

Low Temperature Bonding of Cu Metal through Sintering of Ag Nanoparticles for High Temperature Electronic Application

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Abstract: Lead-based solders bring pollution to the environment and result in health threat to humans. The preparation and application of metallic nanoparticles provide a potential method to develop Pb-free bonding materials. In this article, bonding of Ag-coated Cu bulks was realized through low temperature sintering by directly using the chemically-reduced Ag nanoparticle paste and baked nanoparticle powders at 60 °C, respectively. The results indicate that the capillary flow of paste caused a ring-like deposit on substrate coating, while this phenomenon disappeared when using the powders. Increasing bonding temperature facilitated the sintering, and shear strengths of 20 MPa and 84 MPa was obtained at bonding temperature of 250 °C for 30 min under 20 MPa when using the Ag paste and powders, respectively. High joint strength value of bonded region using paste is due to the high effective bonding pressure and small effective bonded area. Finally, for the extensive application in packaging industry especially for high temperature electronics, challenges such as the improvement of screen and stencil printing ability of paste, avoiding bonding pressure and lowering the cost were pointed out based on our and other researcher's achievements. Cu nanoparticle or Cu-containing nanoparticle mixture pastes are promising but the problems of oxidation and bonding stability must be resolved urgently.

Keywords: Ag nanoparticle paste, Ag nanoparticle powder, lead-free bonding materials, electronic packaging.

1. INTRODUCTION

Lead-based solders are used widely as electronic packaging materials. However, Pb and its compounds result in serious health threat to humans when depositing the Pb-containing electronic devices in the environment. Emerging regulations have targeted the elimination of Pb usage in electronic assemblies in many countries [1, 2]. The development of Pb-free solders has become imperative. For a practical solder a low melting temperature, a high wettability and mechanical integrity, an easy fabrication and an affordable cost are required. Although some Pb-free solders are commercially available, such as Sn-Ag, Sn-Cu, Sn-Ag-Cu, Sn-Bi alloy systems, none of them matches high performance standards [3].

The development of nanojoining gives us a potential method to find suitable Pb-free packaging materials and the relative processing technology [4]. It is well known that, the decrease of size of the particles results in an increase of the diffusion coefficient and a decrease of melting temperature due to the large surface energy and high surface area to volume ratio [5-8]. The size effect can be applied to prepare nanoparticles for low temperature Pb-free interconnect in electronics packaging and assembly. Hirose's research group

has reported a novel bonding process of non-coated or Ni/Ag (or Ni/Au) coated Cu bulks at temperature range of 200-400 °C for 3 min under a pressure of 1-10 MPa by using Ag metallo-organic nanoparticle pastes. In their study, pastes were prepared by mixing different organic solvents and the chemically-reduced Ag nanoparticles with an organic shell [9-11]. Based on the sintering-bonding research of small area SiC chips without bonding pressure, Lu's group further investigated the bonding of large-area (>100mm²) chips at around 275 °C under 1-5 MPa using Ag paste, which was prepared by mixing various organic components (including dispersant/surfactant, binder and thinner) and chemically-reduced Ag nanoparticles [12]. Recently, Hani *et al.* have realized the bonding of fine Cu wire to Cu pellets at temperatures below 160 °C under 5 MPa pressure for 30 min directly using a chemically-reduced Ag nanoparticle paste [13]. The further research results of Hu and Hani *et al.* confirmed that sintered network of Ag nanoparticle paste at only 100°C can work as bonding structures for fine Cu wire to Cu pad applied for polymeric flexible electronics packaging [14]. It is worth to note that sintered joints with Ag nanopastes can be operated at much higher surrounding temperature compared to the soldering temperature. The melting point of the sintered Ag layer close to 960 °C, a melting point of Ag bulks, is much higher than the melting point of the individual Ag nanoparticle in a paste [9].

In this work, two styles of Ag nanoparticle materials were used for sintering bonding: one is original chemically-reduced Ag nanoparticle paste similar to that used in

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reference [13] and another is Ag nanoparticle powders by baking the paste at 60 °C. We compared their characteristics and bondability. The morphology and size distribution of nanoparticles were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Thermal characteristics of Ag nanoparticles were detected using differential scanning calorimetry (DSC). The bondability of Ag nanoparticle paste and nanoparticle powder was evaluated by bonding Ag-coated Cu bulks. The bonding strengths were analyzed by shearing tests. The morphology and microstructures at the interface of the fractured joints were also observed.

