INVESTIGATIONS ON SILVER-COPPER NANOPASTE AS DIE-ATTACH MATERIAL FOR HIGH TEMPERATURE APPLICATIONS

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INVESTIGATIONS ON SILVER-COPPER NANOPASTE AS DIE-ATTACH MATERIAL FOR HIGH TEMPERATURE APPLICATIONS

by

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TABLE OF CONTENTS

CHAPTER 3: MATERIALS AND METHODOLOGY

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ACA	:	Anisotropic conductive adhesive
AFM	:	Atomic force microscopy
CHNS	:	Carbon-hydrogen-nitrogen-sulfur
CTE	:	Coefficient of thermal expansion
DBC	:	Direct bonded copper
DI	:	De-ionized
DSC	:	Differential scanning calorimetry
EEE	:	Electrical and electronic equipment
EF-TEM	:	Energy-filtered transmission electron microscopy
EG	:	Ethylene glycol
FE-SEM	:	Field-emission scanning electron microscopy
ICA	:	Isotropic conductive adhesive
ICSD	:	Inorganic crystal structure database
IMC	:	Intermetallic compound
MEA	:	More electric aircraft
MEMS	:	Micro-electro-mechanical system
MOS	:	Metal-oxide-semiconductor
MW	:	Molecular weight
PWC	:	Printed wiring board
RMS	:	Root-mean-square
RoHS	:	Restriction of hazardous substance
TGA	:	Thermo-gravimetric analysis
TMA	:	Thermo-mechanical analysis

UV-Vis	:	Ultraviolet-visible spectroscopy
WEEE	:	Waste of electrical and electronic equipment
XRD	:	X-ray diffraction

LIST OF SYMBOLS

α	:	Coefficient of thermal expansion
β	:	Shape constant for a Berkovich indentation tip
ε	:	Buildup strain
ρ	:	Bulk density
ρ_T	:	Theoretical density
λ	:	Thermal diffusivity
A	:	Contact area
C_{p1}	:	Specific heat of the sintered nanopaste sample
C_{p2}	:	Specific heat of the reference standard
E	:	Young's modulus
E_i	:	Young's modulus of the Berkovich indentation tip
E_r	:	Reduced Young's modulus for sintered nanopaste
H	:	Hardness
h	:	Indentation depth
h_c	:	penetration contact depth
h_{max}	:	Maximum indentation depth of the sintered nanopaste by an indenter
k	:	Thermal conductivity
ΔL	:	Change of the sample length
L_o	:	Original sample length
M	:	Performance index, the ratio of K/α
m_1	:	Mass of the sintered nanopaste sample
m_2	:	Mass of the reference standard
P	:	Applied load

P_{max}	:	Maximum applied load by an indenter
Q	:	Heat flow per unit area
S	:	Stiffness
T	:	Temperature
ΔT	:	Change of the temperature
T_h	:	Homologue temperature ratio
T_m	:	Melting temperature
T_o	:	Safety operational temperature
v	:	Poisson's ratio of sintered nanopaste
v_i	:	Poisson's ratio of the Berkovich indentation tip
W_d	:	Dry weight of sintered nanopaste sample
W_s	:	Weight of sintered nanopaste sample upon suspended in water
W_w	:	Weight of sintered nanopaste sample upon removed from water
wt%	:	Weight percent
x	:	Heat flow direction from die to substrate
y_1	:	Net heat flow of the sintered nanopaste sample
y_2	:	Net heat flow of the reference standard

LIST OF PUBLICATIONS

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3. K. S. Tan, Y. H. Wong and K. Y. Cheong, "Thermal Characteristic of Sintered Ag–Cu Nanopaste for High-Temperature Die-Attach Application", *International Journal of Thermal Sciences*, vol. 87, pp. 169-177, 2015.

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3. K. S. Tan and K. Y. Cheong, "Effect of Sintering Environment on Silver-Copper Die-Attach Nanopaste", *36th International Electronics Manufacturing Technology Conference (IEMT)*, Renaissance Johor Bahru Hotel, Johor, Malaysia. 11th-13th November 2014.

KAJIAN TERHADAP NANO-PES ARGENTUM-KUPRUM SEBAGAI BAHAN LAMPIR-DAI UNTUK APLIKASI SUHU TINGGI

ABSTRAK

Satu nano-pes argentum-kuprum (Ag-Cu) yang dirumuskan dengan mencampurkan nanopartikel Ag dan Cu dengan penambah organik (pelekat resin, terpineol dan ethylene glycol) telah dihasilkan bagi diaplikasikan sebagai bahan lampir-dai suhu tinggi. Pelbagai peratus berat nanopartikel Cu (20-80 wt%) telah ditambahkan ke dalam nano-pes Ag-Cu, diikuti oleh pensinteran di udara terbuka pada suhu 380 ℃ selama 30 min tanpa bantuan tekanan luar, untuk mengkaji kesan terhadap sifat-sifat fizikal, elektrikal, terma dan mekanikal. Nanopes tulen Ag dan Cu turut disediakan untuk tujuan perbandingan. Keputusan belauan sinar-X menunjukkan fasa $Ag_{97}Cu_3$, Ag_1Cu_{99} dan CuO terbentuk dalam nano-pes Ag-Cu tersinter. Kajian menunjukkan bahawa keliangan didalam nano-pes Ag-Cu tersinter meningkat dengan peningkatan kandungan Cu. Kehadiran keliangan tersebut membuktikan kesannya untuk mengurangkan ketumpatan, saiz bijian, keberaliran elektrik, keberaliran haba dan pekali pengembangan haba (CTE) bagi nano-pes Ag-Cu tersinter. Walaupun keliangan turut menjejaskan kekerasan, kekukuhan dan modulus Young nano-pes Ag-Cu tersinter, namun aliran meningkat telah direkodkan dengan penambahan kandungan Cu. Secara keseluruhan, nano-pes Ag-Cu dengan kandungan 20 wt% Cu menunjukan kombinasi terbaik bagi keberaliran elektrik [2.27×10^5 $(\Omega\text{-cm})^{-1}$] dan haba [159 W/m-K]. Nilai-nilai tersebut didapati lebih tinggi daripada kebanyakan sistem bahan lampir-dai. CTE yang rendah [13×10^{-6} / K] yang berkait dengan nano-pes Ag-Cu tersebut memanfaatkan disebabkan ia mengelakkan pembentukan tekanan haba serius di antara dai dan substrat. Selain itu,

