

INVESTIGATION OF COPPER SINTER MATERIAL FOR DIE ATTACH

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ABSTRACT

Die attach on power semiconductor using lead-free technology has attracted considerable interest. Silver sintering has been considered as one of the most promising lead-free die attach solutions over the past years. Pressure silver sintering by far offers superior thermal and electrical conductivity properties which enables power electronics applications to operate at higher temperature. Although silver sintering is a promising technology, the high cost of silver increases the manufacturing cost and consequently impedes the wide adoption of this technology.

Copper is commonly used in the electronics industry mainly due to its unique properties such as good thermal and electrical conductivity. Hence, it is not surprising that recently there are several academic and industrial researches using copper as an alternative die attach material for sintering process. Nevertheless, copper sintering is usually requires an additional reducing process using hazardous gases such as hydrogen and formic acid to prevent copper oxidation as well as to increase the bonding strength.

In this study, we develop a safe-to-use micro-copper sinter paste for pressure sintering under inert atmosphere.

Key words: copper sintering, die attach material, power electronics

INTRODUCTION

The increasing demand for power electronics packaging with higher power density, higher voltage and smaller size requires the replacement of silicon dies by wide band gap semiconductor devices such as silicon carbide and gallium nitride [1].

Because of their low melting temperature, solder materials are facing a challenge to use as die attach materials for the wide band gap semiconductor devices which require high operating temperature. In recent years, silver sinter materials have attracted considerable attention as die attach materials for high temperature application not only because of its high melting temperature (961°C) but also it possess excellent properties such as high thermal and electrical conductivity. Besides the advantage of high melting point, silver sinter materials do not contain hazardous substances such as lead, mercury and cadmium.

Several studies have demonstrated the feasibility of silver sinter materials as interconnect materials for die attach application and further confirmed silver sinter materials as high reliable interconnect materials via their long term

reliability tests [2–4]. Previous studies revealed high reliable silver sintered joint can be obtained by pressure sintering process with relatively short process time and the increased lifetime of sintered modules enable a cleaner production [5–10]. Silver sinter materials are currently commercially available and additionally the pressure sinter equipment manufactured for high volume production have been increasing over the past years.

High material cost of silver hinders the wide adoption of silver sintering technology despite of its unique properties and high reliability. In order to keep the manufacturing cost as low as possible, it is essential to look for alternative interconnect materials which have lower material cost but relatively similar properties to silver. A number of researches have considered copper as an attractive alternative material because of its relatively low cost and it possess similar thermal and electrical conductivity to silver [11–16]. Copper can be referred as a standard material in electrical engineering, which can be found as a conductor material in electronic assemblies and especially cables. Copper is also used in power electronics especially in the metallization of ceramic substrates and lead frames. It is well known for decades that copper possess several strengths such as good current carrying capacity and high melting point but also weaknesses such as the susceptibility to oxidation.

Recently, copper has been scientifically studied as an alternative interconnect material for die attach applications. The main focus of the studies is using copper-nano particles, which has been reported to be able to form sinter joint at low temperatures by pressureless sintering, however, it often requires complex synthesis process [17–22]. The environmental and health consequences of nanoscale particles are often part of intensive public and scientific discussions which led the European Chemicals Agency (ECHA) to form a group seeking for common ground among experts on scientific and technical issues relating to the implementation of regulations for nanomaterials such as Biocidal Products Regulation (BPR).

In addition to the nanoscale materials, micro-scale copper powder is also actively investigated as an interconnect material. In the case of micro-scale material, a pressure sintering method is often applied. The die attachment on direct copper bonded (DCB) substrates by pressure sintering of copper particles was reported in 2012 [23]. In this study, it has been reported that reduction steps are necessary to remove oxides from the copper surface by using hydrogen or other reducing agents in order to

connect the electronic components to the substrate using copper particles in the sintering process.

Recent results show that for a pressureless sintering process, reduction of copper oxide under a component is usually insufficient to create an interconnection joint with high bonding strength [24]. Besides the concentration of the reduction agent, the sintering temperature also play an important role in determining the bonding strength. [25]. The use of copper as the die attach material still facing many challenges such as copper oxidation and long term reliability and additionally the introduction of reduction processes is challenging for high volume production environment as investment in suitable equipment and special safety handling are required.