2. MATERIALS AND METHODOLOGY

Ag nanoparticles used in this study were prepared using a chemical reduction action in aqueous solution. Silver nitrate (AgNO_3) and sodium citrate dihydrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$), both in analytical grade, were used. Silver nitrate dissolved in deionized water was used as precursors. After the solution was heated to 80 °C, sodium citrate dihydrate solution, which served as a reducing agent, was added slowly while vigorously stirring under an ambient atmosphere. Subsequently the mixed solution was heated to 90 °C, and kept for 1 hour with magnetic stirring. As reduction action occurred, the color of the suspension solution turned from colorless to yellow first, and then to grayish green, which indicated the formation of Ag nanoparticles. Then the suspension solution was cooled to room temperature. The concentration of Ag nanoparticles was increased by centrifugation. The Ag nanoparticle paste was extracted using a syringe after removing the top clear water. Based on this, Ag nanoparticle powder was prepared by baking the concentrated paste at 60 °C. The nanoparticle powder accumulated at the bottom of the glassware baker was collected using a clean spade.

In order to determine the size distribution of Ag nanoparticles, an appropriate concentrated Ag nanoparticle suspension solution (called paste) was detected using a malvern instrument zetasizer. Then the size and shape of the Ag nanoparticles were analyzed by SEM and TEM. Samples for SEM were prepared by dropping Ag nanoparticle paste onto silicon substrates. The TEM specimens were prepared by dripping a few drops of Ag nanoparticle paste onto a carbon-coated copper grid and drying them in air. The thermal property of these silver powders was characterized by DSC methods with heating rate of 10 °C/min from 25 °C to 300 °C in the N_2 atmosphere (purity of N_2 is 99.9%, 40 ml/min).

Ag-coated Cu metals were bonded using Ag nanoparticle paste and powders. The specimens used for bonding experiments consist of two Cu cylinders (Fig. 1). Ag nanoparticle paste was supplied on the facing surfaces of bonding specimens drop by drop. Put the smaller specimen on the top of bigger specimen when enough Ag nanoparticle paste was gathered. Similarly, a thin layer of Ag nanoparticle powders was applied by sandwiched between two Cu metals. Afterwards, these prepared specimens were heated up to the bonding temperature in air under a bonding pressure (bonding temperature: from 150 °C to 250 °C; bonding time: 30mins). The bonding pressure (P_b) is calculated from the equation: $P_b = F/S$, where F is the maximum applied force on

the specimens, and S is the lower surface area of the small Cu metal cylinder. So the nominal bonding pressure is 20MPa (Calculated bonding pressure). The bonding strength was evaluated by measuring the shear strength measured using a thermal-mechanical simulator Gleeble 1500D with a cross-head displacement speed of 5 mm/min at room temperature.

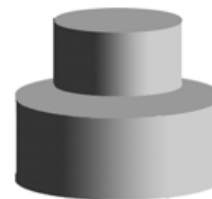


Fig. (1). Schematic illustration of the bonding specimens of big cylinder ($\Phi=10$ mm, $h=5$ mm) and small cylinder ($\Phi=6$ mm, $h=5$ mm).

3. RESULTS AND DISCUSSION

3.1. Characteristics of Ag Nanoparticles

As shown in SEM image (Fig. 2a), most of Ag nanoparticles are spherically shaped particles. Some nanorods are included. The size distribution of these nanoparticles is from 20 nm to 80 nm [15]. This size distribution was also confirmed by an analysis on a malvern instrument zetasizer, in which effective diameter of nanoparticles is 45.4 nm. TEM image (Fig. 2b) displays these nanoparticles are covered by thin organic shells inferred as citrate function group, and these organic shells can prevent the sintering of nanoparticles during storage.