nano-pes Ag-Cu telah menunjukkan suhu lebur 955 ℃, yang membolehkan nano-pes Ag-Cu dapat dipertimbangkan untuk diaplikasikan pada suhu tinggi. Bagi kajian sifat ikatan terhadap persalutan logam, salutan Ag and Au pada substrat Cu masing-masing telah menunjukkan kekuatan ikatan tertinggi (52.6 MPa) dan terendah (34.4 MPa) bagi nano-pes Ag-Cu. Nilai kekuatan ikatan didapati berkait rapat dengan mikrostruktur di antara nano-pes Ag-Cu dan lapisan salutan logam pada substrat. Akhir sekali, untuk mengaplikasikan nanopes Ag-Cu sebagai bahan lampir-dai pada suhu tinggi, nano-pes Ag-Cu telah digunakan untuk melampirkan dai silikon karbida (SiC) pada substrat yang disaluti oleh Ag atau Au. Keseluruhan struktur ikatan tersebut telah lulus ujian penuaan haba pada 770 ℃, mikrostruktur yang telah mengalami proses penuaan haba menunjukkan bahawa nano-pes Ag-Cu merekat dengan baik pada dai SiC dan substrat yang disaluti Ag. Namun, perekatan nano-pes tersebut adalah kurang memuaskan pada dai SiC dan substrat yang disaluti Au.

INVESTIGATIONS ON SILVER-COPPER NANOPASTE AS DIE-ATTACH MATERIAL FOR HIGH TEMPERATURE APPLICATIONS

ABSTRACT

A silver-copper (Ag-Cu) nanopaste formulated by mixing Ag and Cu nanoparticles with organic additives (i.e., resin binder, terpineol and ethylene glycol) which is meant for high-temperature die-attach applications has been developed. Various weight percent of Cu nanoparticles (20-80 wt%) has been loaded into the Ag-Cu nanopaste, followed by sintering in open air at temperature of 380 °C for 30 min without the need of applied external pressure. The physical, electrical, thermal and mechanical properties were investigated. Both pure Ag and Cu nanopastes were also prepared for comparison purposes. X-ray diffraction results showed that $Ag_{97}Cu_3$, Ag_1Cu_{99}, and CuO phases were formed in sintered Ag-Cu nanopaste. Studies revealed that the porosity of sintered Ag-Cu nanopaste increased with an increase of Cu loading, where the presence of porosity has shown its effect in decreasing of density, grain size, electrical conductivity, thermal conductivity and coefficient of thermal expansion (CTE). Although the porosity has also affected the hardness, stiffness and Young's modulus of sintered Ag-Cu nanopaste, yet an increasing trend has been recorded for aforementioned properties, with the increment of Cu loading. Overall, Ag-Cu nanopaste with 20 wt% of Cu loading has offered the best combination of electrical [2.27 x 10^5 $(\Omega\text{-cm})^{-1}$] and thermal conductivity [159 W/m-K], where these values are higher than most of the die-attach systems. The low CTE [13 x 10^{-6}/K] that associated with Ag-Cu nanopaste was good to prevent severe buildup of thermal stress between die and substrate. The Ag-Cu nanopaste has demonstrated a melting temperature of 955 °C, which enables it to be

considered for high-temperature applications. For metallization and bonding attribute studies, Ag and Au coatings on Cu substrate have displayed the highest (52.6 MPa) and the lowest (34.4 MPa) bonding strength for Ag-Cu nanopaste, respectively. The values of bonding strength were found to have a close relationship with the interface microstructure between Ag-Cu nanopaste and metallization layer on the substrate. Finally, to realize Ag-Cu nanopaste as a high-temperature die-attach material, the Ag-Cu nanopaste was used to attach a silicon carbide (SiC) die on a substrate with either Ag or Au coating. The entire bonding structure has passed a three-cycle thermal aging test at 770 ℃. The thermal-aged interface microstructure has shown that the Ag-Cu nanopaste was well adherence to SiC die and substrate with Ag coating, but poor adherence to SiC die and substrate with Au coating.

CHAPTER 1

INTRODUCTION

1.1 Theoretical background

The demand of electronic devices that could be operated at high-temperature (> 500 ℃) is continually increasing for years. This is mainly due to the advancement of technology in various industries such as automotive, aviation, well-logging, nuclear power plant and space exploration. These industries require electronic devices that must not only be able to survive upon expose to high-temperature, but they must also be able to function under such high-temperature condition. For instance, the typical high-temperature applications for those industries are: (i) brake and exhaust gas sensors for automotive (300-1000 ℃) (Johnson *et al.*, 2004; Spetz *et al.*, 1999), (ii) turbine and gas sensors for aviation (~600 ℃) (Dreike *et al.*, 1994; Hunter *et al.*, 2004; Sharp, 1999b), (iii) geothermal sensor for well-logging (~600 ℃) (Neudeck *et al.*, 2002; Sharp, 1999b; Watson and Castro, 2012), (iv) nuclear radiation detector and nuclear reactor for nuclear plant (700-1000 ℃) (Dreike *et al.*, 1994; Kim *et al.*, 2011; Sedlackova *et al.*, 2013), and (v) transmitter, antenna and electromechanical devices for space exploration (> 500 ℃) (Sutton, 2001). For such demanding applications, there is an evolution of electronic device, which transforming from silicon (Si)-based to silicon carbide (SiC)-based, due to the former could only operate at temperature up to 250 ℃.

SiC-based electronic device has documented its success to operate at a temperature exceeding 500 ℃. This is mainly attributed to the wide band gap semiconductor properties (3.26 eV) and high breakdown field strength (3.2 MV/cm) that associated with SiC semiconductor. These properties allow SiC semiconductor to be operated at high-temperature without leakage of current (Chin *et al.*, 2010). Nevertheless, to take full advantages of SiC-based electronic device, there is a need to develop an electronic packaging, which can use for high-temperature applications. The main development areas of electronic packaging include die-attach material, substrate material, wire bonding material, and encapsulation material. Of these, die-attach material has gained particular concern as it is an integral part that provides connection between the SiC device and the substrate.

