High Electrical and Thermal Conductivity of Nano-Ag Paste for Power Electronic Applications

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Abstract

The nano-Ag paste consisted of Ag nanoparticles and organic solvents. These organics would be removed by evaporation or decomposition during sintering. When the sintering temperature was 300 °C, the resistivity of sintered bulk was $8.35 \times 10^{-6} \Omega \text{ cm}$, and its thermal conductivity was $247 \text{ W m}^{-1} \text{ K}^{-1}$. The Si/SiC chips and direct bonding copper (DBC) substrates could be bonded by this nano-Ag paste at low temperature. The bonding interface, sintered microstructure and shear strength of Si/SiC chip attachment were investigated by scanning electron microscopy, transmission electron microscopy and shear tests. Results showed that the sintered Ag layer was porous structure and tightly adhered to the electroless nickel immersion gold surface of DBC substrate and formed the continuous Ag–Au interdiffusion layer. The shear strength of Si and SiC chip attachments was higher than 35 MPa when the sintering pressure was 10 MPa. The fracture occurred inside the sintered Ag layer, and the fracture surface had obvious plastic deformation.

Keywords Nano-Ag paste · Sintering process · Interfacial microstructure · Chip attachment · Shear strength

1 Introduction

The operating temperature of power electronics in hybrid automotive, aerospace, space exploration, deep oil and gas exploration can reach beyond 300 °C [1, 2]. Silicon carbide (SiC) has the excellent high-temperature performance and been identified as one of the potential semiconductor wafers for the future generation of power electronics [3, 4].

However, traditional die-attach materials, such as Pb-based and Sn-based solders, exhibit the poor bonding reliability at high temperature and low thermal and electrical conductivity [5–7]. Many researches have recently been directed toward developing high-temperature materials for

power electronics, which can be used to attach chips and have high thermal and electrical conductivity [8, 9].

Nano-Ag paste has been studied as a possible alternative to Pb-free solder [10, 11]. High surface energy of nanoparticles and consequent strong capillary force make it possible to achieve strong bonding [12–15]. After sintering, the melting point of sintered Ag returns to the melting point of bulk Ag so that the sintered chip attachment could operate at high temperature.

Currently, nano-Ag paste could achieve bonding chips and substrates pressureless, which avoid chip breaking and shorten processing time [16–19]. Nano-Ag paste contains 10–20 wt% organics to ensure the screen printing performance. Although substantial organics removal is a prerequisite for nano-Ag sintering, the residual organics in the sintered layer are inevitable, especially for large-size chip attachment [20]. The reason should be that the paste required the addition of long-chain binders when chip size was large, while long-chain binders required higher temperature to decompose [21]. Residual organics not only reduced the thermal conductivity of sintered layer, but also caused defects such as unconnected regions and voids [22]. More seriously, the sintered chip attachment was prone to produce hot spots during high-temperature application and the chip/substrate plating oxidation failure [23]. How to avoid

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organic residue in the sintered layer is an inevitable choice to improve the thermal conductivity and reliability of sintered chip attachment.

In order to overcome this problem, we prepared the novel nano-Ag paste. The Ag nanoparticles were mixed with many kinds of low-boiling-point organics, which make sure that these organics could be removed by evaporation or decomposition to avoid residual in the sintered layer. In addition, these organics in the paste had the characteristics of gradient decomposition, which could guide the design of assisted pressure sintering profile and prevent from squeezing. The electrical resistivity and thermal conductivity of paste were measured. Si/SiC chips were sintered on the direct bonding copper (DBC) substrate at 300 °C under the low assisted pressure. The bonding interfacial microstructure was investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and the shear strength and fracture surface were also measured and observed.

2 Experimental

2.1 Preparation of Nano-Ag Paste

Ag nanoparticles were purchased from Beijing Tsing lab Co., Ltd. The shape was spherical and quasi-spherical with an average particle diameter of 110 nm. In order to prevent from aggregating, polyvinyl pyrrolidone (PVP) capped Ag nanoparticles and formed the organic shell structure, and the coating thickness was about 2 nm (Fig. 1a). The content of Ag was 98.6 wt% (Fig. 1b).

