan sampel yang lebih baik. Menariknya, mendakan yang lebih halus dan agihan mendakan yang lebih menyeluruh telah diperhatikan dalam sampel gelombang mikro tersinter. Hal ini juga telah membawa kepada peningkatan prestasi kekerasan pada sampel gelombang mikro tersinter (12.0 ± 0.2 HV) berbanding dengan sampel konvensional tersinter (11.2. ± 0.1 HV). Prestasi kebolehbasahan Sn-0.7Cu + 1.0wt.% Si_3N_4 pada Cu-substrat telah dikurangkan sedikit dengan pendekatan pensinteran gelombang mikro, walaubagaimanapun, perbezaan ketebalan sebatian inter metalik yang tidak ketara telah diperhatikan bagi kedua-dua sampel gelombang mikro tersinter dan sampel konvensional tersinter. Secara keseluruhannya, kaedah pensinteran hibrid melalui bantuan gelombang mikro telah menunjukkan pemprosesan yang lebih baik dengan ciri-ciri yang menjanjikan pada seramik bertetulang pateri komposit Sn-0.7Cu + 1.0wt.% Si_3N_4.

Fabrication and Characterization of Hybrid Microwave Assisted Sintering Sn-0.7Cu + 1.0wt.% Si_3N_4 Composite Solder

ABSTRACT

One of the leading choices in upgrading the properties of existing lead-free solder alloys is by composite technology approach, whereby high technical ceramic particles can be added into the solder alloy matrix. Accordingly, Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder was synthesized using powder metallurgy routes which consist of blending, compaction and sintering. This research introduced a hybrid microwave assisted sintering process which can sinter ceramic-reinforced composite solder at approximately 185 ˚C within 2 minutes without holding time and protective inert gas. In order to evaluate the compatibility of hybrid microwave assisted sintering approach in ceramic-reinforced composite solder development, a detailed comparison of the process and properties of conventionally sintered and microwave sintered samples of Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder was performed. Identical sintering temperature at 185 ˚C was used for both types of sintering, in which conventional sintering was performed using a tube furnace in an argon atmosphere for 2 hours. The monolithic Sn-0.7Cu solder sample was also synthesized as control sample in a similar way. Hybrid microwave assisted sintering method showed significant advantages in processing compared to conventional sintering method, such as rapid heating rate, shortened sintering time, less energy consumption and much less expensive equipment. The influence of different sintering methodologies on Sn-0.7Cu + 1.0wt.% Si_3N_4 bulk solder sample were investigated based on the density, porosity, microhardness, microstructures, wettability and intermetallic compound thickness on Cu-substrate. It was noted that microwave sintering method can densify the Sn-0.7Cu + 1.0wt.% Si_3N_4 composite bulk solder green compact in a short time, however, conventional sintered sample showed better density and porosity. Interestingly, finer and well-distributed precipitates were observed in microwave sintered samples. This has led to higher microhardness performance observed in microwave sintered sample (12.0 ± 0.2 HV) compared to the conventionally sintered sample (11.2 ± 0.1 HV). The wettability performance of Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder on Cu-substrate was slightly reduced with microwave sintering approach, however, insignificant difference of intermetallic compound thickness was observed in both microwave sintered and conventionally sintered samples. Overall, hybrid microwave assisted sintering showed better processing with promising properties on ceramic-reinforced Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder.

CHAPTER 1

INTRODUCTION

1.1 Background

Over the past few years, the electronic packaging industry is constantly adapting to consumer demands for more advance electronic devices. According to Accenture (2013) annual report, the current trend of electronics products shows the consumers' purchase intentions are focused on multi-function devices that can offer convenient handling, such as smartphones, tablets, personal computers and high-definition televisions. As a consequence, there are increasing demands for higher interconnections count to be expected in a smaller electronic package. This has led to an urgent need for development of new solder interconnects that can provide high dimensional stability and reliability of joints during processing and usage. Lead-free solders and its soldering processing have been well established in the electronic packaging industry. Interconnect technology is, in fact, presently restructuring towards the second generation of lead-free solder materials (Schueller et al., 2010).

One of the possible choices in upgrading the existing lead-free solder alloys is by introducing secondary particles in the alloy matrix so as to form lead-free composite solder (Guo, 2007). Previous studies on composite solder as summarized by Liu et al. (2012) have shown some valuable results that proved composite solder can effectively enhance solder joint performance. Besides, the number of publications per year retrieved from the Scopus database also shows a clear rising interest within the research community in studying lead-free composite solder (see Figure 1.1). Whilst there has been a recent interest detected within the publications to combine lead-free composite solder and powder metallurgy (PM) method.