Ideally, a die-attach material for SiC device should demonstrate a melting temperature that is higher than 500 °C, which allows it to be operated in a high-temperature environment. It should also demonstrate a low processing temperature, as well as ease to be applied for mass production. Besides, another four main properties required are: electrical and thermal conductivities, coefficient of thermal expansion (CTE), and bonding strength. These properties must display values that are comparable to or superior than the benchmark requirements that listed in Table 1.1.

Table 1.1: Benchmark requirements of various die-attach properties for SiC device (Abtew and Selvaduray, 2000; Bai *et al.*, 2006b; Chin *et al.*, 2010; Chung, 1995; Haque *et al.*, 2012; Lu *et al.*, 2004; Manikam and Cheong, 2011).

Property	Benchmark requirement
Melting temperature	> 500 °C
Electrical conductivity	$\geq 0.71 \times 10^5$ $(\Omega.cm)^{-1}$
Thermal conductivity	≥ 51 W/m-K

Table 1.1: Continued.

Property	Benchmark requirement
Bonding strength	≥ 12.5 MPa
Coefficient of thermal expansion	Close to the die and the substrate

1.2 Problem statement

Over the past decade, conductive adhesives (Gao *et al.*, 2014; Gomatam and Mittal, 2008; Lahokallio *et al.*, 2014; Li and Wong, 2006; Yim *et al.*, 2008) and tin (Sn)-based solders alloys (lead-bearing and lead-free) (Abtew and Selvaduray, 2000; Koo *et al.*, 2014; Kotadia *et al.*, 2014; Liu *et al.*, 2008; Wu *et al.*, 2004; Zeng *et al.*, 2012; Zeng and Tu, 2002; Zhang *et al.*, 2012) have been widely used for level-one interconnection, namely die-attach material, which serves to attach a semiconductor die on a substrate. The wide use of conductive adhesive and Sn based solder alloys are mainly due to the low cost and acceptable electrical conductivity [0.01-0.71 $x10^5$ $(\Omega\text{-cm})^{-1}$] and thermal conductivity [1-66 W/m-K] (Abtew and Selvaduray, 2000; Calame *et al.*, 2005; Gao *et al.*, 2014; Guan *et al.*, 2010; Kisiel and Szczepański, 2009; Kotadia *et al.*, 2014; Lewis and Coughlan, 2008; Navarro *et al.*, 2012; Suganuma *et al.*, 2009). However, with the recent development of SiC device that could be operated at temperature exceeding 500 ℃ (Manikam and Cheong, 2011), conductive adhesive and Sn based solder alloys that melt at a temperature below 315 ℃ (Abtew and Selvaduray, 2000; Kotadia *et al.*, 2014; Lahokallio *et al.*, 2014; Wu *et al.*, 2004; Zeng and Tu, 2002) can no longer meet the operating temperature requirement. The challenge is thus driven to seek a die-attach material that can be operated at temperature higher than 500 ℃.

Bismuth (Bi) (Kim *et al.*, 2014; Shi *et al.*, 2010; Song *et al.*, 2007a; Song *et al.*, 2006; Spinelli *et al.*, 2014; Wang *et al.*, 2014b), gold (Au) (Bazin *et al.*, 2014; Chidambaram *et al.*, 2012; Ding *et al.*, 2013; Huang *et al.*, 2013; Lau *et al.*, 2013; Zhu *et al.*, 2014), and zinc (Zn) (Haque *et al.*, 2012; Haque *et al.*, 2010; Kim *et al.*, 2009a; Shimizu *et al.*, 1999) based solder alloys are next being proposed as alternative solutions. Of these, Bi based solder alloys have generally displayed poor electrical conductivity [0.02-0.12 $x10^5$ $(\Omega\text{-cm})^{-1}$] (Kim *et al.*, 2014; Song *et al.*, 2007a; Song *et al.*, 2006), poor thermal conductivity [7-11 W/m-K] (Lalena *et al.*, 2002; Tschudin *et al.*, 2002) and moderate melting point [262-361 °C] (Lalena *et al.*, 2002; Spinelli *et al.*, 2014; Wang *et al.*, 2014b), which are inadequate to be considered as alternative solutions. Au and Zn based solder alloys, although, have displayed high thermal conductivity [27-110 W/m-K] (Bazin *et al.*, 2014; Kim *et al.*, 2009a; Kisiel and Szczepański, 2009; Suganuma *et al.*, 2009), their electrical conductivity [0.34-0.65 $x10^5$ $(\Omega\text{-cm})^{-1}$] (Bazin *et al.*, 2014; Lau *et al.*, 2013) and melting point [280-383 °C] (Bazin *et al.*, 2014; Kim *et al.*, 2008; Kim *et al.*, 2009c; Lau *et al.*, 2013; Lee *et al.*, 2005; Sheen *et al.*, 2002; Weng *et al.*, 2013) are still lower than the benchmark values (Table 1.1), making them failed to be considered as suitable die-attach materials for SiC device. Au-nickel (Ni), with its high melting point of 980 °C, is an exceptional solder alloy that meets the operating temperature requirement (> 500 °C) of SiC device, but its high soldering temperature at 980 °C has also become a drawback (Kirschman, 1999). Two new die-attachment techniques, namely inter-diffusion bonding of metal film and sintering of metal paste, are subsequently being introduced to overcome the weakness (i.e., high soldering temperature) that is associated with Au-Ni solder alloy. For instance, Au-indium (In) (Mustain *et al.*, 2010; Welch and Najafi, 2008) and silver (Ag)-In

(Chuang and Lee, 2002; Mustain *et al.*, 2010; Wu and Lee, 2013) are particular die-attach materials that utilized inter-diffusion bonding technique to form a joint between metal films at temperature of 206 to 210 ℃ with pressure of 40 to 80 psi. Meanwhile, Ag micropaste (Zhang and Lu, 2002) (i.e., a mixture of micro-sized metal particles and organic additives) and copper (Cu) micropaste (Kahler *et al.*, 2012b) are particular die-attach materials that formed a joint by sintering the micropaste at temperature of 250 ℃ with pressure of 40 MPa. Overall, the advantage of these die-attachment techniques is able to process at a moderate temperature (206-250 ℃), yet the joint formed could be operated at temperature exceeds 495 ℃ (Kahler *et al.*, 2012b; Mustain *et al.*, 2010; Welch and Najafi, 2008; Wu and Lee, 2013; Zhang and Lu, 2002). On the other hand, application of pressure during the process is one of the disadvantages of these die-attachment techniques, which could complicate the manufacturing process and with slight irregularities during application of pressure may lead to cracking of both the die and the substrate (Kahler *et al.*, 2012b; Mustain *et al.*, 2010; Welch and Najafi, 2008; Wu and Lee, 2013; Zhang and Lu, 2002).

