Influence of Ag particle shape on mechanical and thermal properties of TIM joints

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Abstract

Purpose – The purpose of this paper is to develop and test the thermal interface materials (TIM) for application in assembly of semiconductor chips to package. Good adhesion properties (>5 MPa shear strength) and low thermal interface resistance (better than for SAC solders) are the goal of this research.

Design/methodology/approach – Mechanical and thermal properties of TIM joints between gold plated contacts of chip and substrate were investigated. Sintering technique based on Ag pastes was applied for purpose of this study. Performance properties were assessed by shear force tests and thermal measurements. Scanning electron microscopy was used for microstructural observations of cross-section of formed joints.

Findings – It was concluded that the best properties are achieved for pastes containing spherical Ag particles of dozens of micrometer size with flake shaped Ag particles of few micrometers size. Sintering temperature at 230°C and application of 1 MPa force on the chip during sintering gave the higher adhesion and the lowest thermal interface resistance.

Originality/value – The new material based on Ag paste containing mixtures of Ag particles of different size (form nanometer to dozens of microns) and shape (spherical, flake) suspended in resin was proposed. Joints prepared using sintering technique and Ag pastes at 230°C with applied pressure shows better mechanical and thermal than other TIM materials such as thermal grease, thermal gel or thermally conductive adhesive. Those material could enable electronic device operation at temperatures above 200°C, currently unavailable for Si-based power electronics.

Keywords Nano-Ag paste, Assembly, Silver sintering, Micro-Ag paste mixture

Paper type Research paper

Introduction

The trend towards miniaturization of electronic devices and the growing market share of power components require a re-analysis of the cooling processes of electronic equipment (Fan et al., 2020; Fenech et al., 2020; Hansson et al., 2016; Narumanchi et al., 2008; Wenzhong, 2017). Overheating of the electronic takes a big part in the device reliability. Using classical analysis of cooling of the semiconductor device assembled as a part of electronic equipment one can distinguish two critical points. The first point is located at the interface of the semiconductor chip assembled to package. Other critical cooling point can be found between power device and radiator (Figure 1). Because of the miniaturization of the electronic devices, bigger challenge is to dissipate heat from the semiconductor chip to the package. There is a need to investigate new kinds of thermal interface materials (TIM): one for semiconductor chip assembly (TIM1) and other for power

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Microelectronics International © Emerald Publishing Limited [ISSN 1356-5362] [DOI 10.1108/MI-06-2022-0108] device to radiator assembly (TIM2). The TIM1 material need to be highly thermally conductive; moreover, this material must provide a very good adhesion and excellent electrical conductivity. Due to the fact that chip size is getting smaller and smaller, the critical issue is to manufacture TIM1 that not only has high thermal conductivity but also makes good thermal contact with both semiconductor chip and the package. For typical power devices, as a TIM1 material, high-Pb or Au-Sn solders are used (Kim et al., 2021b). Recently, due to environmental factors packaging research and applications go towards lead-free solders (SAC solders) taking into consideration their limitations in higher temperature operation. There is also study on the silver powder sintering technique for assembly of power devices (Chen and Siow, 2021; Paknejad and Mannan, 2017; Yeom et al., 2018). This is especially interesting for devices based on gallium nitride or silicon carbide (SiC) semiconductors and their possibility of application in high power and temperature applications. Those

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Figure 1 Scheme of electronic device mounted in package and on radiator; location of TIM1 and TIM2 is marked



material could enable electronic device operation at temperatures above 200°C, currently unavailable for Si-based power electronics.

