Study on layer formation behavior of Ag joints sintered with pressureless sintering process

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Abstract
This study focuses on investigating the relationship between sintering performance and bond layer thickness for the hybrid Ag paste consisting of micrometer-sized Ag particles and sub-micrometer Ag spherical particles. The surface morphology showed that the sub-micrometer Ag particles would contribute to improving the densification and bonding strength of the sintered Ag joint. The x-ray results indicated that the bonding quality was achieved when the bonding layer thickness was 50 μm, whereas a thinner or thicker bonding layer would lead to cracks or voids. Moreover, the microstructure of sintered Ag joints with different bonding layer thicknesses was found to be in good agreement with the results of x-ray by SEM observations. There are many cracks in the sintering structure due to the insufficient Ag paste when the bonding layer thickness is below 50 μm, while more voids appeared in sintered Ag joints when the thickness reached 120 μm, which was caused by the organic solvent volatilization. In addition, the poor densifications lead to low strengths of 7.6 MPa and 23.42 MPa for 15 μm and 30 μm thickness, respectively. The shear test results revealed that the highest shear strength was achieved on the bonding layer with a 50 μm-thick bonding layer. However, the bonding strength would decrease to 24.67 MPa and 18.64 MPa when the bonding layer thickness was 90 μm and 120 μm, respectively. The SEM observations indicated that many dimples were formed on the fracture surface with a thickness of 50 μm, which was the reason for the enhancement of bonding strength in the sintered Ag joint.

1. Introduction

With the emerging and proliferating interest in newly-developed wide band gap semiconductors such as SiC and GaN, power electronic devices can operate at high temperatures (greater than 250 °C), offering fast switching and lower energy losses, and higher breakdown voltages [1–3]. During the operation process of high-power electronic devices, the die-attach module suffers repeated heating and cooling, and shear stresses appear parallel to the bonding interface due to the mismatch of thermal expansion between the semiconductor die and the substrate. Previous studies have pointed out that solder alloys [4–6] and conductive adhesives [7–9] were not suitable for long-term operation in such harsh environments, due to brittle intermetallic phases for solders and low thermal and electrical properties for conductive adhesives. It has been demonstrated that the sintered Ag paste possesses superior properties, such as high electrical and thermal conductivity, high melting point, low sintering temperature, and excellent reliability [10, 11]. As an alternative replacement for conventional die-attach materials, sintered Ag paste is being extensively studied to deliver maximum efficiency, outstanding performance, and long-term package reliability in high-temperature and high-reliability applications.

The sintered Ag paste is usually composed of Ag particles and organic solvents, and a stable structure can be formed at a low temperature due to the micro-size effect of Ag particles and the assistance of organic solvents. It was reported that Ag nanoparticles paste typically requires high pressure and heating during the sintering process to achieve stable bonding, which is mainly owed to the organic dispersant or protection layer on the
particle surface for preventing spontaneous agglomeration [12]. However, the pressure-assist sintering process would damage the chip surface with the reduction of chip size, and the fabrication process is more complex. Therefore, pressureless sintering has become the main technology pathway for sintered Ag paste in recent years. In order to achieve the high reliability of sintered Ag joints under the pressureless sintering process, micrometer-sized or hybrid Ag particles are more attractive than nanoparticles because they contain fewer organic additives [13–16]. Compared with spherical Ag sub-micrometer particles, hybrid particles with Ag micro-flakes and nanoparticles applied to die-attach sinter bonding have demonstrated higher shear strength in the pressureless sintering process due to larger necking growth [17]. Moreover, another advantage of hybrid particles is higher resistance to mechanical creep and cracks during thermal cycling.

In addition to Ag particles size, sintering temperature and heating rate are also important factors affecting the properties of pressureless sintered Ag joints. It should be pointed out that surface diffusion is the dominant mechanism in low-temperature sintering, while grain boundary or lattice diffusion dominates in high-temperature sintering [18]. These researches showed that low sintering temperature will affect the densification of the bond layer, and high sintering temperature will lead to grain coarsening [19, 20]. Some studies also reported that a higher heating rate would be conducive to minimizing the aggregation of Ag particles during the high sintering temperature [21, 22]. On the other hand, the heating rate should be low enough to allow the adequate outgassing of the solvent without disrupting the densification behavior of the bonding layer [23]. Therefore, a suitable pressureless sintering process is particularly important for obtaining high-reliability sintered Ag joints.

