

Original Article



Improved sinter-bonding properties of silvercoated copper flake paste in air by the addition of sub-micrometer silver-coated copper particles

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ABSTRACT

To promptly form a bondline with high thermal stability and thermal conductivity using a small amount of expensive Ag, dies were attached to Ag finishes by pressure-assisted sinter bonding at 300 °C using micrometer-sized Ag-coated Cu (Cu@Ag) flakes. Small Cu@Ag particles of 350 nm size were also added to the paste to increase the sinterability by increasing the contact points, which resulted in a bimodal paste. The dewetting of the Ag shells in Cu@Ag induced initial sintering, and the rearrangement of the 350 nm Cu@Ag particles as well as the bending of the Cu@Ag flakes under pressure effectively filled the voids between the particles. As a result, shear strengths of almost 20 MPa and 28.9 MPa were obtained after only 1 and 5 min of sinter bonding, respectively. In addition, a bondline with a unique near full density microstructure was achieved in the 5 min sinter-bonded sample.

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1. Introduction

With the prevalence of the semiconductors and electronic products, solder alloys have been adopted for the bonding and mounting of various electronic components [1-10]. Although soldering technology enables many components to be simultaneously bonded within a relatively short time below 8 min [3], reliability problems still remain in its application in various fields, especially in high-temperature applications. A

typical soldering technique presents low melting temperature of formed bondline similar to its process temperature, which results in reliability issues such as remelting or a drastic reduction in mechanical properties in applications where the bondline is exposed to high temperature. Hence, the high-temperature solder alloys also cannot but have constraints at working temperatures approaching 300 °C [4–14]. Another notable drawback of Sn- or Pb-based solders is their low thermal conductivity [8], which can severely accelerate

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thermal degradation of heat-generating devices and neighboring parts.

The pursuit of methods to form more reliable bonding parts at high temperatures has motivated studies to develop different bonding techniques and materials. For example, Sibased power devices are exchanging with ones of wide band-gap material such as SiC owing to their high power conversion efficiency and low switching loss, concurrently pursuing the junction temperature increase up to 300 °C for expansion of a design window for a module system [9-14]. Therefore, current common soldering technique using high Pb solders for bonding of the high-efficiency power devices on a direct-bonded copper (DBC) substrate should be altered with rapid expansion of electric vehicle industry. Although transient liquid phase bonding, a modified soldering technique, has been extensively researched [15-17], reliability concerns due to the brittle nature of the bondline composed of intermetallic compounds (IMCs) and formation of irregular voids in the bondline even after long bonding time still remained [15,17]. The reliability in the brittle bondline containing voids can drastically drop and be linked to fatal results under vibration environment during typical vehicle operation [18,19]. The low thermal conductivities of IMCs are also one of properties to be enhanced [20].

Thus, sinter bonding using silver (Ag) particles has currently being explored as a hotter research topic with the low-temperature sinterability as well as high melting point of Ag [21-29]. The mechanical properties containing ductility and extremely high thermal conductivity of Ag are also advantageous [22,23,26-29]. Nevertheless, the high material cost of Ag is a big problem in the industrial use, and the cost increases more significantly with the reduction of particle size that affects the sinterabilty. Furthermore, the addition of nanoparticles in a paste prepared for mass production raises a practical challenge with regard to ensuring a homogeneous dispersion of particles during mixing of the paste and accomplishing the desired rheological properties. Additionally, the bondline composed of pure Ag raises the concerns about ion migration, which can be suppressed by Cu-based bonding material [30-34]. These drawbacks can be simultaneously reduced with the use of micrometer- and submicrometer-sized Ag-coated copper (Cu@Ag) particles because the core material is low-coat Cu [35-38]. Moreover, the bondlines composed of larger volume of Cu can hinder the ion migration of Ag, and the thermal conductivity may be similar to that of Ag. Although total volume of Cu is much

larger than that of Ag, the oxidation of Cu particles in air will be suppressed owing to the coverage of Ag shells.