In recent years, a strategy of reducing the size of metal particle in metal paste, from micron to nano, has been introduced, which it is named as nanopaste (i.e., a mixture of nano-sized metal particles and organic additives). The reduction of particle size aims to increase the chemical driving force of metal particle and thus contributes to eliminate the application of pressure during sintering. Ag nanopaste (Bai *et al.*, 2007a; Bai *et al.*, 2007b; Bai *et al.*, 2005; Bai *et al.*, 2006b; Chen *et al.*, 2008; Lu *et al.*, 2014; Lu *et al.*, 2009; Mei *et al.*, 2011b, 2011c; Yu *et al.*, 2009; Zheng *et al.*, 2014) and Cu nanopaste (Krishnan *et al.*, 2012; Nishikawa *et al.*, 2011;

Yamakawa *et al.*, 2013) are the leading candidates of this strategy, where they could be sintered at temperature of 280-400 ℃ without the need of applying any pressure during sintering. Positive results were obtained for these sintered nanopastes, namely: (i) no existent of die-shifting issue as the nanopaste does not undergo liquid-state transformation during sintering (Bai *et al.*, 2007a; Bai *et al.*, 2007b; Krishnan *et al.*, 2012); (ii) high electrical [2.50-2.60 x10^5 (Ω-cm)$^{-1}$] and thermal conductivity [200-240 W/m-K] (Bai *et al.*, 2005; Bai *et al.*, 2006b; Lu *et al.*, 2004; Mei *et al.*, 2012; Zheng *et al.*, 2014); (iii) high bonding strength [2-54 MPa] could be attained with atomic inter-diffusion between the nanopaste and the metallization layer on a die or substrate (Bai *et al.*, 2007b; Nishikawa *et al.*, 2011; Yamakawa *et al.*, 2013); (iv) lower Young's modulus was detected for sintered nanopaste as compared to bulk materials and solder alloys; this is important to reduce the build-up of thermal stress among the die, die-attach and substrate in an operating device (Bai *et al.*, 2007b; Bai *et al.*, 2005; Bai *et al.*, 2006b; Mei *et al.*, 2012; Zheng *et al.*, 2014) and (v) high melting point at 960-1083 ℃ has meet the operating temperature requirement of a SiC device (> 500 ℃) (Bai *et al.*, 2007b; Kahler *et al.*, 2012b; Lu *et al.*, 2004; Lu *et al.*, 2014; Mei *et al.*, 2011c; Zheng *et al.*, 2014). Despite that, both Ag nanopaste and Cu nanopaste are actually having their own limitations, where Ag nanopaste has limited to its high cost and low electrochemical migration resistance (Lu *et al.*, 2014; Mei *et al.*, 2011a); whereas Cu nanopaste is easy to oxidize. To overcome the oxidation issue, additional time (1h) is needed to anneal the Cu nanopaste in nitrogen environment (Krishnan *et al.*, 2012; Yamakawa *et al.*, 2013).

For these reasons, an Ag-aluminum (Al) nanopaste (Manikam *et al.*, 2012; Manikam *et al.*, 2013c) is introduced, which aimed at surpassing the preceding limitations of Ag nanopaste and Cu nanopaste. This Ag-Al nanopaste not only could tailor the cost to be cheaper than that of Ag nanopaste, it also able to be sintered at 380 ℃ in air atmosphere without the need of additional annealing process in nitrogen environment. Ag-Al nanopaste has displayed electrical conductivity [1.01 $x10^5$ $(\Omega\text{-cm})^{-1}$] that is better than Sn, Bi, Au and Zn solder alloys [0.02-0.71 $x10^5$ $(\Omega\text{-cm})^{-1}$], but it is still worse than Cu micropaste [1.29 $x10^5$ $(\Omega\text{-cm})^{-1}$], Ag micropaste [4.17 $x10^5$ $(\Omega\text{-cm})^{-1}$] and Ag nanopaste [2.50-2.60 $x10^5$ $(\Omega\text{-cm})^{-1}$] (Bai *et al.*, 2005; Bai *et al.*, 2006b; Kahler *et al.*, 2012b; Manikam *et al.*, 2012; Zhang and Lu, 2002; Zheng *et al.*, 2014). Furthermore, Ag-Al nanopaste has also displayed thermal conductivity [123 W/m-K] that is better than Sn, Au, Bi and Zn solder alloys [7-110 W/m-K], but it is still worse than Ag micropaste [80-220 W/m-K] and Ag nanopaste [200-240 W/m-K] (Bai *et al.*, 2005; Bai *et al.*, 2006b; Kahler *et al.*, 2012b; Manikam *et al.*, 2012; Zhang and Lu, 2002; Zheng *et al.*, 2014).