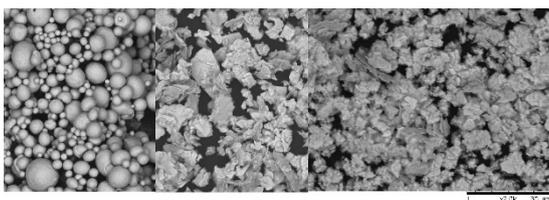
The aim of this study is to investigate the feasibility of using copper as an interconnect material without a prior application of a reduction process.

EXPERIMENTAL

In this study, different copper powders were used to prepare copper pastes. The copper pastes were then used to sinter copper strips and also used as interconnect materials to attach two copper foils as well as to bond Si dies on DCB substrates. To evaluate the copper pastes, several tests were performed on the samples and the detail are described in the following sections.

Copper paste

Copper pastes used in this study were made by mixing copper powder with organic solvents. Different copper powders were evaluated in this study. One spherical-shaped copper powder and two flake-shaped powders were sourced for the experimental tests as can be seen in Figure 1. The particle size D50 of the flake-shaped powder 1 and 2 is specified as 3.6 μm and 3.3 μm , respectively. The two copper flakes types differ in particular by the coating around the powders.



a) b) c)

Figure 1. SEM images of a) spherical copper powder, b) copper flake 1 and c) copper flake 2 with 2000x magnification

Three copper pastes with a metal content of about 75 wt% were prepared by mixing the micro-scale copper powders shown in Figure 1 with organic solvents and subsequently the pastes were milled using a three-roll mill to ensure a better dispersion of copper powders in the paste.

Sintering of Copper strips

Copper strips were prepared by printed copper paste on Al_2O_3 ceramic substrates using stencils with a thickness of 200 μm . The printed copper strips with a dimension of 40 mm x 10 mm were dried at 353 K in an oven to

remove the organic solvent. After drying, the samples were pressed at room temperature and 573 K for 3 min with a pressure of 10 MPa under N_2 atmosphere. During the sintering process, the copper strips were covered by a polytetrafluoroethylene (PTFE) foil to protect the press stamp.

After pressing, the copper strips were removed from the ceramic substrate and examined by optical microscope and SEM. After metallurgical pretreatment, Cross-sectional pictures were evaluated using Hitachi TM-1000 scanning electron microscope (SEM) coupled with Burker energy dispersive X-ray spectroscopy (EDX).

Specimen for tensile shear test

Copper foils were cleaned with citric acid to reduce the oxide layer which may have formed during storage. The copper paste was printed on the copper foil using a stencil with a dimension of 10 mm x 10 mm and a thickness of 200 μm . The printed paste was then dried at 353 K under air atmosphere. Afterward, a second copper foil was placed on the dried copper paste and the assembly was pressed at 573K under N_2 atmosphere with a pressure of 10 MPa.

For characterization of the powder solidification, the tensile shear strength of the bonding layer was measured. For this investigation, two copper foils with a dimension of 40 mm x 10 mm x 1 mm were attached using the copper pastes by pressure assisted process (Figure 2.a) and subsequently conducted destructive tests using Shimadzu EZ-L tensile testing machine (Figure 3a).