Reduction of bonding time in the sinter-bonding process can be a crucial research motivation because it was directly linked to productivity of the die bonding which is considered as a bottleneck step in the production of a power module. If the bonding time was reduced to several minutes from current several tens of minutes, the current die bonding using high Pb solder might be exchanged more aggressively with the sinter bonding.

Therefore, a rapid sinter-bonding technique with sufficient strength within several minutes using micrometer- and submicrometer-sized Ag-coated Cu particles was suggested in this study. To enhance the sinterability more effectively by increase of surface area, micrometer-size flake-type Cu@Ag particles were selected as a main material, and submicrometer-size Cu@Ag particles were additionally mixed. Our study was focused on obtaining rapid sinterbonding speed at 300 °C that is slightly lower than the bonding temperature of current high Pb solders such as Pb–5Sn.

2. Experimental work

2.1. Preparation of particles

Cu flakes (CFL07, size: 6.5–7.2 µm, Join M) were purchased, and 20 wt% Ag coating to fabricate a core-shell structure was carried out as follows; Cu flakes of 3 g were immersed into a pretreatment solution and stirred at 200 rpm for 2 min at room temperature (RT) to remove the oxide layers from the surfaces. The pretreatment solution was prepared by dissolving 2.09 M of ammonium sulfate ((NH₄)₂SO₄, 99%, Samchun Pure Chemical) and 150 mM of ammonium hydroxide (NH₄OH, 28-30%, Daejung Chemicals & Metals) in 100 mL of deionized (DI) water. After the pretreatment, the Cu flakes were washed three times with DI water and maintained in suspension by dispersing in 100 mL DI water. A Ag plating solution was made by dissolving 3.56 M of NaOH (98.0%, Samchun Pure Chemical), 0.89 M of ethylenediaminetetraacetic acid (EDTA, C10H16N2O8, 99.0%, Samchun Pure Chemical), and 0.11 M of silver nitrate (AgNO₃, 99.8%, Hojeonable) in 50 mL of DI water. The Ag plating solution was added dropwise into the Cu flake suspension at the rate of 10 mL/min under continuous stirring of 10 mL/min at RT, after which, the mixed solution was



Fig. 1 – SEM images of (a) Ag-coated Cu flakes and (b) sub-micrometer Ag-coated Cu particles.



Fig. 2 – Cross-sectional BSE images of upper and lower bondlines sinter-bonded under a pressure of 10 MPa at 275 °C using bimodal Ag-coated Cu particles for (a) 1, (b) 3, and (c) 5 min. (d) EDS mapping image of (b).

stirred at the rate of 250 rpm for 10 min to complete the Ag coating reaction. The fabricated Cu@Ag flakes were washed twice successively with DI water and methyl alcohol (95%, Hanwha Chemical) and then dried at RT in a vacuum chamber.

Approximately 350 nm Cu particles were synthesized inhouse and coated with 20 wt% Ag using a reported method [35].

2.2. Preparation of paste

Pastes containing both the Cu@Ag flakes and submicrometer Cu@Ag particles were prepared by mixing with a reducing formulation blended in-house. The weight ratio of flake-tosubmicrometer particle was fixed as 7:3, which means that the addition amount of small particles is sufficient to provide enhanced sinterability by increasing total contact areas, compared with the void fraction anticipated from arrangement of the flakes during printing. The weight ratio of particle-to-vehicle was adjusted to 85:15 to obtain appropriate viscosity for subsequent printing.



Fig. 3 – Schematic diagrams showing formation mechanism of near full density bondline.

2.3. Sinter bonding method

Both dies and substrates for die sinter-bonding tests were prepared with a dummy Cu plate of dimensions $3 \times 3 \times 1 \text{ mm}^3$ and $10 \times 10 \times 1 \text{ mm}^3$, respectively, finished with an approximately 1 µm-thick Ag layer. The Ag finishes were formed by electroless plating on the Cu plates. The prepared paste was printed on the Ag-finished substrate through a stencil mask with a slit volume of $3 \times 3 \times 0.1 \text{ mm}^3$. After printing, the die was placed under aligning on the printed pattern and the resulting sandwich-structure was heated to 300 °C in air; the heating rate was approximately 37.5 °C/s. The sinter bonding was conducted under the external pressure of 10 MPa from the start of hating, which was maintained for entire bonding time.