Apart from using the experimental method to study the properties of sintered Ag joints, numerical simulation is also a favorite method for predicting reliability variation. By means of the numerical simulation investigation, it has been proved that thermodynamic properties and cracking behaviors of sintered Ag joints heavily rely on the bonding layer thickness. Villacarlos et al [24] reported that it is possible to experience adhesive or cohesive failure if the bonding layer thickness is lower, due to higher principal stress and strain on the die. Qin et al [25] found that sintered Ag layer thickness significantly affected the energy release rate of the interfacial crack and vertical crack, and they concluded that the thinner sintered Ag layer had a higher cracking risk. Although some numerical simulations on bonding layer thickness have been carried out, the thickness effect on Ag sintered joints microstructure and mechanical properties is still unclear. In this paper, the thickness of sintered Ag joints ranging from 15 μm to 120 μm was used in die-attachments under the pressureless sintering process. Both interface microstructure and the shear fracture behaviors of the bonding layer were analyzed for a comprehensive understanding of the reliability of the sintered Ag joints.

2. Experimental procedures

In this study, we used the sintered Ag paste that was acquired from Nihon handa Co. Ltd. The sintered Ag paste contained micrometer-sized Ag particles and nanomicrometer Ag spherical particles, which mixed with an organic solvent system to prevent the Ag particles from agglomerating and aggregating, as shown in figure 1(a). Based on a previous study [26], the gaps among large-size Ag particles would be filled by Ag nanoparticles, which allow the sintered Ag layer to obtain a high density and excellent electrical conductivity. Prior to Ag sintering,
500 nm Ni and 200 nm Au was sputtered orderly on the back side of the Si die chip (4 mm × 4 mm × 0.3 mm), and 3 μm thick Ni and 1 μm Au were electroplated on the top side of the direct bonding copper (DBC) substrates (10 mm × 10 mm × 3 mm). A temperature profile was applied without pressure during the sintering process in the N₂ atmosphere, as shown in figure 1(b). In the sintering process, the specimens were preheated at 150 °C for 30 min to evaporate the most solvent for reducing the porosity. Then the specimens would be heated to 250 °C and held for 90 min for sintering. Finally, the sample would be allowed to cool down.

The sintered Ag paste was evaluated under two different conditions, one uncovered and one covered. For the uncovered state, the sintered Ag paste with 50 μm thickness was printed onto a glass substrate whose dimensions were 9 mm² in order to study its surface and cross-sectional morphology. In addition, simulated die-attach structures were used to investigate the bond layer formation behavior of sintered Ag joints. The sintered Ag paste with various thicknesses was stencil printed onto the metalized DBC, after which the Si chip was mounted. To calculate the shrinkage of the sintered Ag paste, the height of each printed Ag paste and corresponding sintered Ag joint were scanned by a step profiler (XP-200) before and after sintering, respectively, as shown in figure 2. After sintering, there were five thicknesses sintered Ag joint, namely 15 μm, 30 μm, 50 μm, 90 μm, and 120 μm.

As a non-destructive examination, x-ray machines (XSCAN-H160-OS, XAVIS, Korea) were used to observe cracks and voids in sintered Ag joints. For destructive inspection, an ion-milling polishing machine (IM4000, Hitachi, Tokyo, Japan) was used to prepare the cross-sectional joints with sintered Ag joints of various thicknesses. A scanning electron microscope (SU8020, Hitachi) was used to observe the surface morphology and microstructural evolution of sintered Ag joints of different thicknesses. As for the die-attach module shear strength test, five samples of each thickness of sintered Ag joint were tested by a bond-tester (Dage4000 bond tester, Noran DAGE, Aylesbury, UK) at a speed of 30 μm s⁻¹ and a height of 30 μm, and the average shear strength of five sintered Ag joint structures was determined for each type.
3. Results and discussion

3.1. Surface morphology and sintering mechanism

Figure 3(a) shows the surface image of the sintered Ag paste after sintered, according to the Ostwald ripening theory, Ag particles coalesce into larger particles by grain boundary diffusion as well as lattice diffusion [27]. Based on the overall morphology, it can be seen that hybrid Ag paste has developed into a uniform and continuous porous structure, as depicted in figure 3(b), which suggests that the sintered Ag paste possesses a favorable sinterability at a pressureless and low-temperature environment.