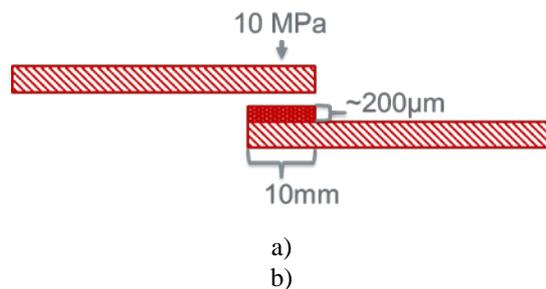
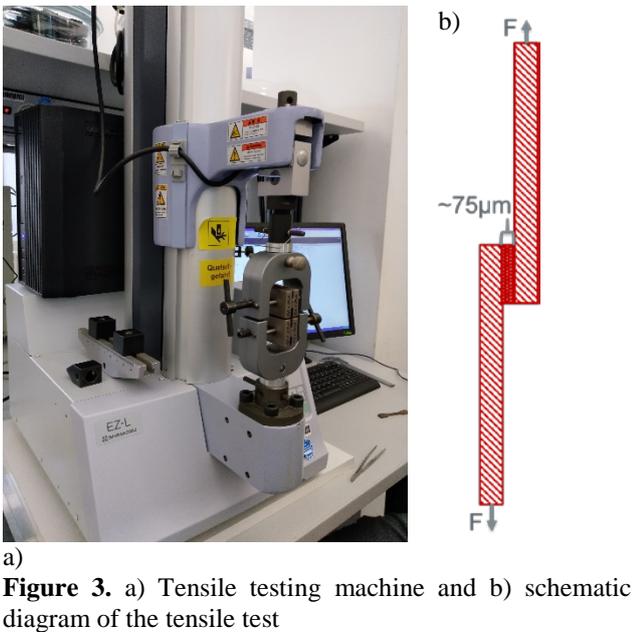


Figure 2. a) Schematic diagram of sample prepared for the tensile shear test, b) bonded copper foils sample



a) **Figure 3.** a) Tensile testing machine and b) schematic diagram of the tensile test

The assembled test specimens (Figure 2.b) were clamped vertically in the tensile testing machine (Figure 3a) and the samples were loaded until breakage at a constant test speed of 1 mm / min. The shear force was calculated from the maximum measured force and the area of the printed paste deposit. After tensile test, the failure mode was observed by optical microscope.

The measured tensile shear strength and the fracture patterns are used to evaluate the interconnection between the copper sintered layer and the surface of the copper foil.

Preparation of Die attach samples

The copper pastes produced in this study were also used as interconnect material to attach dies on DCB substrates. In addition to the previous three copper pastes, a paste with a mixture of the two flake-shaped powders (copper flake 1 and copper flake 2) with a ratio of 1:1 was prepared and used as interconnect material.

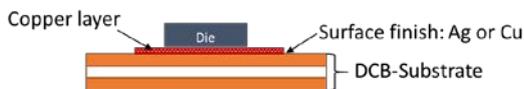
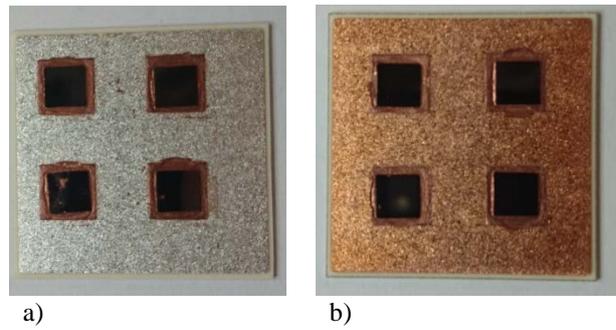


Figure 4a. Schematic diagram of the die attach samples

The assembly process for the die attach samples is based on the methods and common procedures of silver sintered materials. The copper sinter paste was printed on the substrate using a stencil with a thickness of 200 μm and then dried at 353 K in an oven for 30 min to remove the organic solvents. Subsequently, Ag metallized silicon dies with a size of 4 mm x 4 mm were attached to the dried copper paste with a placement force of 2000 g for 4 seconds. Subsequently, the sample was pressed at 573 K with a pressure of 15 MPa under N_2 atmosphere for 3 min.



a) **Figure 4b.** Die attached on DCB substrates with a) silver metallization and b) bare copper surface using copper pastes

The bonding strength of the copper layer was characterized by means of a destructive die shear test. Die shear measurement is a standard test method to determine shear strength of bonding materials in accordance with MIL-STD-883. It is based on a measure of force applied to a semiconductor die attached to a substrate using copper paste as a bonding material. Figure 5 shows the schematic diagram of die shear test. A Nordson Dage 4000 plus machine (Figure 6) was used to measure the shear strength of assembled samples.