2.4. Characterization

The size and morphology of the used Cu@Ag particles, the microstructures of formed bondlines, and fracture surfaces after shearing were observed using a high-resolution scanning electron microscope (HR-SEM, SU8010, Hitachi). The bonding strength of the bondline was determined from the maximum stress value measured during shearing of the bonded die at the speed of $200 \,\mu$ m/s from the height of $200 \,\mu$ m above the surface of a substrate using a bond tester (Dage-4000, Nordson Dage).

3. Results and discussion

3.1. Morphology of used particles

Fig. 1(a) and (b) show the SEM images of the Cu@Ag flakes and sub-micrometer Cu@Ag particles used in this study, respectively. The average sizes of the particles after the plating of 20 wt% Ag to form Ag shells are approximately 7.0 μ m and 350 nm, respectively, which are not significantly different from those of the initial Cu flakes and sub-micrometer Cu particles because the Ag coating proceeds by a galvanic replacement reaction between the Cu surface and Ag ions during plating. The average thickness of Ag shells in the Cu@Ag flakes was several tens of nanometers from a cross-sectional image of the particles.

3.2. Microstructures of bondlines

Fig. 2 shows the backscatter electron (BSE) images of the upper and lower bondline microstructures after sinter bonding at 275 °C under an external pressure of 10 MPa using the micrometer-sized Cu@Ag flakes/sub-micrometer Cu@Ag particles. The samples sinter-bonded for only 1 min (Fig. 2(a)) show sintering among the Ag shells at the contacts between the Cu@Ag flakes. However, the 350 nm Cu@Ag particles exhibit two different behaviors: sintering due to contacts with the Cu@Ag flakes in the narrow voids between the Cu@Ag flakes, and rearrangement in the wide voids between the Cu@Ag flakes. In the former case, the sintering between the Ag shells occurred by the dewetting of the Ag shells due to a large lattice mismatch of 11.7% when the temperature exceeded 200 °C [36−38]. The dewetting occurred preferentially in the



Fig. 4 – Cross-sectional BSE images of upper and lower bondlines sinter-bonded under a pressure of 10 MPa at 300 °C using bimodal Ag-coated Cu particles for (a) 1, (b) 3, (c) 5, and (c) 10 min.

350 nm Cu@Ag particles because dewettability is inversely proportional to particle size, that is, to the curvature radius of a particle [35–38]. The Ag nodules formed on the surface of a 350 nm Cu@Ag particle by dewetting can behave temporarily like a quasi-fluid and shows flowability owing to the Gibbs—Thomson effect [39,40]. The average thickness of the Ag shells in the 350 nm Cu@Ag particles was approximately 10 nm. Thus, the Ag shells in the 350 nm Cu@Ag particles promptly participated in sintering with a quasi-fluid behavior immediately after forming contacts with all the neighboring Ag-coated Cu particles. With increasing temperature, the Ag shells in the Cu@Ag flakes were dewetted, which accelerated the sintering between the Ag shells. Although the average thickness of the Ag shells in the Cu@Ag flakes was calculated as several tens of nanometers owing to a drastic decrease in the specific surface area, the peaks of the bumpy surfaces formed by dewetting may still provide a quasi-fluid behavior. This is evident from the sintering of the micrometer-sized



Fig. 5 – Shear strengths of dies sinter-bonded at different temperatures for different times using a paste containing both micrometer-sized Cu@Ag flakes and 350 nm Cu@Ag particles.

Cu@Ag flakes as well as the 350 nm Cu@Ag particles with the Ag finishes at the upper and lower bondlines.