The sintered morphology of Ag pastes is important for high-strength joints, so an understanding of the sintering mechanism of Ag paste is essential for the packaging and manufacture of power electronic devices. Based on the previous study [28], the densification of the sintered Ag layer was dependent on the decomposition of organics and the diffusion mechanism. Figure 4 exhibits the sintering schematic diagram of the sintered Ag paste. Firstly, the mixed Ag particles were evenly distributed in the organic solvent under the action of dispersants. As the sintering temperature increased, the dispersants gradually decomposed, and the micrometer...
Figure 6. Cross-sectional images of sintered Ag joints with various bonding layer thickness. (a) 15 μm, (b) 30 μm, (c) 50 μm, (d) 90 μm, (e) 120 μm.
Ag particles came into contact with each other driven by high surface energy. On the other hand, because of diffusion in the lattice and grain boundaries, Ag nanoparticles were incorporated into the micrometer Ag particles, which was also used as a bridge for forming necks between the micrometer Ag particles. Finally, a dense and uniform sintered structure was obtained after removing all organic material.

3.2. Microstructure of various bond layer thickness

As noted in previous studies \[11, 29\], the porosity of the sintered Ag joint has a strong correlation with the bondline thickness. Therefore, it is necessary to study the voids variation of sintered Ag joints on various bond layer thicknesses. After the pressureless sintering process, the specimens of the sintered Ag joints bonding Si chip on the DBC substrate were characterized with x-ray scanning inspection to identify voids and cracks, as shown in figure 5. Generally, voids or cracks would present white sites, while void-free regions feature black shades. It was observed from figures 5(a) and (b) that some micro-cracks are located at the edge of sintered Ag joints when the bonding layer thickness is thin. As the bonding layer thickness increased to 50 μm, the sintered Ag joint was well fabricated with no void or crack, as shown in figure 5(c). On the other hand, voids appeared when the bonding layer thickness reached 90 μm, the number of voids increased gradually with the increase of the bonding layer thickness, and the porosity toward the edge was lower than the center, as presented in figures 5(d) and (e). From the x-ray results, it was confirmed that the bonding layer thickness would be controlled within an appropriate range in order to obtain a dense sintered Ag joint structure.

The variation of voids associated with a thinner bond layer thickness could be attributed to the tension of bonding \[29\]. During the sintering process, the dimension of both the chip and DBC substrate did not change. However, the sintered Ag layer displays shrinkage behavior, due to the continuous volatilization of organic solvents and the emergence of the necking effect for Ag particles. Based on the thermomechanical simulation result \[30\], the lower bond thickness of sintered Ag layer triggers higher principal stress and strain on the joint. Therefore, the shrinking thickness of the Ag paste and the fixed dimension of the Si chip and DBC substrate inevitably resulted in the tension within the joint. As a result of the tension applied to the Ag joint, the shrinking tendency of sintering was suppressed and consequently resulted in a higher porosity. If a thicker bond layer thickness, such as 90 μm, is generated, this may be caused by the outgassing factor at sintering \[31\]. Based on the previous study \[32\], due to the slow evaporation of the solvent for thicker Ag paste layer, the sintering action is not completed in time, resulting in regions with a high level of voids in the sintering process. During sintering, the organic materials vaporized, which caused the vapor to exit toward the edge. Since the vapor will exit at the edge first, it will diminish from the edge first, thus resulting in a lower porosity at the edge.