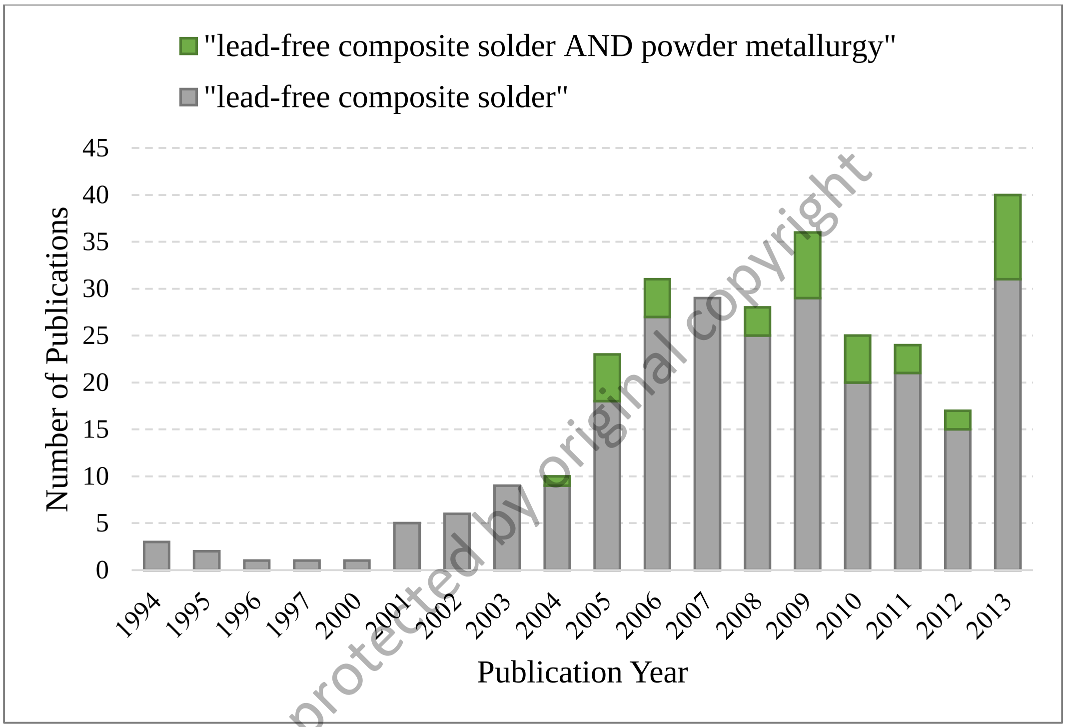

Figure 1.1: Number of publications retrieved from Scopus website database by year when using the search term “lead-free composite solder” and “lead-free composite solder AND powder metallurgy” in the Article Title, Abstract, and Keywords fields for all document types.

Accordingly, this study is carried out to explore the increasing research interest in composite solder using ceramic reinforcement materials through PM routes. Although the PM method was regularly utilized among the researchers to synthesis ceramic-reinforced composite solder (Mohd Salleh et al., 2013), the sintering step is still quite a challenge as it consumes significant amount of energy, time, and cost. Thus, there exists a great need for exploring advance heating method capable of providing a rapid heating rate for the sintering of ceramic-reinforced composite solder compacts.

Literature studies show microwaves can be utilized to sinter composite solder when in compacted powder form (Rybakov et al., 2013). Gupta and Wong (2005) have successfully produced an experimental setup that utilizes energy efficient two-directional microwave heating mechanism that is capable of sintering metal-based materials rapidly. The two-directional microwave assisted heating mechanism may also be called hybrid sintering. This is because the heating of the compacted powder come from two directions, i.e., direct heating within the compact materials and radiant heating from the microwave susceptor material arranged around the compact.

Further investigation has been carried out by Babaghorbani and Gupta (2008) to compare the effects of different sintering methodologies, i.e., conventional radiant sintering and the prior hybrid microwave assisted sintering setup initially developed by Gupta and Wong (2005) on the commercial lead-free Sn-3.5Ag solder alloy compacts. It was revealed that the tensile properties of Sn-3.5Ag samples were enhanced when using microwave assisted sintering method approach. The microwave sintered Sn-3.5Ag samples showed 21%, 17%, and 19% higher in 0.2% yield strength, ultimate tensile strength, and work of fracture, respectively, when compared to conventionally sintered samples.