The nano-Ag paste contained Ag nanoparticles and organic solvents. In order to obtain the excellent printability, organic solvents included dispersant, binder and thinner [24]. The content of organics was about 12.5 wt%, and the weight percentage of the individual organic was decided by

Refs. [25–28]. Polyethylene glycol (analytical grade) was used to adjust viscosity, and polyvinyl alcohol (analytical grade) was used to prevent cracking during sintering.

The thermal properties of nano-Ag paste were characterized by combination of thermogravimetry (TG) and differential scanning calorimetry (DSC, STA449F3, Netzsch). The temperature ranged from 50 to 600 °C in air, and the heating rate was 10 °C/min. The density of the sintered sample was measured by an automatic density machine (AccuPyc II 1340). Microstructure of nano-Ag paste was characterized by scanning electron microscopy (SEM, Merlin Compact, Zeiss).

2.2 Thermal Behavior and Electrical Property

To identify the microstructural change of paste during sintering, nano-Ag paste was deposited onto a glass slide with approximately 100 µm thick and sintered at 200–300 °C for 5–60 min. Microstructural change was observed by SEM. Thermal diffusion coefficient of the sintered sample was measured by a laser flash thermal conductivity analyzer (LFA447, Netzsch) and a Fourier-transform infrared spectroscopy (FTIR) spectrometer. In order to prepare a standard sintered sample, the paste was filled into a mold with a diameter of 13 mm and then sintered at 300 °C for 15 min under 3 MPa in air. After cooling, the sintered sample was ground to a standard size sample having a diameter of 12.7 mm. The thickness of sintered sample was 0.5 mm. Three samples were measured to obtain the thermal diffusion coefficient in order to avoid the influence of cracks. Porosity and pore size of the sintered Ag layer were calculated from cross-sectional SEM images. The total cross-sectional area of layer (A) and the pore area (A_p) were measured. Porosity (f) was obtained from the equation $f = \Sigma A_p / A$ [23].

Fig. 1 **a** SEM image of Ag nanoparticles and the thickness of PVP coating; **b** TGA–DSC curve of Ag nanoparticles heated in air at 10 °C/min

The resistivity of nano-Ag paste was measured by four-probe method. The thickness of printing paste was about 100 μm . The calculation formula was as follows [29]:

$$\rho = (V_{23}/I_{14})(\pi/\ln 2)Wf_4(a), \quad (1)$$

where V is the voltage, I is the current, W is the thickness of sintered sample, a represents the relative thickness and $a = W/S$. S is the probe spacing, and $S = S_1 = S_2 = S_3$. $f_4(a)$ is the correction function of printing paste thickness.

2.3 Sintering Process by Nano-Ag Paste

The size of Si and SiC chip was 2.5 mm $\times$ 2.5 mm and 1.85 mm $\times$ 1.85 mm, and their thickness was 300 μm and 400 μm , respectively. Back metallization of chips was the sputtered Ag layer with 0.7 μm . The substrate was a direct bonding copper (DBC) alumina ceramic, and the size was 14 mm $\times$ 14 mm. The thickness of copper was 300 μm , and the thickness of alumina ceramic was 635 μm . Top metallization of DBC substrate was Ni(P) (5 μm)/Au (70 nm), namely electroless nickel immersion gold (ENIG) surface. The ENIG layer contained approximately 10 at.% phosphorus (P). A stencil in 100 μm thickness was used to print nano-Ag paste on the DBC substrate, and chip attachment process could be completed the following steps: print, dry, pick and place, and sinter. Figure 2a shows the image of chip attachment. Sintering equipment was a self-made sintering machine. The actual sintered layer structure was sputtered Ag layer/sintered Ag layer/Au layer. The shear strength of chip attachment was measured by a Dage 2400 shear tester with a 100 kg (Fig. 2b). Five sintered chip attachments were tested for each experimental condition and obtained the average shear strength. The fracture surface of chip attachment was investigated by SEM.