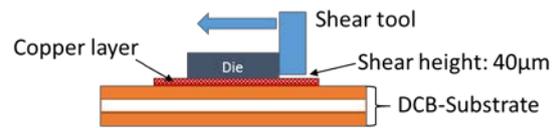


Figure 5. Schematic diagram of die shear test

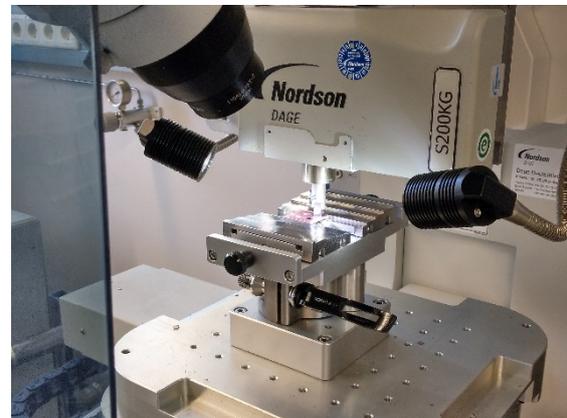


Figure 6. Die shear machine with test specimen

After the die shear test, the failure modes of the sintered layer were evaluated by optical microscope and SEM.

RESULTS

Investigation of Copper strips

Figure 7 a) and b) show the copper strips after pressing at room temperature under N_2 atmosphere for 3 min. Parts of the copper strips were peeled off during the removal of the PTFE foil. After pressing at room temperature, powder solidification could neither be detected for the spherical nor the flake-shaped copper powder.

The process temperature of 573 K showed heterogeneous results. After pressing at 573 K, the copper strip consists of spherical copper powder (Figure 7 c) was mainly removed together with the PTFE foil. The fracture behavior of the spherical copper powder indicates that no compound was formed. On the other hand, the flake-shaped particles (Figure 7 d) solidify into a copper strip without any fragments or visible defects of the copper layer. Some of the copper strips consist of the flake-shaped particles, a dark discoloration was observed partly at the edges indicating the oxidation of the copper surface. Simple mechanical processing shows that the dark discoloration of the copper takes place only superficially.

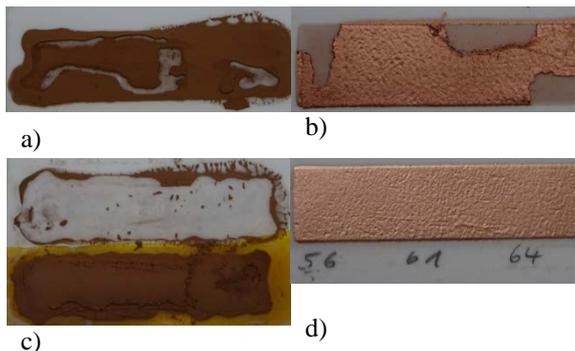


Figure 7. After pressing at room temperature, copper strips with a) spherical powder and b) copper flake 2. After pressing at 573 K under N₂ atmosphere, copper strips with c) spherical powder and d) copper flake 2.

The results above show that it was not possible to produce copper strips with spherical-shaped copper powder as well as by pressing at room temperature. As a result, the evaluation of bonding strength was only conducted for the copper strips consist of the two flake-shaped copper powders after pressing at 573 K.

Figure 8 shows the SEM images with a 1000x magnification of the cross sections of a copper stripe consisting of copper flake 1 produced at 573 K under N₂ atmosphere with an additionally enlarged detail with a magnification of 5000x. The image shows that copper flake 1 still has distinct pores in the layer, the contour and orientation of the individual copper flakes can be seen clearly as well.