Based on preceding facts, Ag-Cu nanopaste is introduced, which aimed to overcome the weakness that associated with Ag-Al nanopaste. Cu was chosen to replace Al in a nanopaste formulation because it has the second best electrical and thermal conductivities among other metals (Callister, 2007), and it has coefficient of thermal expansion that is comparable with Ag (Table 1.2), making it suitable to be used with Ag in a nanopaste formulation. Moreover, the price of Cu is comparable to that of Al ("Current pricing on precious, platinum, non ferrous, minor and rare earth metals," 2013), which is able to meet the cost constraint in electronic packaging. Although Cu and Al are ductile materials, Cu has higher tensile strength than Al (Table 1.2) (Callister, 2007), where higher bonding strength is predicted for Ag-Cu nanopaste if compared with Ag-Al nanopaste (Manikam *et al.*, 2013c;

Morisada *et al.*, 2010; Yan *et al.*, 2012). Based on galvanic series, Cu has standard electrode potential that is close to Ag if compared with Al to Ag, which minimize the tendency of two metals, i.e., Ag and Cu, to interact galvanically, and thus reduces the risk of galvanic corrosion (Chawla and Gupta, 1993). Ultimately, Cu has a melting temperature that is drastically higher than that of Al; this might make the melting temperature of Ag-Cu nanopaste become drastically higher than that of Ag-Al nanopaste.

Table 1.2: Properties of bulk Ag, Cu, Au and Al.

Property	Ag	Cu	Au	Al	Ref
Electrical conductivity [x 10^5 (Ω-cm)$^{-1}$]	6.80	6.00	4.30	3.80	(Callister, 2007)
Thermal conductivity [W/m-K]	428	398	315	247	(Callister, 2007)
Coefficient of thermal expansion [x 10^{-6} /K]	19.7	17.0	14.2	23.6	(Callister, 2007)
Tensile strength [MPa]	170	200	130	90	(Callister, 2007)
Young's modulus [GPa]	74	110	77	69	(Callister, 2007)
Ductility [% elongation]	44	45	45	40	(Callister, 2007)
Melting point [℃]	962	1085	1064	660	(Callister, 2007)
Price on March 2013 [$ US / kg]	1012.36	7.61	56717.06	1.89	("Current pricing on precious, platinum, non ferrous, minor and rare earth metals," 2013)
Standard electrode potential [V]	+0.800	+0.340	+1.420	-1.662	(Callister, 2007)

In this work, the Ag-Cu nanopaste is formulated by mixing Ag and Cu nanoparticles with organic additives. This nanopaste can be sintered at 380 ℃ in open air without the need of applying external pressure. The study covered the detailed investigation of the physical, electrical, thermal and mechanical properties of Ag-Cu nanopaste with various Cu loadings, as these properties are crucial for die-attach applications. Further investigations were also carried out to assess the workability of Ag-Cu nanopaste as a die-attach material for SiC device, which is mainly for high-temperature applications.

1.3 Research objectives

The primary aim of this research is to formulate an Ag-Cu nanopaste that can be used for high-temperature die-attach applications, yet it can be processed at a low-temperature. Various physical, electrical, thermal and mechanical properties of Ag-Cu nanopaste were systematically investigated, which include density, porosity, electrical conductivity, thermal conductivity, coefficient of thermal expansion, melting temperature, hardness, Young's modulus and bonding strength. These properties must be properly investigated in order to demonstrate the suitability of Ag-Cu nanopaste as a die-attach material for high-temperature applications (Table 1.1). With this primary aim in mind, the following objectives are to be achieved:

1. To formulate an Ag-Cu nanopaste by mixing metallic nanoparticle and organic additives, and determine its optimum sintering temperature and environment.

2. To investigate the physical, electrical and thermal characteristics of Ag-Cu nanopaste with various Cu loadings.
3. To investigate the mechanical properties of Ag-Cu nanopaste and its bonding attributes on different metallization layers.
4. To apply Ag-Cu nanopaste for attaching SiC die on Cu substrate and aluminium nitride direct bonded Cu substrate.

1.4 Scope of study

In this research work, Ag-Cu nanopaste was first formulated by mixing Ag and Cu nanoparticles with various loadings of organic additives. The rheology of nanopaste was next analyzed to determine an optimized formula for Ag-Cu nanopaste. Various sintering temperatures and environments were used to sinter the nanopaste which was aimed to obtain an optimized sintering condition. The research was next continued to investigate the physical, electrical, thermal and mechanical properties of Ag-Cu nanopaste with various weight percent of Cu loadings. The Ag-Cu nanopaste with optimized properties was selected for further investigation on its bonding attribute on different metallization coatings. Finally, the workability of Ag-Cu nanopaste as a high-temperature die-attach material has been investigated, where it was used to attach a SiC die on either Cu substrate or aluminium nitride direct bonded Cu substrate. The entire bonding structure has undergone a thermal aging test, followed by a cross-section failure analysis.

Various characterization techniques have been used in this work, where they are classified into physical, electrical, thermal and mechanical characterizations. For physical characterization, rheometer was used to reveal the viscosity of nanopaste. Field emission scanning electron microscope (FE-SEM) and atomic force microscope (AFM) were used to characterize the surface morphology and topography of sintered Ag-Cu nanopaste. X-ray diffraction (XRD) was used to identify the phases, and co-linear four point probe system was used to measure the electrical conductivity of sintered nanopaste. For thermal characterization, differential scanning calorimetry (DSC) was used to determine the melting temperature of raw Ag and Cu nanoparticles. It also used to determine the melting temperature and specific heat of sintered Ag-Cu nanopaste. Besides, thermo-gravimetric analysis (TGA) was used to determine the burn off temperature of organic additives used in Ag-Cu nanopaste. The thermal diffusivity and thermal expansion attributes of sintered nanopaste were measured by using nanoflash laser and thermo-mechanical analysis (TMA) systems, respectively. As for mechanical characterization, nanoindentation technique was used to determine the hardness, stiffness and Young's modulus of sintered Ag-Cu nanopaste; whilst, lap shear test has been performed by using Instron universal testing machine to obtain the bonding strength of Ag-Cu nanopaste. The lap shear test was also performed on Cu substrate with various metallization coatings in order to understand the bonding attributes of Ag-Cu nanopaste.