With an increase in bonding time to 3 min (Fig. 2(b)), the total void fraction decreased; thus, most of the 350 nm Cu@Ag particles participated in sintering. Furthermore, the Ag layer coating at the interfaces of the sintered Cu@Ag flakes partially disappeared, resulting in the initiation of sintering between the Cu cores. The diffusivity of Cu in the Ag matrix is much higher than that of Ag because of the low activation energy, causing an out-diffusion of Cu through the locally thin Ag layer [41]; eventually, at an elevated temperature, the particles were sintered by Cu when the particles with a Cu core came into contact with other particles. Considering the dewetting sequence, Cu sintering between the 350 nm Cu@Ag particles would be more frequent. In addition, the area sintered by Cu at the upper and lower bondlines increased with a decrease in total void fraction. The energy dispersive spectroscopy (EDS) of Fig. 2(b) (Fig. 2(d)) shows again the sintering (in the arrowed regions) between the Cu cores.

For a bonding time of 5 min (Fig. 2(c)), the bondline exhibits a near full density microstructure. Compared with the bonding times reported in similar studies using pure Ag particles [23,42], the bonding time required to achieve near full density was significantly shorter in this study. The short bonding time is attributed to the bending deformation of the micrometer-sized Cu@Ag flakes and the continuous rearrangement of the 350 nm Cu@Ag particles under 10 MPa pressure. During the bending deformation of Cu@Ag flakes under pressure, the neck of a sintered 350 nm Cu@Ag particle can be easily detached owing to the minute contact area. Hence, the detached 350 nm Cu@Ag particle would move and finally settle at the bottom of a deformed void from a sparse position at the initial void where it existed. The rearrangement of the 350 nm Cu@Ag particles is an unexpected merit of the addition of tiny Cu@Ag particles. In summary, the repetition of the bending of the Cu@Ag flakes under pressure and the rearrangement of the 350 nm Cu@Ag particles decreased the total void fraction with an increase in bonding time. Furthermore, the remaining Ag in the detached Cu@Ag particles can again play a role in filling the remaining tiny voids due to the presence of Ag nano-nodules. The formation mechanism of the near full density bondline is displayed in Fig. 3. It is anticipated that the initiation and propagation of a crack in the near full density bondline will be suppressed, which will enhance the reliability of the joint. Moreover, Cu sintering at the interfaces of the sintered Cu@Ag flakes was more frequent in the 5 min sinter-bonded sample. The porosity of the 1 min, 3 min, and 5 min bondlines calculated using open source image processing program was 9.20%, 2.88%, and 0.01%, respectively.

To achieve faster sinter bonding and obtain a dense bondline, the bonding temperature was increased by 25 °C. The BSE images of the upper and lower bondlines sinterbonded at 300 $^\circ\text{C}$ are displayed in Fig. 4. In the samples sinter-bonded for 1 min (Fig. 4(a)), large voids are present, even though Cu@Ag aggregates were formed by sintering between the 350 nm Cu@Ag particles. In addition, the sinter bonding of the aggregates at Ag finishes and Cu sintering at the interfaces of the sintered Cu@Ag flakes are observed. In the samples sinter-bonded for 3 min (Fig. 4(b)), the void fraction decreased. Furthermore, the Cu sintering fraction in the sintered Cu@Ag flakes significantly increased. Thus, the all bimodal particles were closely sintered with the presence of a few large voids. With the close sintering of the Ag finishes, hardly any void was present at the interfaces of the bondline/ Ag finish. These results indicate that the sintering degree of the sample sinter-bonded for 3 min at 300 °C is higher than that of the sample sinter-bonded for 3 min at 275 °C due to the high bonding temperature. With an increase in bonding time to 5 min (Fig. 4(c)), the density of the bondline drastically increased due to improved sintering. Moreover, the dense microstructure was maintained after sinter bonding for 10 min (Fig. 4(d)). In summary, the near full density microstructure of the bondline is achieved after only 5 min of sinter bonding at 300 °C.

3.3. Shear strength

Fig. 5 shows the shear strengths of the dies sinter-bonded using the bimodal paste as a function of temperature and time. The strength increased with an increase in bonding time for both 275 and 300 °C bonding temperatures. However, for the samples bonded at 275 °C, the strength of the dies bonded for 5 min do not approach that (20 MPa) of the sample bonded using Pb–5Sn [43,44]. Hence, the temperature is insufficient to replace the current soldering technology using high Pb solders by the sinter-bonding technique using the bimodal paste.