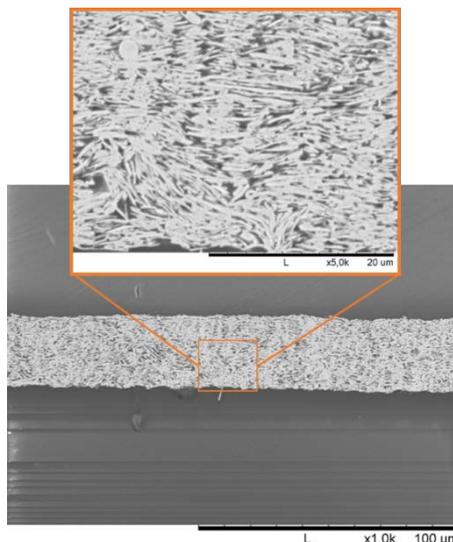


Figure 8. SEM images of the cross section of a copper stripe with copper flake 1 with 1000x magnification and enlarged with 5000x magnification

Figure 9 shows the SEM images with a 1000x magnification of the cross section of a copper stripe of copper flake 2 produced at 573 K under N₂ atmosphere and additionally enlarged detail with a magnification of 5000x. It can be seen that the copper layer appears to be more compact with less and smaller pores and in contrast to Flake 1, the contour of the copper flakes 2 is less noticeable.

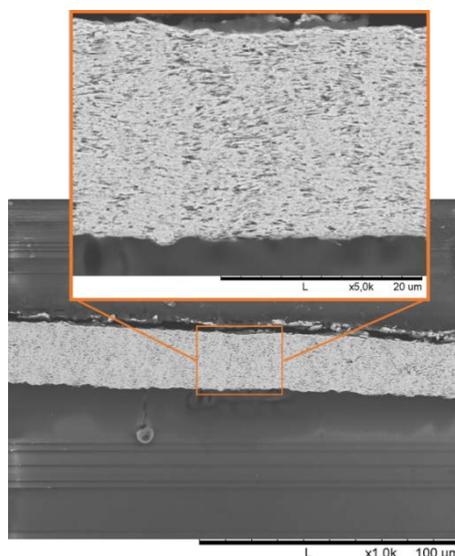


Figure 9. SEM images of the cross section of a copper stripe with copper flake 2 with 1000x magnification and enlarged detail with 5000x magnification

Results of Tensile Shear Test

After pressing, the copper layer between the copper foils resulted in 75 µm thickness. The results of the tensile shear test are shown in Figure 10. For the spherical copper powder, no strength values could be determined, since all the specimens were destroyed before or during handling and clamping in the tensile testing machine. The copper flake 1 and 2 achieved different shear strength. 5 samples were sheared to generate an individual box plot. Figure 10

shows that copper flake 2 achieved an average shear strength of 3.2 N/mm² which is higher than copper flake 1 with an average shear strength of 1.2 N/mm².

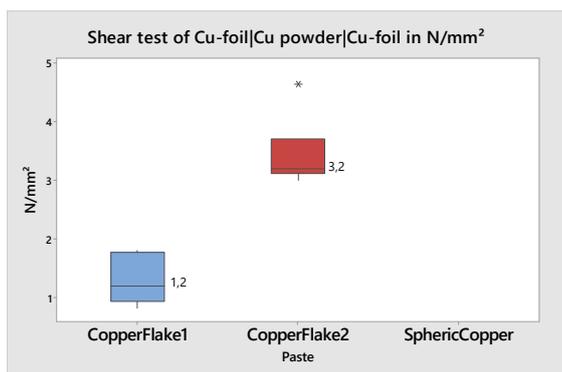


Figure 10. Tensile shear strength values of the connected copper foils

In order to compare with silver sintered material, tensile shear measurements were also performed on the samples using commercially available silver sintered material as interconnect material. The results reveal that the silver sintered material achieved an average shear strength of 9.8 N/mm², which is higher than the shear strength achieved by the samples prepared by copper material.

Figure 11 and 12 show the fracture patterns of the copper foil after the tensile shear test. The optical images of the fracture patterns illustrate a cohesive break in the copper layer was obtained for the pastes consist of the flake-shaped copper powders where the copper layers can be found on both copper foils (Figure 11a & b). In addition, discoloration in the copper layer was observed from the optical images demonstrating the oxidation of copper layer. In contrast, adhesive break was obtained for the paste consists of the spherical-shaped powder where copper layer was only found on the printed copper foil (Figure 12).