1.5 Thesis outline

This thesis is organized and divided into 5 chapters. Chapter 1 provides an overview of high-temperature electronic packaging, followed by the issues and challenges faced in the development of high-temperature die-attach material, research objectives, and scope of study. Chapter 2 covers the detailed literature review, which corresponds to the background theories adopted in the study. Chapter 3 presents the systematic methodology that was employed in this research. Chapter 4 focuses on the results and discussion from the characterizations. Finally, Chapter 5 summarizes the overall findings of this study and concluded with appropriate recommendation for future works.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In recent years, electronic devices are continually improving for high-temperature applications, mainly due to the increasing demand from various industries such as automotive, aviation, well-logging, nuclear power plant and space exploration (Chin *et al.*, 2010). These electronic devices are fabricated by using a wide band-gap semiconductor, namely silicon carbide (SiC), which aim at overcoming the limitation of low operating temperature (< 250 ℃) that exhibited by conventional silicon (Si)-based electronic device (Chin *et al.*, 2010). The current research trend is thereby targeted to develop electronic packaging that is in line with the SiC-based electronic device, which is able to operate at high-temperature. This chapter begins by reviewing the evolution of electronic device from Si-based to SiC-based, followed by their applications. The chapter will next cover an overview of electronic packaging and the materials used for high-temperature applications. Since this research is focused on developing a die-attach material, the basic requirements of a die-attach material will be discussed. Next, the detailed literatures for high-temperature die-attach materials will be systematically covered. Finally, this chapter will review the factors affecting the mechanical, electrical, and thermal properties of die-attach material.

2.2 Definition of high-temperature for electronic device

"High-temperature" is a term that subject to various interpretations, where it can be defined as a temperature that is greatly higher than a typical standard operating temperature (Chin *et al.*, 2010). For instance, automotive, well logging and space exploration industries have defined the term of "high-temperature" as an operating temperature at beyond 125 °C (Johnson *et al.*, 2004), 300 °C (Palmer and Heckman, 1978) and 500 °C (Hagler *et al.*, 2011), respectively. Hence, it is inadequate to define the term of "high-temperature" in accordance to respective industry. The definition must be determined from a group of variety industries, followed by taking into considerations the operating temperature of various electronic devices in the group. Manikam and Cheong (2011) are the leading researchers who proposed three ranges of temperature based on a group of variety industries. "High-temperature" is defined as a range of temperature that operates at beyond 500 °C; whilst, "medium-temperature" and "low-temperature" are defined as another ranges of temperature that operate at 300-500 °C and < 300 °C, respectively (Manikam and Cheong, 2011). In this thesis, these three ranges of temperature will be used; whereby "high-temperature" is fixed at a temperature that higher than 500 °C.

2.3 Evolution of semiconductor in electronic device

Over the past decade, Si has emerged as the most widely used semiconductor materials in electronic devices. This is mainly due to its interesting attributes, such as (i) able to be produced in a large defect-free single crystal, (ii) able to grow a stable native oxide layer (SiO_2) that possesses superior dielectric properties, (iii) has appropriate hardness that allows large wafer can be handled by either hand or machine, (iv) able to be doped with small amount of impurities (e.g. phosphorus or boron), which formed either n-type or p-type semiconductor, and (v) relatively cheap in cost because of its relatively abundance in the earth crust (Chante *et al.*, 1998; Harper, 2003). However, with advances in technology for recent years, there is a demand of electronic devices that could be operated at high-temperature (≥ 500 ℃) and harsh environment. The electronic devices that based on Si semiconductor, with a maximum operating temperature of 250 ℃, have become no longer meet the requirement of high operating temperature. The low operating temperature (≤ 250°C) of Si semiconductor is actually attributed by its narrow band-gap (1.12 eV), in which leakage of electric current happens if it is operated at temperature beyond 250 ℃ (Chante *et al.*, 1998). As a result, it is crucial to seek a semiconductor material that is capable to operate at temperature beyond 250 ℃.

In recent years, SiC semiconductor, with its large band-gap (3.26 eV), has identified as a promising candidate to overcome the limitation of Si semiconductor in an electronic device, due to its operating temperature is drastically improved to 400 ℃ and above. Besides that, SiC semiconductor could also display a few advantages of Si semiconductor, such as (i) able to produce high quality single crystal with low defect, (ii) able to grow native oxide of SiO_2, and (iii) able to selectively dope of either n-type or p-type (Friedrichs and Rupp, 2005). These advantages are also contributing to make SiC becomes an interesting alternative semiconductor material for high-temperature applications. Table 2.1 provides a comparison of semiconductor properties between Si and SiC. It can be seen that the

SiC with large band-gap has offered a high breakdown electric field (3.2 MV/cm), which is approximately ten times higher than that of Si (0.3 MV/cm) with narrow band-gap. High breakdown electric field allows the SiC to be operated at a high-temperature without leakage of current if compared to Si. The thermal conductivity of SiC (3.7 W/cm-K) is approximately two times higher than that of Si (1.5 W/cm-K); this is good for heat dissipation in an operating electronic device.

Table 2.1: A comparison of semiconductor properties between Si and SiC (Chelnokov and Syrkin, 1997; Chin *et al.*, 2010; Zolper, 1998).

Property	Si	SiC
Band-gap (eV)	1.12	3.26
Dielectric constant	11.80	9.66
Breakdown electric field (MV/cm)	0.3	3.2
Thermal conductivity (W/cm-K)	1.5	3.7
Saturated electron velocity (cm/s)	1×10^7	2×10^7
Electron mobility (cm^2/Vs)	1400	1000
Hole mobility (cm^2/Vs)	600	115
Melting point (℃)	1417	2827
Physical stability	Good	Excellent
Process maturity	Very high	High

In addition, the saturated electron velocity of SiC (2×10^7 cm/s) is also two times higher than that of Si (1×10^7 cm/s); this indicates that SiC can be operated at much faster speed if compared to Si. A SiC semiconductor is actually made up of Si and C atoms that held by a strong bond. The Si-C bond, in fact, is stronger than Si-Si bond that contains within a Si semiconductor. This strong bond provides better physical and chemical stabilities over the SiC semiconductor, which allows it to be operated in a high-temperature and harsh environment (Chin *et al.*, 2010).

2.3.1 Demands of high-temperature electronic device

Nowadays, consumer electronic products, such as personal computer, cell phone, television and washing machine, have become an integral part in our daily lives. These products are commonly made up of Si-based electronic device, where it is continuously strike to decrease its feature size, increase its operating speed, and reduce its power consumption. Normally, the consumer electronic products are designed to be operated at temperature below 200 ℃; thereby Si-based electronic device is sufficient to meet the requirements of those products.