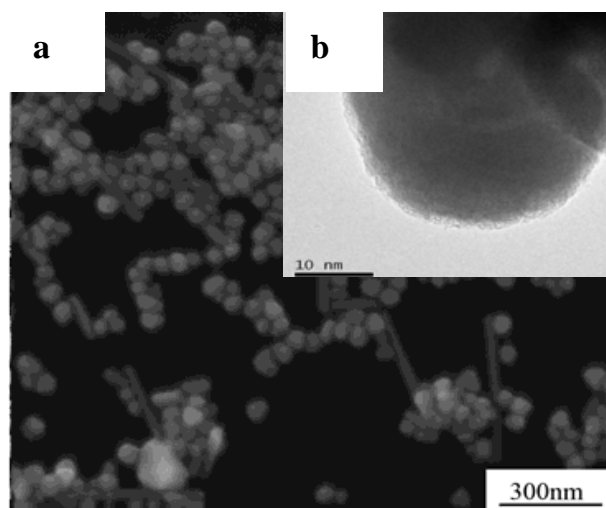


Fig. (2). Images of the synthesized silver nanoparticles: a-SEM and b-TEM.

The morphology of Ag nanoparticle powders is confirmed by SEM image (Fig. 3a, b) and TEM image (Fig. 3c). These powders are actually many microscale plates (Fig. 3a). The micron-sized plates consist of nanoscale particles with diameter of about 40 nm (Fig. 3b). Each nanoparticle still has an organic shell when observed using TEM, while no sintering of Ag nanoparticles occurs (Fig. 3c). The formation of microscale plates is thus due to the agglomeration of adjacent nanoparticles. It should be note

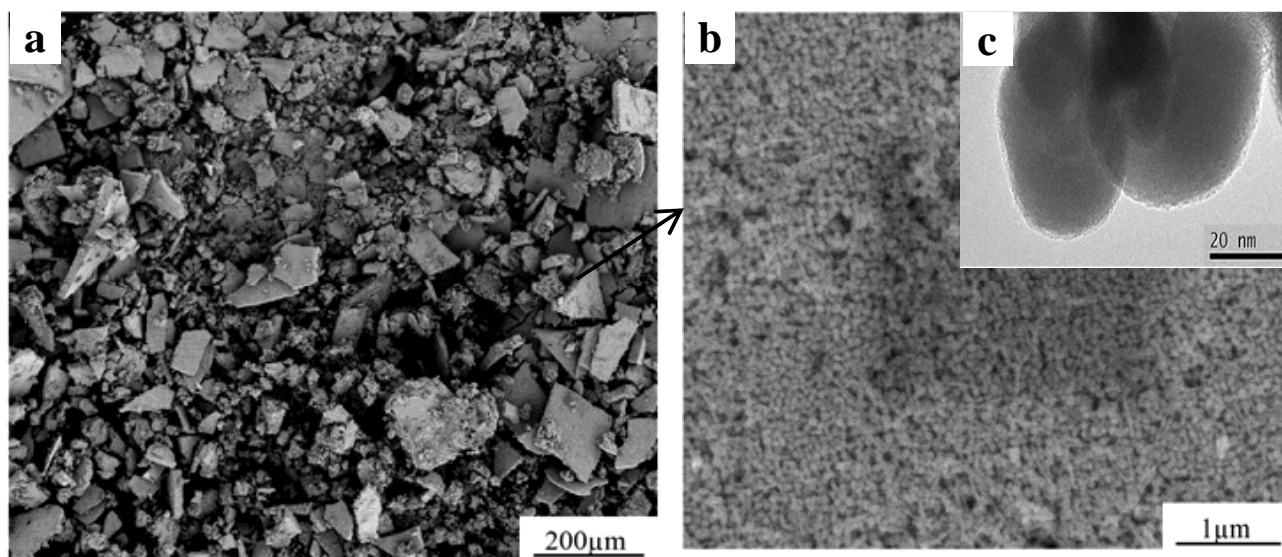


Fig. (3). SEM (a, b) and TEM (c) image of Ag nanoparticle powder.

that there are some differences between agglomeration and sintering. Agglomeration is a procedure in which powder compacts are attracted together by weak forces (Van der Waals/electrostatic forces, etc). While the sintering is a procedure that the material is bonded together by solid necks of significant strength such as metallic force [16]. It is reasonable to reduce that these Ag nanoparticles are agglomerated together due to weak bonding, since organic shells still cover on the surfaces. This indicates that the nanoparticle powder can be used as a bonding material.