Figure 11. Fracture of the joints with a) spherical powder b) copper flake 1 and c) copper flake 2 on the copper foil



Figure 12. Fracture of the joints with spherical powder on the copper foil

Microscope images (Figure 13) further confirm the pastes consist of flake-shaped copper powder achieved a cohesive failure mode. The images demonstrate that the failure pattern of the paste consists of flake-shaped copper powder (Figure 13a) and the commercially available silver sintered material (Figure 13b) are relatively similar where the copper layer can be found on the unprinted copper foil.

In addition to the fracture images, the microscope images on some samples show a darker discoloration of the copper layer (Figure 13a). The sign of oxidation can be found mostly on the edges of the bonding layer and was not observed at the same area in each sample.

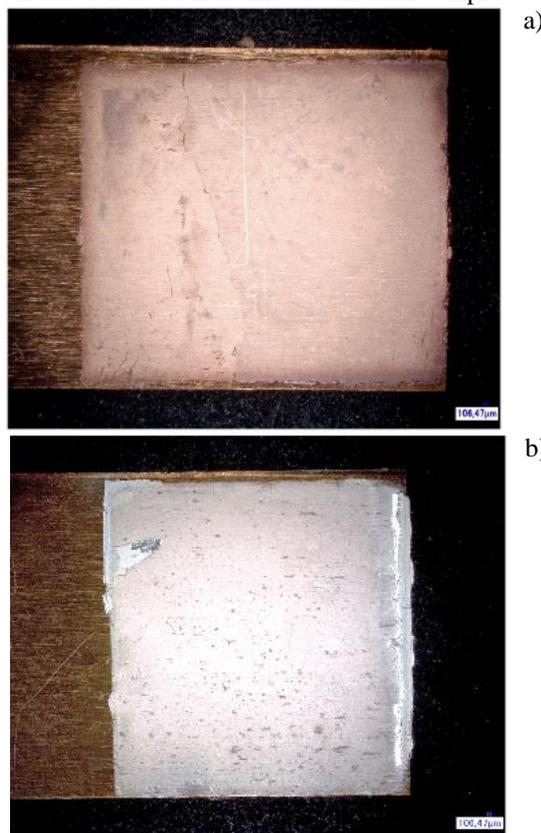


Figure 13. Microscope images of the residues of pastes with a) copper flake 2 and b) silver sinter material on the unprinted copper foil

This observation suggests that the oxidation does not start from the center of bonding layer but it occurs from the outside. Presumably, the oxidation caused by the remaining oxygen content during the sintering process or during the cooling process without continuous supply of nitrogen.

Results of Die Shear Test

The die shear strength was conducted for the die attach samples to evaluate the bonding strength of copper sintered joint. It was not possible to measure die shear strength for the samples assembled by the paste with spherical copper powder because the silicon dies peeled off immediately after pressing at 573 K. This observation illustrates that the copper paste made by spherical copper powder is not able to create interconnection joint between die and substrate.

The results of the shear strength measurement are shown in a box plot diagram (Figure 14). 12 dies were sheared off for each paste to generate an individual box plot. It can be seen from Figure 14 that copper flake 2 achieved the highest die shear strength ($> 30 \text{ N/mm}^2$) among the three copper pastes, whereas, copper flake 1 obtained the lowest die shear strength ($< 10 \text{ N/mm}^2$). The paste with the mixture of copper flakes 1 and copper flake 2 achieved the average die shear strength above 15 N/mm^2 . The results indicate that the interconnection joint created by copper flake 1 is poorer than that by copper flake 2. The lower die shear strength of the paste with the mixture of copper flakes might be attributed to the poor adhesion of copper flake 1. A similar trend was observed for all 3 copper pastes in which there is no significant difference in the average die shear strength for Ag metallized and bare copper substrates.

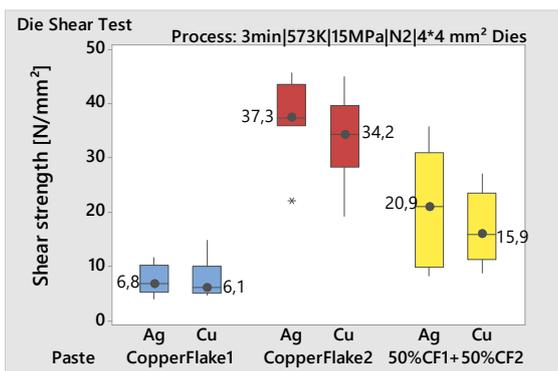


Figure 14. Boxplot of shear strength for silver and copper DCB substrate

The material contrast of the silver metallized surface and the copper die attach material facilitates the interpretation of the fracture patterns by light microscope or SEM and the detection of copper by EDX. For the samples on copper substrate, the interpretation is therefore more difficult.