However, for the samples bonded at 300 °C, the strength increased to 20 MPa even for a considerably short bonding time of 1 min. This extremely rapid sinter-bondability is attributed to sintering between the Ag shells and the subsequent Cu sintering both at the interfaces between the particles and the interfaces between the particles and Ag finishes due to the increase in total contact area by the combination of flakes and sub-micrometer particles. The strength continuously increased to 28.9 MPa with an increase in bonding time to 5 min due to the reinforcement of Cu sintering, and saturated at bonding times above 5 min.



Fig. 6 – Low- and high-magnification BSE images of fracture surfaces of bondlines fabricated at 275 °C for different bonding times: (a) 1, (b) 3, and (c) 5 min.

3.4. Fractography

The low- and high-magnification BSE images of the fracture surfaces of the dies sinter-bonded at 275 °C for different bonding times after shear testing are displayed in Fig. 6. After a short bonding time of 1 min (Fig. 6(a)), fracture frequently occurred at the interface between the Ag finish of a chip and the Cu@Ag particles, which indicates a partial interface failure. Furthermore, the sintering between the Cu@Ag flakes and 350 nm Cu@Ag particles was not severe even in the Cu@Ag particles stained on the Ag finish, which shows a distinction

between the flakes and 350 nm particles. Similar results are observed for the dies sinter-bonded for 3 min (Fig. 6(b)). However, the facture surface changed for a bonding time of 5 min (Fig. 6(c)); the entire area of the fracture surface is covered with Cu@Ag particles, which indicates an obvious coherent failure. Moreover, the 350 nm Cu@Ag particles are considerably sintered between the Cu@Ag fakes, and dimples, which can be formed by the ductile deformation of the metallic phase, are observed in the fracture surface.

Fig. 7 shows the fracture surfaces of the dies sinter-bonded at 300 $^{\circ}$ C as a function of the bonding time. All the observed



Fig. 7 — SEM images of fracture surfaces of bondlines fabricated at 300 °C for different bonding times: (a) 1, (b) 3, (c) 5, and (d) 10 min.

fractures can be characterized as coherent fracture. In the fracture surfaces of the 1 min sinter-bonded samples (Fig. 7(a)), the 350 nm Cu@Ag particles are coarsened with the Cu@Ag fakes, and shear bands, which are normally formed by strong metallic bonding, are also observed. The degree of sintering and the prevalence of the shear bands in the fractured surfaces increased with an increase in bonding time to



Fig. 8 – High-magnification BSE image of the fracture surface of bondlines fabricated at 300 $^\circ C$ for 10 min.

5 min. However, honeycomb-like fracture surfaces were observed for the 5 min sinter-bonded sample (Fig. 7(c)), and the total surface area of the 10 min sinter-bonded sample increased (Fig. 7(d)).



Fig. 9 – Shear strengths of dies sinter-bonded at 300 $^\circ C$ for different times using the bimodal paste comprising only spherical Cu@Ag particles.



Fig. 10 — Cross-sectional BSE images of upper and lower bondlines sinter-bonded under a pressure of 10 MPa at 300 °C using the bimodal paste comprising only spherical Cu@Ag particles for different bonding times: (a) 1, (b) 3, (c) 5, and (c) 10 min.

To identify the honeycomb-like surfaces, the fracture surface of the dies sinter-bonded at 300 °C for 10 min was observed with the high-magnified BSE mode (Fig. 8). The EDS results of region 1 indicated an excessive Ag content, whereas those of region 2 indicated a nearly pure Ag composition. The EDS results of region 3 showed a mixed composition with a large amount of Cu and a small amount Ag. These results imply that the honeycomb-like surface is the interface between the Cu dummy die and the Ag finish layer. Therefore, the saturation of the shear strength of the samples bonded for 5-10 min at 300 °C is considered to be a result of the

insufficient adhesion of the Ag finish layer. The effect of $300 \degree C$ bonding temperature on the degree of adhesion at the interface between a Cu dummy die and Ag finish is another research topic [45].