Because the sintered Ag layer of chip attachment was porous structure, an ion milling machine (Leica, EM TIC 3X) was used to polish the samples to obtain the clear and accurate cross-sectional microstructure. Interfacial microstructure between the sintered Ag layer and DBC substrate was investigated using transmission electron microscopy (TEM, JEOL JEM-2100). TEM samples were cut out using a focused-ion-beam apparatus (FEI Helios G4 CX).

3 Results and Discussion

3.1 Physical Properties of the Nano-Ag Paste

3.1.1 Thermal Decomposition Characteristics of Nano-Ag Paste

Figure 3a shows the TG-DSC curve of nano-Ag paste. An exothermic peak was detected at around 313 $^{\circ}\text{C}$ in the DSC curve, and a great weight loss occurred in the paste. The Ag content in the paste was 87 wt%. The nano-Ag paste had the characteristics of gradient decomposition, which could guide the design of sintering profile to obtain the high-quality chip attachment.

Thermal decomposition of paste could be divided into three stages. Stage I (< 150 $^{\circ}\text{C}$): the low-boiling-point organics, such as ethylene glycol and water, began to decompose in this stage, and the paste weight was reduced by 4.3%. The decomposition of organics reduced the fluidity of paste, which prevented the chip from being buried and shorted by the squeezed paste. Stage II (150–250 $^{\circ}\text{C}$): the large molecular weight organics, such as polyethylene glycol, gradually began to decompose, and the paste weight decreased by 1.4% and remained stable for a certain period of time. The paste was further cured in this stage. Stage III (250–300 $^{\circ}\text{C}$):

Fig. 2 **a** Image of chip attachment sintered by nano-Ag paste; **b** measurement of shear strength of chip attachment

Fig. 3 **a** Thermal decomposition of nano-Ag paste (heating rate 10 °C/min, air atmosphere); **b** FTIR spectra of the nano-Ag paste sintered at different temperatures

Fig. 4 Resistivity of nano-Ag paste sintered at different temperatures for 15 min

the polyvinyl alcohol was decomposed, and the paste weight was reduced by 7.3%.

The disappearance of C–O–C (stretching, 1109 cm⁻¹; antisymmetric stretching, 1370 cm⁻¹) peaks in the FTIR spectrum of paste after sintering at different temperatures demonstrated that the polyethylene glycol was gradually evaporated and decomposed [30], as shown in Fig. 3b. The band observed at 2862 cm⁻¹ indicated an asymmetry in stretching mode of CH₂ group [31]. The disappearance of these peaks with increasing temperature should correspond to the decomposition of polyvinyl alcohol.

Table 1 Comparison of resistivity of nano-Ag paste reported by different companies

Silver particle paste	Sintering parameters	Resistivity (Ω cm)
Heraeus' mAgic paste	280 °C/–	8.00E–05
DOWA silver nano-paste	180 °C/90 min	5.00E–05
NBE's nano-paste	280 °C/–	3.80E–05
Harima's NH4000 paste	250 °C/60 min	2.50E–06
Harima's NPS-HB paste	150 °C/90 min	1.40E–05
ALPHA Argomax® paste	190–300 °C/–	2.50E–05
This study	275 °C/15 min	2.31E–05
	300 °C/15 min	8.35E–06

3.1.2 Electrical Property of Nano-Ag Paste

Electrical resistivity is a critical parameter for die-attach materials and related to the current handling capability and electrical power loss [32]. Figure 4 shows the resistivity of nano-Ag paste after sintering at different temperatures. The sintered microstructures directly affected the electrical property of paste. As the sintering temperature increased, organics in the paste were gradually decomposed and sintered microstructure became dense, which improved the electrical property of the sintered bulk. When the temperature reached 300 °C, the resistivity of sintered bulk was 8.35×10^{-6} Ω cm. Compared with the reported resistivity of Ag paste (Table 1), the resistivity of paste prepared in this paper was lower, which made them a potentially excellent die-attach material with high electrical performance, and could meet the requirement of power electronics [33].