Ag nanoparticle powders were analyzed using DSC in a temperature of 50 °C to 300 °C in a flowing nitrogen atmosphere (Fig. 4). When the temperature was increased above 150 °C, some organic component covered on the nanoparticles may oxidize or burn out by reacting with the surface adsorbed oxygen or traced oxygen in N₂ flow. And the exothermic peak may be caused by this chemical reaction. After the burn out of the organic components, the shell of nanoparticles disappears. The nanoparticle starts to aggregate, and therefore sintering of nanoparticle will occur. Our recent work confirmed that the sintering of Ag nanoparticles will start at 150 °C [15]. From the above analysis, it is known that the bonding temperature should be higher than 150 °C to make sure the sintering-bonding process occurs.

3.2. Morphology and Microstructure of the Joints

A ring-like structure appears at the surface of the joint bonded using Ag nanoparticle paste (Fig. 5a). This ring-like shape region, which has a brighter color, is the actual contact area between Ag nanoparticle paste and the two Cu pellets (Fig. 5a). At higher magnification, the microstructure of this ring like shape region displays a dimple structure, which suggests ductile behavior in sintered Ag nanoparticles (Fig. 5b) [17]. This ring like shape is formed during the Ag nanoparticle paste coating process. When the Ag nanoparticle paste is dripped on the Cu metal surface, simultaneous evaporation of the water occurs. At the perimeter, all the liquid is removed and the drop shrinks. But

the radius of the drop cannot shrink, as its contact line is pinned. To prevent the shrinkage, liquid must flow outwards. The liquid evaporating from the edge is replenished by the liquid from the interior. The resulting outward flow can carry most of the dispersed Ag nanoparticles to the edge. The Ag nanoparticles will gather at the edge of the drop and leave a dense ring-like deposit along the perimeter [18].

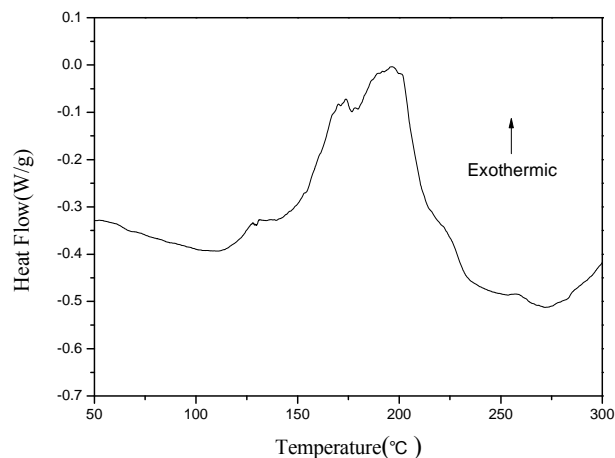


Fig. (4). DSC curves of the as-prepared Ag nanoparticle powder.

Hani *et al.* reported that this coffee ring effect can be suppressed by multi-deposition of the nanoparticles [13]. In that study, Ag nanoparticle paste was used to bond Cu wires to Cu foils, and the influence of coffee ring effect was not obvious. In this study, the coffee ring effect can not be well suppressed by multi-deposition process.

A dense Ag sintered layer forms between the two Cu pellets after bonding when using Ag nanoparticle powder under a pressure of 20 MPa (Fig. 6a). The coffee ring effect totally disappears, because these Ag nanoparticles are placed in solid forms. It should be noted that the agglomeration of the Ag nanoparticles will reduce the surface energy to some extent. In this case each nanoparticle still has an organic