The usefulness of TIM depends not only on the thermal conductivity of the TIM material but also on its bond line thickness (BLT) and ability to create a good interface with metallization of the substrate and the chip (Kim et al., 2021a; Villacarlos and Pulutan, 2020). The surface of joint is also factor in TIM properties; therefore, for assessment of the TIM, there is a need for thermal interface resistance (TIR) factor to be introduced. The TIR includes not only parameters of TIM material but also area of surface joint, quality of the interfaces and BLT. The best values of TIR below 5 mm²K/W are obtained for solders (Hansson et al., 2016). The solders itself have the thermal conductivity in the range of 40–80 W/mK. The chip to the substrate joint is formed by the diffusion process at the interface - this ensures low TIR values. For the wide range of TIM materials, thermal grease, thermal gel or thermally conductive adhesive typical TIR is in the rage form 10 up to 200 mm²K/W, when BLT is 20–200 μ m. One can deduce that reduction of BLT is easy solution to obtain lower TIR value. The measurements and computer simulations show that the best properties of Ag-based TIM are achieved for BLT in the range from 20 up to $50 \,\mu m$ (Villacarlos and Pulutan, 2020). Additionally, lower BLT triggers higher principial stress and strain on the die which may cause adhesive or cohesive failure. Increase of BLT not only reduces stress in joint layer but also increases thermal resistance of the TIM. Typical thickness of Ag-based TIM is lower than $60 \,\mu\text{m}$. The main challenge in Ag powders or solder joints in TIM formation is voids formation in joint layer (Ruifen et al., 2018). The porosity of the layers based on Ag powders depends not only on the presence of resins and/or solvent but also on the particle size of the Ag powder. Powders that contain more nano-Ag particles than micro-Ag particles after sintering are more prone for voids formation (Ruifen et al., 2018). In the paper Chen et al. (2020), it was shown that TIM based on micron/submicron Ag particles for SiC-TEG chip to DBC substrate (Cu/Si₃N₄/Cu) have 0.50-0.52 K/W thermal resistance. When high-Pb solder was used (Pb5Sn), thermal resistance was significantly higher 0.65-0.67 K/W. Additionally, cyclic electrical stress was also performed (2A, 110V) to study its influence on the thermal resistance. Joints manufactured with micron/submicron Ag pastes after 20,000 electrical stress cycles had same thermal resistance as in the beginning of the test. For the Pb5Sn joint only after 8,800 stress cycles, sample temperature raised above

290°C and thermal resistance was too high. In the paper Kim *et al.* (2021b), analysis of the pastes with micro-Ag powder for assembly of the SiC dummy chips to the DBC substrate with ENEPIG metallization is presented. It was concluded that those TIM joint do not lose their adhesive properties after 1000 h ageing in 250°C. All the cited studies conclude that further investigation of Ag powder pastes for TIM1 application in semiconductor chip to metallic substrate assembly is possible. In this paper, authors investigate the Ag powder grain size and shape on the mechanical and thermal properties of TIM1. The investigated TIM1 is applied between the gold Si chip contact and the gold package substrate.

Experiments and test methodology

The Ag-based pastes used in this study are mixtures of powders of specific particle size and viscosity suspended in resin. The Ag powders and pastes based on those powders are manufactured by Amepox Microelectronics, Łódź, Poland. Table 1 presents types of pastes. Pastes have different shape and size of the Ag particles with constant ratio of Ag powder phase (90 Wgt.%) to resin (10 Wgt.%).

TIM AT paste is a mixture of only Ag flake size particles size $3-7 \mu m$ in resin. Paste TIM 3 is a mixture of Ag nano particles and Ag flake particles $(1-3 \mu m$, smaller than those in TIM AT) in resin. TIM AT2M composition is based on spherical Ag particles size dozens of micrometers mixed with the same Ag flakes as in the TIM3 paste.

Properties of the pastes were assessed based on adhesion between Si chips (size: $3 \times 3 \times 0.52$ mm) with Ti/Au bottom mounting metallization and Cu substrate with Ni/Au (3μ m/1 μ m) metallization. Therefore, TIM1 joint is formed between Au metallization on the Si chip and Au on the substrate.