In fact, ceramic-reinforced Sn-0.7Cu/Al_2O_3 and Sn-3.5Ag/SnO_2 composite solders have been successfully synthesized by Nai et al. (2010) using the same experimental hybrid microwave assisted sintering setup. It was found that the microwave technology can be a great assistant in a sintering process since it consumes much lower energy than conventional sintering with lesser heating time.

1.2 Problem Statements

Among the developed lead-free solders available in the market nowadays, eutectic Sn-0.7Cu solder alloy was widely used as the alternative standard for both lead- and silver-free solder alloys. However, eutectic Sn-0.7Cu solder alloy has a high tin (Sn) composition which can induce coarsening of phases within the solder matrix and the solder/substrate interfaces when imposed by thermal stress for a period of time. Moreover, eutectic Sn-0.7Cu solder alloy has low mechanical properties credibility since this alloy composition prone to brittle whisker intermetallic compound growth that can leads to serious reliability issues (Delserro, 2006). Recent studies attempted by various researchers to reincorporate small addition of non-reactive secondary particle such as Al_2O_3, ZrO_2, and Si_3N_4 (Zhong & Gupta, 2008a, 2008b; Mohd Salleh et al., 2012) to form composite solders have shown great enhancement in mechanical properties of the monolithic Sn-0.7Cu solder matrix. Hence, more efforts should be done on upgrading the promising lead-free Sn-0.7Cu alloy solder through composite technology.

In electronic packaging, lead-free solders are used to join a ceramic cap and a multilayer ceramic substrate in a multichip module. However, the solder joint suffers from a poor resistance to thermal fatigue since the monolithic solder alloy generally has much higher thermal expansion coefficient (CTE) than that of a ceramic (Tong, 2011). Since there is a need for a solder with a low CTE, the composite solder can be modified using specific ceramic reinforcement that possess low CTE properties. Among the various ceramic reinforcement materials that have been introduced in the lead-free composite solder development nowadays, the high technical ceramic materials such as Si_3N_4 can be a great candidate as a reinforcement material to enhance the monolithic Sn-0.7Cu solder matrix. Accordingly, Si_3N_4 is a light ceramic material with a low CTE and has been widely used for extreme engineering applications because of its excellent mechanical performance at both ambient and elevated temperatures (Trice & Halloran, 2000). Prior studies have simultaneously shown improvement in both solderability and mechanical performance in Sn-0.7Cu solder matrix by adding 1.0 weight percentage (wt.%) of micron-sized Si_3N_4 particles (Mohd Salleh et al., 2011, 2012). Realizing the capability of the Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder, more testing should be done on such composition to fully explore the composite solder potential.

The ideal way to reincorporate ceramic powder particulates into the monolithic solder matrix is by utilizing PM routes under solid state processing consist of mixing, compaction, and sintering. However, the sintering step consumes significant amount of energy, time, and cost. Regarding the sintering step, literature studies showed microwave technology can be utilized to sinter composite solder in compacted powder form. Among the primary advantages given by this method are microwave assisted sintering consumes much lower energy with lesser heating time compared with conventional sintering method. Usually, enhanced mechanical properties of ceramic-reinforced composite material can be obtained with finer microstructures distribution. While, fine microstructures distribution can be obtained through the manipulation of sintering temperature and time. Microwave technology can be a great assistant in a sintering process since the heat produced by this method is self-generated by the sintered material. Hence, its utilization in ceramic-reinforced lead-free composite solder is highly recommended.

The result of the literature search at the start of this present research revealed that no hybrid microwave assisted sintering work has been made to synthesize ceramic-reinforced Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder. Accordingly, the purpose of this research is to evaluate the possibilities of implementing hybrid microwave assisted sintering technique through powder metallurgy routes, particularly for ceramic-reinforced Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder production. The nature of the topic focuses on the impact of different sintering methodologies related to processing and properties of Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder, since comparisons of the sintering adaptation of powder metallurgy made with different sintering techniques are still lacking.

1.3 Aim and Objectives

The main aim of this research is to clarify any properties enhancement or advantages that hybrid microwave heating process can possibly offer if such attempt is preferred rather than conventional radiant heating on Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder. The research intends to achieve this aim through the following objectives:

i) To assemble and calibrate an experimental set-up capable of sintering solder compacts using both microwave heating and radiant heating simultaneously.

ii) To synthesize Sn-0.7Cu monolithic solder and Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder via powder metallurgy routes using different sintering methodologies, i.e., without sintering, with conventional sintering, and with hybrid microwave assisted sintering.

iii) To investigate the influence of different sintering methodologies on the density, porosity, microhardness, microstructure, and solderability performance of Sn-0.7Cu monolithic solder and Sn-0.7Cu + 1.0wt.% Si_3N_4 composite solder.