Table 2 Thermal conductivity calculation method of the sintered Ag layer [34, 36, 37]

Calculation method	Equation	Parameters
Wiedemann–Franz (W–F) calculation	$\lambda = LT\sigma$	L : proportionality constant $2.44 \times 10^{-8} \text{ W } \Omega \text{ K}^{-2}$ σ : electrical conductivity T : temperature
Thermal diffusivity calculation	$\lambda = \alpha C \rho$	α : thermal diffusivity C : specific heat ρ : sample bulk density
Porosity calculation	$\lambda = \lambda_0(1-\xi)^{3/2}$	ξ : the porosity λ_0 : the thermal conductivity of solid material

Fig. 5 Thermal conductivity of Ag paste measured by different evaluation methods

3.1.3 Thermal Property of the Nano-Ag Paste

Thermal conductivity is also an important consideration for die-attach materials and significantly affects the reliability and lifetime of chip attachment. However, the measurement of thermal conductivity for the porous sintered Ag layer is a challenging technology. Currently, there are three methods, as listed in Table 2. The most common method is to measure the thermal diffusivity of the paste (α , $\text{mm}^2 \text{s}^{-1}$) by laser flash thermal conductivity analyzer, and thermal conductivity (λ , $\text{W m}^{-1} \text{K}^{-1}$) could be calculated by the equation [34]:

$$\lambda = \alpha C \rho \quad (2)$$

where C is the specific heat capacity in J m^{-1} , and ρ is the density in g cm^{-3} . In this study, the average thermal diffusivity was $82.259 \text{ mm}^2 \text{s}^{-1}$ and the density was 8.6 g cm^{-3} after sintering at 300°C , and the specific heat capacity was

$0.35 \text{ J g}^{-1} \text{K}^{-1}$ [34]. The thermal conductivity of nano-Ag paste was $247 \text{ W m}^{-1} \text{K}^{-1}$. It should be noted that the porosity had the significant influence on the thermal conductivity, and this value was obtained when the porosity of the sintered sample was 19%.

Figure 5 shows the thermal conductivity of sintered Ag paste obtained by three methods. Lu et al. [35] compared the thermal conductivity of various die-attach materials. It was found that the thermal conductivity of Pb–Sn solder was $51 \text{ W m}^{-1} \text{K}^{-1}$, high-Pb solder was $70 \text{ W m}^{-1} \text{K}^{-1}$ and Au–Sn solder was $58 \text{ W m}^{-1} \text{K}^{-1}$. The high thermal conductivity of sintered Ag was a guarantee of the thermal properties of chip attachment and could meet the thermal management requirement of power electronics.

3.1.4 Sintering Behavior of nano-Ag Paste

Figure 6 shows the surface morphology change of nano-Ag paste sintered at different temperatures in air. When the paste was sintered in a furnace at 200°C , nanoparticles agglomerated together and many sintered necks formed. While the size of sintered neck was small, the particles connected by sintered necks were distributed in an island shape. When the temperature reached 250°C , the sintered necks grew up and pores gradually shrank into the closed voids, and the characteristics of nanoparticles disappeared. As the sintering temperature increased, these voids gradually shrank in size and became spherical. When temperature reached 300°C , the entire sintered bulk became relatively dense, and the number of closed voids sintered was remarkably reduced. Figure 7 shows the effect of sintering temperature on the porosity. The sintered microstructure gradually became dense, and the porosity decreased with the increasing in sintering temperature. This implied that the mass transfer between the nanoparticles occurred, and the sintering temperature had the significant effect on the change in surface morphology of nano-Ag paste.

Fig. 6 Surface morphology evolution of the nano-Ag paste with different sintering temperatures (sintering time 15 min, in air)

Fig. 7 Influence of sintering temperature on the porosity of the sintered Ag

The sintering temperature was selected at 300 °C, and the effect of sintering time on the surface morphology change is shown in Fig. 8. When the sintering time was 5 min, a large number of particles formed the sintered necks. While the sintering time extended to 20 min, the morphology of paste did not change obviously and densification behavior became slow, which indicated that the dense sintered layer had been realized. Figure 9 shows the effect of sintering time on the porosity. The porosity of the sintered microstructure gradually decreased with the increasing in sintering time. For power electronics, it is desirable to obtain a dense and crack-free sintered Ag layer to ensure electrical and thermal conduction. From the effects of sintering temperature and time on the surface morphology, the sintered Ag microstructure could form the numbers of the sintered necks at ≤ 300 °C and meet the requirement of power electronics. In addition, the sintered layer had less dependence

Fig. 8 Surface morphology evolution of the nano-Ag paste with different sintering time (sintering temperature 300 °C, in air)

Fig. 9 Influence of sintering time on the porosity of the sintered Ag

on the sintering time, and the rapid chip attachment process could be realized by this paste in short time.