3.5. Comparison with spherical Cu@Ag particles

To verify that the increase in total contact area by the flakes positively affects rapid sinter bonding, sinter bonding was conducted after substituting the flakes with Cu@Ag particles having an average size of 2.0 μ m. Micrometer-sized core Cu



Fig. 11 – BSE images of fracture surfaces of bondlines fabricated at 300 °C using the bimodal paste comprising only spherical Cu@Ag particles for different bonding times: (a) 1, (b) 3, (c) 5, and (c) 10 min.

was purchased from Join M, and Ag plating was performed inhouse by the above-mentioned process. The shear strength of the dies sinter-bonded at 300 °C for different times using the bimodal paste comprising only spherical Cu@Ag particles is displayed in Fig. 9. For a bonding time of 1–5 min, the strengths are lower than those of the samples sinter-bonded using the paste composed of both flakes and 350 nm particles. However, for the samples sinter-bonded for 10 min, the strength of the samples fabricated using the paste composed of only spherical Cu@Ag particles is the same as that of the samples fabricated using the paste composed of nn particles. The samples fabricated using the paste composed of only spherical Cu@Ag particles is the same as that of the samples fabricated using the paste comprising both flakes and 350 nm particles.

Fig. 10 presents the cross-sectional BSE images of the bondlines sinter-bonded under 10 MPa at 300 °C using the bimodal paste comprising only spherical Cu@Ag particles. In the samples sinter-bonded for 1 min (Fig. 10(a)), shape change was not observed for the 2 µm Cu@Ag particles. Thus, the sintering among the Ag shells between the Cu@Ag particles is slight only at the small-area contacts and the sintering degree is the same as that of the bondline/Ag finish interfaces. However, with increasing bonding time, the total void fraction of the bondline decreased and the contours of the 350 nm Cu@Ag particles became blurry. The near full density bondline was achieved after bonding for 10 min due to the slow rearrangement of the 350 nm Cu@Ag particles because the 2 µm Cu@Ag particles did not change shape. Therefore, the acceleration of sinter bonding by the flakes can be attributed to two main reasons: the increase in total contact area and the bending behavior of the flakes.

The fracture surfaces of the dies sinter-bonded at 300 °C using the bimodal paste comprising only spherical Cu@Ag particles are displayed in Fig. 11. Although the fracture surfaces of the all dies bonded for up to 10 min are obtained with the sintered bondline, the sintering degrees are different. In the die sinter-bonded for 1 min (Fig. 11(a)), the contours of both small and large Cu@Ag particles in the surface are distinct due to slight sintering. With an increase in bonding time to 3 and 5 min (Fig. 11(b) and (c)), numerous tiny voids are formed in the surfaces due to intense sintering. The blurring of the particle contours due to sufficient sintering was observed in the facture surface of the sample sinter-bonded for 10 min, which exhibited a high shear strength of 28.8 MPa.

4. Conclusions

To achieve rapid sinter bonding in air using low-cost particles, 7 µm Cu@Ag flakes and 350 nm Cu@Ag particles were mixed with a reducing formulation to form a paste and an external pressure of 10 MPa was applied. The sinter bonding was slow at a bonding temperature of 275 °C, but proceeded rapidly at 300 °C. Thus, a shear strength of almost 20 MPa was achieved only after 1 min of bonding at 300 °C. With an increase in bonding time to 5 min, the strength increased to 28.9 MPa and a stable bondline with a near full density microstructure was obtained. The increase in total contact area by the use of flakes and the dewetting of Ag shells in Cu@Ag resulted in rapid sintering. In addition, the bending of the flakes and the resultant rearrangement of the 350 nm Cu@Ag particles enhanced the bondline density and strength with increasing bonding time. All the fractures observed after bonding at 300 °C were characterized as coherent fracture, and the formation of shear bands due to strong metallic bonding was observed.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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