On the other hand, the demands of industrial electronic components, such as radiation and pressure sensors, are slightly varied to those consumer electronic products. The industries are seeking for electronic components that are capable to be operated at high-temperature, high-power and harsh environment. The Si-based electronic device, with its low operating temperature (< 250 ℃), is therefore no longer fulfill the requirements of industry electronic components. SiC-based electronic device, with its high operating temperature (> 400 ℃), is next emerging as a promising candidate to overcome the limitations of conventional Si-based electronic device. For instance, the hydrocarbon sensor that made up of SiC has proven able to operate at temperature up to about 800 ℃ (Shields, 1996).

Over the years, the demand of high-temperature electronic devices has shown a steady growth due to the continuous technology advances in various industries. Oil and gas industry, in particular, is one of the leading industries that have high demand on the high-temperature electronic devices. This industry requires a lot of fairly sophisticated sensors that to be installed in vicinity to the drilling head (Chin *et al.*, 2010). During a well drilling operation, the sensors are used to monitor the health of drilling head, as well as, used to measure the drilling depth as a function of temperature. This is due to the temperature variation in earth crust, which can be ranging up to 600 ℃ for the deepest drilling depth that can be attained by current drilling technology (Chin *et al.*, 2010; Sharp, 1999b). For well logging process (down-hole measurement), the sensors are used to acquire the down-hole information, such as surrounding geologic formation and saturation of hydrocarbon (oil and gas) (Watson and Castro, 2012). This information is important to determine the amount of hydrocarbon that can be extracted from the well. Finally, during the hydrocarbon extraction process, the sensors and electronic systems are used to monitor the pressure, temperature, vibration, and flow rate of hydrocarbon; this is to ensure an optimized productivity from the well, while also prevents any catastrophic disaster (Chin *et al.*, 2010; Sharp, 1999b; Watson and Castro, 2012).

Aviation is another industry that requires a large volume of high-temperature electronic devices, which arise from the main goal that moving towards the "more electric aircraft" (MEA) (Reinhardt and Marciniak, 1996; Santini *et al.*, 2013; Watson and Castro, 2012). Traditional commercial aircraft is operated with a centralized control system, which involved large amounts of complex wiring, piping and connector interfaces to transmit the signal and power from the central electronic controller to the mechanical, hydraulic and pneumatic systems that located in an aircraft (Santini *et al.*, 2013). In line with the target of MEA, distributed control system is being introduced to replace the centralized control system in an aircraft, where the electronic controllers are placed near to the engines (Watson and Castro, 2012). This system offers five main advantages: (i) it reduces the complexity of wiring interconnections, thereby reducing the maintenance complexity and cost; (ii) it reduces the amount of long and heavy wiring and piping systems, thereby saving the weight of an aircraft; (iii) it increases the control reliability because of a number reduction in connector pins; (iv) it increases the survivability of an aircraft since malfunction of certain electronic controllers still can allow an aircraft landing safely; and (v) it provides better fuel efficiency and increases performance of an aircraft (Reinhardt and Marciniak, 1996; Watson and Castro, 2012). The trade off, however, is the electronic controller needs to be operated at high-temperature environment that is close proximity to the engine. For instance, the electronic controller that monitors rotational speed of turbine disk in an aircraft engine, it has to withstand an elevated temperature up to 600 ℃ (Nieberding and Powell, 1982). Another example is the electronic controller and sensor that used for combustion emission monitoring; they need to operate to the temperature ranging up to 800 ℃ (Hunter *et al.*, 2004; Sharp, 1999b).

The automotive industry is a fairly substantial market, which requires large quantities of high-temperature electronic devices. This is due to the evolution of the automotive industry that is transforming from mechanical and hydraulic systems to an electromechanical system (Huque *et al.*, 2008). The evolution is mainly aimed to improve fuel efficiency and reduce emissions of an automobile. Consequently, more sensors and signal-conditioning components are being installed into an automobile in order to precisely control the valve timing (Chin *et al.*, 2010; Sharp, 1999a). Nowadays, an advanced automobile contains approximately 100 sensors, where these sensors are used to monitor the health of engine, angular position and speed, automatic brake system, power steering, and exhaust system (Fleming, 2001; Sharp, 1999a). Those sensors are being installed in various locations of an automobile, in which the operating temperature is also varied according to the install locations, as shown in Table 2.2. For instance, the sensors for automatic braking and exhaust systems are working under an ambient temperature of 300 ℃ and 1000 ℃, respectively (Sharp, 1999a; Spetz *et al.*, 1999).

Table 2.2: Automotive maximum operating temperatures (Johnson *et al.*, 2004; Spetz *et al.*, 1999).

Sensors install locations	Maximum operating temperature (℃)
On-engine	150-200
In-transmission	150-200
On wheel-automatic brake system	150-300
Cylinder pressure	200-300
Exhaust system	Up to 1000

Space exploration is a niche market of electronic devices, but its operating temperature and environment is rather high and harsh. Starting from the launch of the space shuttle, the sensors are used to monitor the combustion of hydrocarbon at a temperature up to 1000 ℃ and above, as well as, used to detect any leakage of hydrocarbon (Sutton, 2001). This is to ensure a safe journey from the earth to the outer space. For Venus planet exploration, the surface temperature of this planet is ranging between 460 ℃ and 480 ℃, with a surface pressure of 92 bars, carbon dioxide and nitrogen atmospheres, and sulfuric acid cloud coverage at a distance of 50 km from the surface (Cressler and Mantoot, 2013). The electronic devices that have landed on this planet must be able to withstand those harsh conditions first, followed by executing the given missions on this planet.