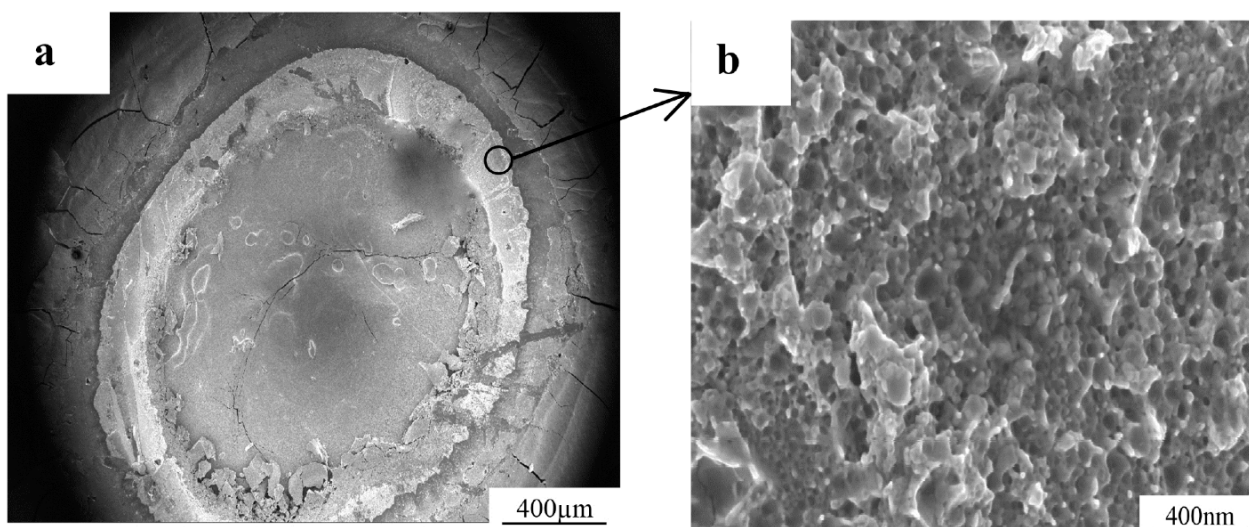


Fig. (5). SEM image of interface microstructure of joint using Ag nanoparticle paste: **a**-lower magnification; **b**-higher magnification (bonding temperature: 200 °C; calculated bonding pressure is 20 MPa; bonding time: 30 mins).

shell and no sintering of Ag nanoparticles occurs. So the nanoparticle powders can be used as bonding materials. Some simple structures appear when observing the interface microstructure of the joint at higher magnification. The dimple surface structure indicates the formation of a good interface (Fig. 6b). Ag nanoparticle powders can be used as lead-free materials to bond Cu metal bulks at low temperatures, and this proves potential useful for application in electronic packaging industry. Nevertheless, the bonding pressure using Ag nanoparticle paste or powders is still too high. A further study to decrease of bonding pressure is necessary in the future.

3.3. Mechanical Property of the Joints

The shear strength of the joints was measured when using Ag nanoparticle paste and powders at different temperatures (Fig. 7). The joint strengths increase with the rising of bonding temperature. The shear strength of bonding using Ag nanoparticle powder increases to 19.6 MPa at 250 °C.

Similarly, the shear strength of the joint is increased to 84.2 MPa at bonding temperature of 250 °C using Ag nanoparticle paste. A higher bonding temperature is beneficial to joint formation. Higher temperatures favor the sintering process of Ag nanoparticles, and allow for better diffusion and contact between the Ag nanoparticles and base metal [15, 19]. The shear strength using Ag nanoparticle paste is higher than the reported result [9]. The Ag nanoparticles used there are Ag metallo-organic nanoparticles. In this study, the current Ag nanoparticle paste is directly condensed from Ag nanoparticle solution through centrifugation (almost pure nanoparticles). The differences of particle size of Ag nanoparticles and the variable organic content between the present work and other reports may lead to different joint strengths. Besides that, a higher bonding pressure also enhances the joint strength.

It can be seen that joint strength using Ag nanoparticle paste is higher than that using Ag nanoparticle powder under