Adhesion of the Si chips was tested in shear test, according to the IPC-TM650 standard. The shear force was measured with 0.1 N accuracy, and shear area was calculated with 0.1 mm² accuracy. For adhesion test, sample was placed vertically in the vice holder. Then shear tool was placed below the chip. After making sure that the shear tool is placed correctly, the force was applied by shear tool on the chip until chip was removed from substrate. Peak force was registered, and shear strength was calculated. The IPC-TM650 standard specifies that the minimal adhesion of the chip should be higher than 5 MPa.

Measurements of the thermal properties of the TIM1 needs to prepare special samples and design and manufacture dedicated measurement setup (Figure 2; Burzo and Li, 2018; Fenech *et al.*, 2020; Fosnot and Galloway, 2015; Kisiel *et al.*, 2021). The special sample for thermal measurements consists

 Table 1
 Investigated composition of Ag pastes

Paste ref.	Ag powder composition	Resistivity
TIM AT	Ag flake size 3–7 μ m	$1.2 imes 10^{-4} \Omega cm$
TIM 3	Nano powder Ag size 160 nm (60 Wgt.%) + Ag flake 1– 3 μ m (40 Wgt.%)	$2.2\times10^{-4}\Omega cm$
TIM AT2M	Ag sphere size dozens of micrometers (60 Wgt.%) + Ag flake 1–3 μ m (40 Wgt.%)	$3.08\times 10^{-4}\Omega cm$

Figure 2 Scheme of designed measurement setup for thermal resistance measurements



of two Cu sheets single side galvanically covered by Ni/Au joined together with investigated paste (TIM1). The comparison method for thermal measurements was used. First dummy, pure Cu sample (0.93 mm) with graphite foil on both sides, was put in measurement setup. The role of graphite foil is to stabilize thermal resistance at both interfaces of the dummy sample and the measurement setup. Knowing dimensions and temperature distribution along measurement column and Cu thermal conductance in function of temperature, it is possible to calculate thermal resistance between T₃ and T₄ planes (Figure 2). Subtracting thermal resistance of dummy Cu sample form the total result gives internal thermal resistance of measurement setup. After those preparation measured sample, both sides covered by graphite foil, was placed in the measurement column. Again, thermal resistance between T₃ and T₄ planes was measured. Now, internal thermal resistance of measurement setup is subtracted from thermal resistance measurement. The result consists of following thermal resistances:

- two Cu sheets with Ni/Au metallization;
- paste; and
- two interfaces paste-Au.

Next, subtracting known thermal resistance two sheets Cu, desired value of thermal resistance is calculated. Additionally, knowing the area of the joint, it is possible to calculate TIR of investigated pastes. To have reference to the Ag paste measurements, a sample was joined using SAC solder (peak 250°C, 20 s), and its thermal properties was measured. Gentle pressure was applied when preparing reference sample to ensure similar joint thickness of SAC solder and Ag paste. For all the Ag pastes same two stage curing process was applied: heating rate 4°C/min, 160°C for 30 min + 230°C for 60 min (Myśliwiec *et al.*, 2022).

Measurements of performance parameters of examined pastes

Thermal interface materials at paste.

TIM AT paste is a mixture of Ag flake shape $3-7 \mu m$ particles and resin. It has the lowest resistivity of all the examined pastes.