On the other hand, high-temperature electronic devices are also being used in other industrial applications, including nuclear power plant and industrial production machine. The nuclear power plant utilized a lot of sophisticated sensors that are made up of SiC semiconductor. These sensors are being used wisely to detect the neutron formation and its liberated radiation at a temperature of 700 ℃ (Kim *et al.*, 2011; Sedlackova *et al.*, 2013). Besides, the sensors are also being installed in surrounding the storage tanks of nuclear waste, which function to detect any leakage of radioactive and hazardous substances, at a temperature of up to perhaps 150 ℃ (Dreike *et al.*, 1994). All of these applications could be beneficial to prevent any possibility of catastrophic disaster happens to the nearby citizens. For industrial production applications, such as ammonia production plant, the sensor is being used to monitor the synthesized concentration of ammonia, where it must able to withstand a high temperature, up to 500 ℃, that to be applied in the production process (Timmer *et al.*, 2005). Other industrial applications include temperature, pressure, flame indicator, and ultraviolet radiation sensors, which operate at a medium- to high-temperature, ranging between 450 ℃ and 1050 ℃ (Casady and Johnson, 1996; Shields, 1996). In summary, the applications of high-temperature electronic device are a large variety in accordance with the industrial requirements. Although the market of industrial electronic components is not as large as consumer electronic products, but its market has steadily grown in recent years, and therefore the market cannot be ignored too.

2.4 An overview of electronic packaging

High-temperature electronic device has been realized with the development of SiC semiconductor technology. Nevertheless, the electronic device cannot be worked without a proper packaging. Therefore, the packaging, or more specifically termed as electronic packaging, must be developed in order to connect the electronic device to other components of electronic package, namely substrate, heat sink, printed wiring board and power source (Tummala, 2001). Figure 2.1 illustrates a three-level hierarchy of electronic packaging. At first-level of electronic packaging, also known as device level packaging, an electronic device or chip is to be connected to a package that serves as protecting, powering and cooling mediums. This level of packaging also functions to provide signal transmission from a chip to the package, or vice versa, where electrical conductive pads on both the chip and the package are to be connected via wire bonding. The first-level electronic package is next interconnected to a second-level electronic package, which is typically a printed wiring board (PWB). It is because a single device or chip does not generally form into a system, as a typical system requires a number of different types of active and passive devices that to be assembled and interconnected via a printed wiring board. The motherboard is next used to connect several pieces of printed wiring boards and functions to provide an integration of entire system; this is typically referred to as third-level of electronic packaging (Tummala, 2001).

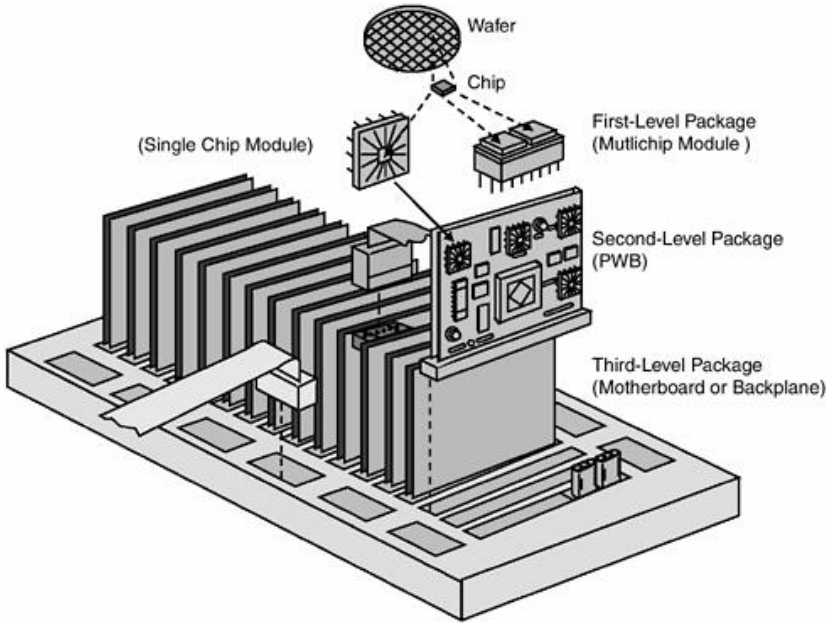

Figure 2.1: Hierarchy of electronic packaging (Tummala, 2001).

2.5 Materials for level-one electronic packaging

Currently, the development of high-temperature electronic device has focused on two broad areas, which are semiconductor device fabrication and its packaging. A substantial research effort has been spent on the development of electronic device that based on SiC semiconductor which aims for high-temperature applications. This is owned to its intrinsic wide band gap semiconductor with excellent physical and mechanical properties. Various electronic devices have been successfully produced by SiC semiconductor, such as the metal-oxide-semiconductor (MOS) field effect device that is designed for gas sensor applications (Soo *et al.*, 2010). The SiC-based sensor has displayed a high sensitivity and selectivity for sensing a variety of gases, namely hydrogen, oxygen, and hydrocarbon. Table 2.3 provides a summary of various gases that can be sensed by current SiC-based gas sensors, in which the operating temperature of those sensors can be ranged up to 1000 ℃.

Table 2.3: A summary of various gases sensed by SiC-based gas sensors.

Gas sensed by SiC sensor	Maximum operating temperature (℃)	Reference
H_2	150-800	(Ghosh and Tobias, 2005)
O_2	300-800	(Ghosh and Tobias, 2005)
CO	650	(Baranzahi *et al.*, 1997)
H_2S	325	(Weng *et al.*, 2008)
CH_4	350	(Soo *et al.*, 2010)
C_2H_6	650	(Baranzahi *et al.*, 1995)
C_3H_6	350-700	(Kandasamy *et al.*, 2005)
C_4H_{10}	650	(Baranzahi *et al.*, 1995)
C_xH_y	500-1000	(Werner and Fahrner, 2001)

Although the SiC-based electronic devices, such as gas sensors, have been well designed for high-temperature applications, but these devices cannot be worked without a proper electronic packaging that acts to transmit the signal from a device to other computerized systems, as well as, to provide a protection for the device in against of its surrounding harsh environments (Kirschman, 1999). For device level packaging (level-one of electronic packaging), there are four main areas need to be concerned for high-temperature applications, namely die-attach material, substrate material, wire bonding material, and encapsulation material. These electronic packaging materials must be properly designed, where their properties must also be properly addressed in fulfillment of the requirements for high-temperature applications. In this thesis, the primary focus is on die-attach material that serves to attach an electronic device or die on a substrate. The die-attach material is actually an integral part of electronic package, as it acts as an interface layer between a die