3.2 Chip attachment sintered by nano-Ag paste

3.2.1 Microstructure of the chip attachment

Figure 10 shows the cross-sectional microstructure of the chip attachment. The sintered Ag layer of the chip attachment was porous structure. The excellent bonding interfaces could be observed among the sintered Ag layer, SiC chip and ENIG surface substrate. At the SiC chip/sintered Ag layer interface, metallurgical bonding was formed between the sintered Ag layer and the sputtered Ag layer. At the sintered Ag layer/ENIG surface interface, the Ag nanoparticles were completely spread on the ENIG surface and formed a dense sintered Ag layer with a thickness of about 0.5 μm . The average porosity of sintered Ag layer was about 19%. Ag, Au, Ni

Fig. 10 Cross-sectional microstructures of SiC chip attachment: **a** overview of chip attachment; **b** SiC chip/sintered Ag layer interface; **c** sintered Ag layer; **d** sintered Ag layer/ENIG surface

Fig. 11 Elemental distribution of cross-sectional chip attachment: **a** overview of chip attachment; **b** Ni; **c** Ag; **d** Au; **e** P; **f** Cu

and P distributions were clearly separated at the interface, which indicated that little Ag diffused into the Ni(P) layer, as shown in Fig. 11.

Bonding interface between Ag and ENIG surface was investigated using TEM, and the FIB sample position was close to ENIG surface side (Fig. 10d). Figure 12 shows the TEM images of cross-sectional SiC chip attachment. The Ag particles sintered together and formed the amount of twins in the sintered layer (Fig. 12b). The sintered Ag layer tightly adhered to the ENIG surface of DBC substrate, and high-resolution TEM image showed that no voids were observed at the interface. The sintered Ag layer/ENIG surface consisted of the sintered Ag layer/Ag–Au interdiffusion layer/Ni(P) layer, as shown in Fig. 12c. Ag and Au could form complete solid solutions due to the same crystal structure, and atoms easily diffused each other without considerable lattice distortion. Ag atoms diffused across the whole immersion Au layer and formed the continuous Ag–Au interdiffusion layer with about 50 nm thickness (Fig. 12d, f). The interdiffusion behaviors between the sintered Ag layer and the Au layer in the ENIG surface resulted in the formation of good metallurgical sinter bonding [38]. HRTEM image shows the obvious bonding interface of Au/Ni(P) (Fig. 12e), and the result indicated that Ag and Au atoms did not diffuse into the Ni(P) layer, which acted as the Ag and Au diffusion barrier due to the amorphous structure [39].

3.2.2 Shear Strength of Chip Attachment

Figure 13 shows the shear strength of the sintered Si/SiC chip attachments under different sintering pressures. When the pressure did not apply, the average shear strength of Si and SiC chip attachment was 14 MPa and 10 MPa, respectively. When the sintering pressure was 3 MPa, the average shear strength of both attachments exceeded 27 MPa. Further increasing the sintering pressure, the shear strength increased slowly. When the sintering pressure was 10 MPa, the strength of SiC chip attachment reached 35 MPa and that of Si chip attachment reached 45 MPa. Therefore, with the increasing in sintering pressure, the shear strength increased gradually. It should be noted that the SiC chip attachments have the higher strength and thickness than Si chip attachment although the chips had the same surface finish metallization. The possible reason should be that the Si chip was easier to break than the SiC chip during shearing (Fig. 14a, c), which resulted in the difference of chip attachment strength. It was this reason that the shear strength error bar of Si chip attachments became high as the sintering temperature increasing.