For mechanical test, TIM AT was applied on a substrate by pin transfer method (five dots per chip, one dot = 0.2 mg). Preparing sample for thermal study, few dots of paste was applied evenly on the sample surface (substrate of Cu with NiAu metallization). Four Si chips were placed on each substrate. Each chip was pressed into the paste with 0.3 MPa pressure. Then sintering process was performed. For pressureless version, no pressure was applied on the chip during sintering. For pressure sintering DBC ceramic $(10 \times 10 \text{ mm})$ was placed on all four chips for even pressure distribution on all four chips. Pressure of 1 or 2.4 MPa was applied during whole sintering process. During pressure sintering process, the tool that applied pressure was also heated to sintering temperature. In Table 2, results of adhesion measurements for TIM AT paste are presented; those are results of testing four samples. Thickness of joint was in the range of $30 - 40 \,\mu\text{m}$. There was a clear dependence of adhesion on the applied pressure during sintering process. The repeatability of the adhesion results is better for samples sintered without pressure than for samples with applied pressure. In both cases, adhesion is at the limit of acceptance for this test (>5 MPa). Observation of the surface after the shear test indicates a cohesive crack. Independently from adhesion, study samples for thermal investigation were prepared (Table 3). For thermal study, two samples for each experiment were prepared in the manner described in Experiments and Test Methodology paragraph. There is dependance observed of thermal resistance and applied pressure. Samples sintered without pressure have three times as high thermal resistance as samples sintered with 1 MPa pressure. Moreover, sample where no pressure was applied have significantly bigger joint thickness. Application of pressure during sintering process causes Ag flakes to concentrate more, that forms paths for better heat transfer (Figure 3). It is worth noting that the thermal resistances of samples of similar thickness: SAC solder, and TIM AT paste are comparable, even though a thin layer of resin is observed at the interface of the Ag-paste and Au metallization.

Thermal interface materials 3 paste

In the literature there are reports that addition of Ag nanoparticles can improve performance parameters of TIM joints (Chua and Siow, 2016; Paknejad and Mannan, 2017). For testing this idea TIM3 paste was developed using 60 Wgt.% of Ag nanoparticles and only 40 Wgt.% of flake shape $1-3 \mu m$ Ag microparticles. Samples for investigation of TIM 3 paste adhesion and thermal parameters were prepared in the same way as in TIM AT paste. In Table 4, results of adhesion measurements are presented – four samples were used. Table 5 contains results of thermal properties of TIM3 paste are characterized by worse adhesion and higher thermal resistance than TIM AT samples with similar thickness. Explanation of this results is that with same joint thickness in TIM 3 and TIM AT, there is a difference in number particles

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Substrate	Chip	Pressure	Joint thickness	Adhesion
Cu with	Si with	No	34 μm	4.0 ± 0.9 MPa
Ni/Au	Ti/Au	1 MPa	30 μ m	5.5 ± 2.3 MPa

Substrate	Chip	Pressure	Thickness	Thermal interface resistance (TIR) [mm ² K/W]
Cu with Ni/Au	Cu with Ni/Au	Soldering SAC alloy	35 µm	7
				8
Cu with Ni/Au	Si with Ti/Au	No	60 μ m	21
				23
		1 MPa	$30 \mu \mathrm{m}$	6
				7

Table 3 TIM at paste - results of thermal study

Figure 3 TIM AT paste, cross-section through sample for thermal study sintered with 1 MPa pressure



Table 4 TIM3 paste - results of adhesion assessment

Substrate	Chip	Pressure	Thickness	Adhesion
Cu with	Si with	No	25 µm	2.3 ± 0.5 MPa
Ni/Au	Ti/Au	2.4 MPa	15 μ m	1.9 ± 0.4 MPa

Table 5 TIM3 paste - results of thermal study

Substrate	Chip	Pressure	Thermal interface resistance (TIR) [mm ² K/W]
Cu with	Si with	No	38
Ni/Au	Ti/Au		42
		2.4 MPa	11
			14

(TIM3 paste contain far more particles) and interfaces between Ag particles. Heat has many more interfaces and particles to pass. Cross-section of TIM3 sample is presented in Figure 4. It is clearly observable that the increased layer density in the middle of the cross-section and the increased porosity at the interface with the Au layer. Porosity was assessed based on the number of voids in the layer in a qualitative manner. Form the surface inspection after shear test cohesive crack in the paste near Au interface was observed. Taking those results into consideration, further study on TIM 3 paste will not be continued. It was decided to make TIM AT2M paste with micrometer Ag grain size.