SEM cross-sectional observations were made to investigate the reasons for the difference in x-ray images with different bond layer thicknesses. The SEM images with the bonding layer thickness of 15 μm, 30 μm, 50 μm, 90 μm, and 120 μm are shown in figures 6(a)–(e), respectively. As shown in figure 6(a), a micron-sized porous network structure was formed by Ag grains necking growth. With a magnified view of sintered Ag layer,
there are many voids among the sintering structure. The cross-sectional morphology of the 30 \( \mu \text{m} \) thickness bonding layer is shown in figure 6(b). Although the sintering Ag layer showed a denser microstructure, there were still some small micro-voids due to the lack of hybrid Ag paste during the sintering process. When the bonding layer thickness is increased to 50 \( \mu \text{m} \), a sound-sintered Ag joint was achieved with the densest microstructure, as shown in figure 6(c). However, as the bonding layer thickness increased to 90 \( \mu \text{m} \) and 120 \( \mu \text{m} \), because the removed path of the solvent became longer in the sintering process, more voids defects were formed in the bonding layer, as illustrated in figures 6(d) and (e). These observations are consistent with the x-ray detection results.

### 3.3. Mechanical properties of various bond layer thicknesses

The mechanical properties of the various bonding layer thickness were evaluated by the shear strength test. Figure 7 depicts the bonding strength of the sintered Ag joints with different bond layer thicknesses. The bonding strength of the specimen with 15\( \mu \text{m} \) thickness only has 7.6 MPa, and the bonding strength reached 23.42 MPa and 29.46 MPa, which corresponded to the thickness of 30\( \mu \text{m} \) and 50\( \mu \text{m} \), respectively. However, the bonding strength gradually decreased with the increase of bonding layer thickness over 50\( \mu \text{m} \), and the bonding strength decreased maximally by 36.7% when the thickness increased by 120\( \mu \text{m} \).
In order to further study the variation of shear strength for various bond layer thicknesses, SEM images of the fracture surface of the specimens are shown in figure 8. As can be seen in figure 8(a), the fracture surface of the sintered Ag joint for 15 μm thickness is covered by scattered Ag particles. With a magnified view of the fracture surface in figure 8(b), there are few dimples representing ductile fracture on the fracture surface, indicating that the breaking occurred at the interface between the Ag layer and DBC substrate. Figures 8(c) and (d) showed the fracture surface for the 50 μm thickness bonding layer, and the fractured surface indicated a typical ductile necking for sintered Ag joint. In this case, it is clear that Ag particles were perfectly sintered, and the presence of pore coarsening showed that sintering took place. Moreover, all other organic substances mixed in the paste evaporated, and only the porous network structure consisting of Ag particles remained at the joints. For this reason, the shear strength of the Ag joint with 50 μm bond layer thickness was higher than that of the thinner Ag joint. In the case of the specimen with the bonding layer thickness of 120 μm, although there still are a few dimples on the fracture surface, the fracture surface shows an obvious coarsening phenomenon in particle necking. It has been reported [31, 33] that the void formation is the increased amount of solvent due to thicker bond layer thickness, which rapidly evaporated from the edge but could not easily escape from the joint center. This in turn inhibits the initiation of necking between the particles and delays densification.

4. Conclusions

In this work, the correlation between microstructural evolutions, mechanical properties, and bonding layer thickness was investigated. For the sintering process, the presence of Ag nanoparticles was critical in the formation of sintering necks bridging due to the high surface energy, which increased the density of the bonding layer. The cross-sectional images revealed that the microstructure of sintered Ag layer is directly affected by the bond layer thickness. When the thickness is relatively thinner, there are more cracks formed at the edge of the die-attach module, which is caused by tension of bonding. A considerably dense and uniform structure was obtained by the thickness of 50 μm. However, the microstructure showed a deterioration phenomenon with the increase of bond layer thickness. In addition, the bonding strength can also be related to the bond layer thickness. The bonding strength is 7.6 MPa for 15 μm thickness, while the bonding strength increased to 29.46 MPa with a thickness of 50 μm. Nevertheless, the shear strength would decrease when the thickness is relatively thicker. The bonding strength variation can be explained that the appropriate thickness sintered Ag layer would produce many dimples for ductile fracture, while a thinner or thicker sintered Ag layer possesses cracks or pores, which would lead to the transformation from ductile fracture to brittle fracture.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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