The applied sintering pressure prompted the density of sintered Ag layer and decreased the porosity, which improved the shear strength of chip attachment [40]. During the pressure-assisted sintering, the applied pressure

Fig. 12 TEM images of the sintered Ag layer/ENIG surface interface: **a** low-magnification image; **b–d** high-magnification image; **e** high-resolution TEM image of Ag/Ni(P) interface; **f** high-resolution TEM image of sintered Ag layer/Ag–Au interdiffusion layer interface

was finally transferred to the contact interface among particles and substrate; thus, the local stress at these contact points was significantly higher than the stress in the pressureless sintering (Table 3). High sintering pressure was considered to be helpful for the solid diffusion at the bonding interfaces [41].

Figure 14 shows the fracture surface of Si/SiC chip attachments. Because the sintered Ag still covered the entire bonding area both on Si/SiC chip and DBC substrate, it proved that fracture occurred inside the sintered Ag layer. Nano-Ag paste could wet the chip and ENIG surface fully during sintering, which provided a necessary

condition for the formation of reliable bonding interface [48].

For the Si chip attachment, some residual Si chip remained on the fracture surface of DBC side (Fig. 14a). The sintered Ag layer could be clearly observed, and its fracture surface had obvious plastic deformation, in support of the metallurgical bonding explanation (Fig. 14b, d).

Recently, a series of new Ag pastes are developed based on the nano-characteristics. Some micron-sized particles were added to the nano-Ag paste, which decreased the initial porosity compared with the single size paste [49]. Chen et al. [50] prepared the paste with only micron-scaled Ag flake particles and found that the best die shear strength could reach 33.9 MPa at 250 °C without pressure in air.

Fig. 13 Effects of the sintering pressure on shear strength of chip attachment (sintering temperature 300 °C, sintering time 15 min)

The mixed-size, even full micro-size paste could reduce the sintering conditions and cost. This will be the mainstream trend of nano-Ag paste in the future.

4 Conclusions

In this work, the nano-Ag paste consisted of Ag nanoparticles and organic solvents. The peak decomposition temperature of paste was 313 °C, and Ag content was 87 wt %. The sintering temperature and densification processes directly affected the electrical and thermal properties of paste. When the sintering temperature reached 300 °C, the resistivity of sintered bulk was $8.35 \times 10^{-6} \Omega \text{ cm}$, and the thermal conductivity was $247 \text{ W m}^{-1} \text{ K}^{-1}$. In addition, the sintering temperature had significant effect on the surface morphology of paste.

This nano-Ag paste could bond chips and DBC substrates at low temperature. The sintered Ag layer was porous structure and tightly adhered to the ENIG surface of DBC substrate. Ag atoms diffused across Au layer and formed the continuous Ag–Au interdiffusion layer with about 50 nm thickness. The average shear strength of chip attachment was higher than 35 MPa when the sintering pressure was 10 MPa. The fracture occurred inside the sintered Ag layer, and the fracture surface had the obvious

Fig. 14 Fracture morphology of sintered **a–b** Si chip attachment and **c–d** SiC chip attachment (sintering temperature 300 °C, sintering time 15 min, pressure 3 MPa)

Table 3 Shear strength of chip attachment sintered by Ag paste at 300 °C

Chip attachment	Sintering parameter	Shear strength(MPa)	Refs.
Die-to-DBC (Ni/Ag surface)	300 °C/20 MPa	20	[42]
Cu-to-Cu joint (Ag surface)	300 °C/0 MPa	22	[18]
Cu-to-Cu joint (Ag surface)	300 °C/5 MPa	50	
SiC-to-AlN joint (Cu surface)	300 °C/30 min	41	[28]
SiC-to-Si ₃ N ₄ joint (Ag surface)	300 °C/0.4 MPa/60 min	21	[43]
Cu-to-Cu disk joint	300 °C/5 MPa/5 min	36	[44]
Cu-to-Cu disk joints	300 °C/2.5 MPa	15.5	[45]
Cu-to-Cu disk joints	300 °C/10 MPa	55	[27]
Bare Cu joint	300 °C/60 min/0 MPa	20.7	[46]
SiC-to-DBC joint (Au surface)	300 °C/10 min	15	[47]
Si-to-DBC joint (Ni/Au surface)	300 °C/10 MPa/15 min	45	This work

plastic deformation, indicating that the chip attachment achieved the metallurgical bonding.

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