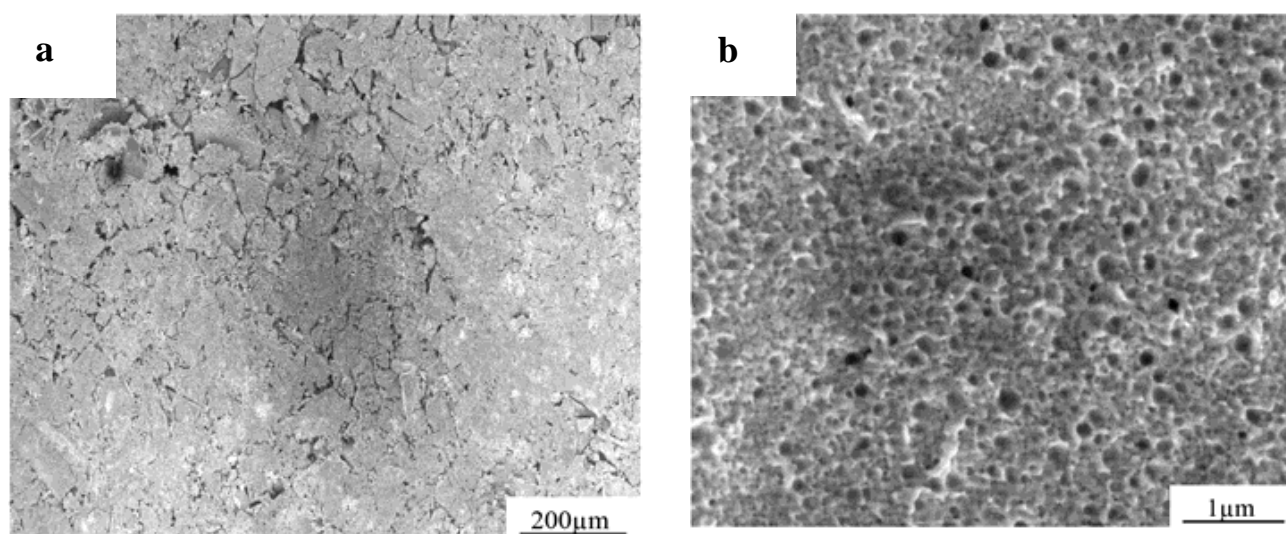


Fig. (6). SEM image of interface microstructure of joint using Ag nanoparticle powder: **a**-lower magnification **b**-higher magnification (bonding temperature: 200 °C; calculated bonding pressure:20 MPa; bonding time:30 mins).

the same conditions. This is because the actual bonding area was smaller than the lower surface of the Cu metal cylinder due to the coffee ring effect. This coffee ring area can be exactly determined. By using the coffee ring area, the estimated bonding pressure is about 80 MPa. Thus, the difference of the shear strength using Ag nanoparticle paste and Ag nanoparticle powder mainly comes from the different processing pressure applied for bonding.

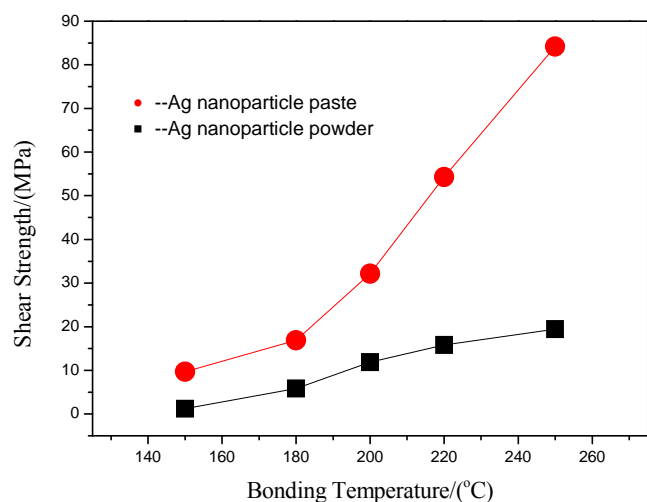


Fig. (7). Shear strength of joints which were bonded using Ag nanoparticle paste and powder at different temperatures.

3.4. Bondability and Challenges with Nanoparticle Paste

In general, as an ideal substitute for Pb-based solders, metallic nanoparticle paste must possess the features, including a good sintering-ability at relatively low processing temperatures, good flowability and viscosity for screen and stencil printing. Our results demonstrated that both the original chemically-reduced Ag nanoparticle paste without any adding of other organic components and the baked Ag powders at 60 °C can be used directly to bond the Cu substrate at low temperature, but an appropriate pressure must be provided in order to improve the bonding strength. Recently, Hu *et al.* reported that condensed Ag paste can bond Cu wires to Cu electrodes at a temperature as low as 100°C, very promising for polymeric flexible electronics [13, 14]. Hirose's group [9-11] proved that the good flowability on Cu and Ag or Au coating was achieved when appropriate solvents were added into the chemically-reduced Ag nanoparticles. High quality joints were achieved when the bonding area is smaller than the cylinder head surface of 5 mm in diameter. However, the bonding pressure generally at 5 MPa is still needed. Furthermore, up to today, few experiments are dedicated to explore whether organic components will inevitably remain in the sintered Ag layer when the bonded area becomes larger such as 100~200 mm² or more. In order to improve the bondability, Lu's group added three kinds of organic components into the commercial Ag nanoparticles, such as fish oil as dispersant, polyvinyl alcohol as binder and polymer with short hydrocarbon chains as thinner [12, 20]. The investigation indicated that the small area attachment of SiC chips to substrates with Ag, Au and copper can be metallized at about 275 °C without pressure. Meanwhile, for a large-area (>100mm²) chip attachment a designed multi-step heating

profile a pressure (about 5 MPa) is still needed to reduce the organic remains.