Figure 4 Cross-section of TIM3 joint between two Cu sheets with Ni/Au metallization, sintered under 2.4 MPa pressure



Thermal interface materials AT2M paste

To make the TIM AT2M paste, a mixture of spherical Ag particles with a size of dozen micrometers and Ag flakes with a size of $1-3 \mu m$ was used. Results of adhesion measurements are presented in Table 6, and results of thermal characterization are in Table 7. Very good adhesion above 5 MPa was obtained for both pressure and pressure less sintering. Inspection of the surface after the shear test indicates a cohesive crack. At the same time, results of thermal characterization clearly shows that application of pressure during sintering process significantly reduces thermal resistance of the joint. In Figure 5,

Table 6 TIM AT2M paste – results of adhesion asses	sment
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Substrate	Chip	Pressure	Thickness	Adhesion
Cu with	Si with	No	30 μm	11.7 ± 1.7 MPa
Ni/Au	Ti/Au	2.4 MPa	17 μm	8.5 ± 1.9 MPa

Table 7 TIM AT2M paste - results of thermal study

Substrate	Chip	Pressure	Thickness	Thermal interface resistance (TIR) [mm ² K/W]
Cu with Ni/Au	Si with Ti/Au	No	38 <i>µ</i> m	23
				24
		2.4 MPa	15 μ m	5
				6

Figure 5 Joint TIM AT2M between Cu sheet with Ni/Au metallization and Si chip with Ti/Au metallization, sintering without pressure



the cross-section of the TIM joint formed pressure less between Si chip with Ti/Au metallization and Cu substrate with Ni/Au metallization is presented. In the joint, there are many voids observed, they influence thermal resistance, but are not critical for adhesion properties of the sample. Usage of pressure during sintering process reduces thermal resistance of TIM (Table 7).

Energy-dispersive X-ray spectroscopy analysis near interface shows presence of both Au and Ag (Figure 6). That may indicate surface diffusion between Au metallization and Ag particles and explain good mechanical properties of the joint.

Figure 6 Cross-section of TIM AT2M joint between Si chip with Ti/Au metallization and Cu sheet with Ni/Au metallization, pressure less sintered



Note: EDS analysis of element composition in selected points

Reliability tests of adhesion for joints formed with TIM AT2M paste were performed: samples were aged at 125°C for 144 h. After this test, adhesion was above 10 MPa for all tested samples.

Conclusion and remarks

The influence of the shape and grain size of Ag powders in the developed pastes on the adhesion and thermal parameters of TIM1 joints were investigated. In Figure 7, the effect of paste composition (TIM AT– flake tens micrometer), TIM3 (micron flake+nano) and TIM AT2M (sphere+ flake micron) on adhesion and TIR is compared.

Joints made with paste containing Ag flake size particles size $3-7 \,\mu\text{m}$ (TIM AT paste) are comparable to SAC solder TIR, in the range of $6-7 \text{ mm}^2$ K/W. That is better result than typical TIM based on thermal grease, thermal gel or thermally conductive adhesive (min 10 mm²K/W). Unfortunately, these connections are not characterized by sufficiently good adhesion. Ag pastes based on a mixture of nano-Ag and micro-Ag (TIM3 paste) are characterized by poor adhesion and high thermal resistance, due to the builtin numerous interfaces between nano and micro particles with a BLT of approximately $30 \,\mu m$. Pastes based on a mixture of spherical Ag grains with a size of dozen micrometers and micrometer-sized flakes (TIM AT2M) are characterized by the best adhesion (above 10 MPa) and the smallest TIR (5-6 mm²K/W), similar to SAC solders. Research on these pastes will be continued in terms of reducing the porosity of the TIM1 layer, which should improve the thermal properties of the TIM joint.

Figure 7 Graphs comparing adhesion (left) and thermal interface resistance (right) of all investigated TIM1s



Note: In the right graph each bar represents a separate sample

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