Besides, Maruyama and Ogura *et al.* [21, 22] developed a special paste to replace high-temperature Pb-rich solder, which contains a small amount of solvent, but primarily consists of micro-scale Ag powders with a diameter of 300 nm and alkoxide passivated Ag nanoparticles with an average diameter of 5 nm. Using this formula, the connection of Au-coated Si diode chips to Cu bases at 350 °C in N₂ without external pressure was realized, obtaining a diode package with electrical and thermal properties similar to those of Pb-5Sn soldered joints. However, the shear strengths about 12-14 MPa of joints of Cu cylinder with 5 mm in diameter to Cu plate bonded in N₂, only about half of the strength when soldered in air, and one third when soldered under a pressure of 1 MPa. Furthermore, although the pressureless sintering packaging has been achieved, the relatively high sintering temperature of 350 °C, low joint strength and N₂ ambient requirement are still issues for application. Further research for pressureless sintering packaging in air at relatively low bonding temperatures such as lower than 250 °C is imperative. The optimization of the particle size, (dispersant/surfactant, binder and thinner) and their amounts may be the key parameters for investigation.

Another challenge facing the extensive electronics packaging and assembly applications involves the lack of Ag metal and the relatively high cost of Ag nanoparticle and Ag nanoparticle paste preparation. The synthesis of Ag nanoparticle has been simplified by Hirose's research group [23]. They exploited a novel bonding process through *in situ* quick formation of Ag nanoparticles at only around 130 °C-160 °C by the chemical reduction action of Ag₂O microparticles with triethylene glycol as a reducing agent. Using the paste mixture of Ag₂O microparticles and triethylene glycol, joints with a 60 MPa tensile strength of Au-coated Cu cylindrical specimens and a dense sintered Ag layer were obtained with the bonding conditions of 250 °C temperature for only 5 min under a pressure of 5 MPa.

In the meantime, Morisada *et al.* developed another sintering-bonding technology to reduce the cost and the Ag ion migration, which would cause short circuit [24]. In their experiments, a special paste by mixing the 7.9 nm nanoparticles with 72.0 mass %Ag, or 498.0 nm nanoparticles with 98.4 mass %Cu and terpeneol was used to bond Cu to Cu cylinders with 5 mm in diameter. During the paste preparation, a constant mass ratio of 3:7 was used for the terpeneol: metal. The results indicated that it was difficult to obtain strong joints using Cu nanoparticles (actually the average size in microscale), while the addition of Ag nanoparticles into Cu nanoparticles significantly increased the joint strength. A shear strength of 50 MPa and a four time higher ionic migration resistance compared with counter-electrodes made only of Ag nanoparticles were obtained under the conditions of Cu50%-Ag50% metal ratio, and 350 °C bonding temperature for 5 min under 10 MPa. Particularly, the research group in University of Waterloo recently realized the sintering-bonding of 500 μm diameter Cu wire to Cu pad by directly utilizing the original chemically-reduced Cu nanoparticle paste [25]. The joint with a shear strength of about 14 MPa was obtained at only 200 °C under a pressure of 5 MPa. However, the oxidation

and thus the bonding stability of Cu nanoparticles are not well studied. Further systematical and profound investigations for this are desired.

4. CONCLUSIONS

Both original chemically-reduced Ag nanoparticle paste and the baked Ag powders at 60 °C can be used directly to bond the Ag-coated Cu substrates at low temperature and an appropriate pressure. A ring-like deposit was formed when dripping the paste on the substrate coating due to the coffee ring effect, while no ring-like deposit appeared when using the powders. High temperature was beneficial to sintering-bonding, and shear strengths of 20 MPa and 84MPa were obtained at bonding temperature of 250 °C for 30 min under 20 MPa when using the Ag paste and powders, respectively. High joint strength value when using paste is attributed to the high effective bonding pressure and small effective bonded area.

Challenges in nanoparticle paste sintering-bonding for packaging applications are as follows: 1) Improving the particle dispersion, flowability and viscosity for screen and stencil printing, 2) Greatly reducing or avoiding bonding pressure, 3) Lowering the sintering temperature and cost. Although the problems of oxidation and bonding stability urgently need to be resolved, Cu nanoparticle or Cu-containing nanoparticle mixture paste are relatively promising.

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