After die shear tests, the substrates sintered by the paste consists of copper flake 2 were examined by optical microscope and SEM. The results reveal that cohesive break in the copper layer were obtained and this observation explains the high shear strength achieved by the paste consists of copper flake 2.

Figure 15 shows the die shear failure mode and it can be seen that mostly cohesive break in the copper layer was obtained where a copper layer can be found on the die backside and also on the substrates surface after die shear tests. Only in some corners of the die attach area the

surface material of DCB substrate becomes visible (area A shown in Figure 15a).

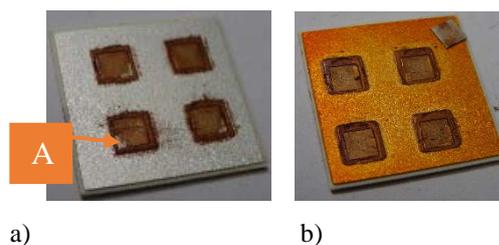


Figure 15. Die shear failure mode of a) Ag metallized substrate b) bare copper substrate

Figure 16 illustrates the SEM image of the Ag metallized substrate after die shear test. The areas marked with A and B in Figure 16 are the silver metallization and the remaining copper joint, respectively. With a magnification of 1000x, the image shows that even in the corners, where the metallization of the substrate optically emerges, adhesion of copper to the surface is still observed.

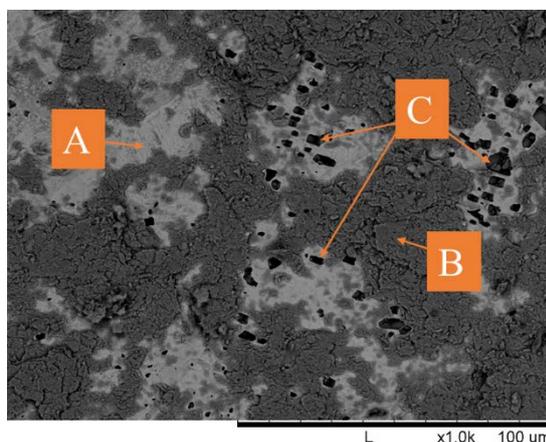


Figure 16. SEM image with a magnification of 1000x of the fracture mode of silver metallized substrate after die shear test

In addition, holes on the surface of Ag metallized DCB substrate can be seen from the SEM image (area C marked in Figure 16). These defects could not be found on the surface of the original Ag metallized DCB substrate before sintering. The holes were only observed on the Ag metallized DCB substrate sintered by the paste consists of copper flake 2. It is believed that the Ag metallized layer was peeled off during die shear test which created the holes on the substrate surface. Presumably, this phenomenon is caused by the strong bonding strength of copper joint on the Ag metallized DCB substrate.

CONCLUSION

In summary, 3 different copper powders were tested for their feasibility to use as die attach material for pressure sintering process without prior treatment with reducing agents such as hydrogen or formic acid. The results illustrate that spherical-shaped copper powder was not able to create copper joints by pressure sintering process

at 573 K with a pressure of 10 MPa for 3 min under N₂ atmosphere. In contrast, it was possible to create copper joints by pressure sintering process using flake-shaped copper powders. The shear strength and the failure mode results show that copper flake 2 created stronger copper joint than copper flake 1. This study demonstrate that it is feasible to use copper paste as interconnect material for die attach application by pressure sintering process, even without reducing agents.

Nevertheless, this research is still ongoing and further improvements are needed. Copper oxidation on the outer edges of copper strips was observed and it is highly likely that a fluctuating or insufficient nitrogen flow during sintering or cooling process led to the copper oxidation. As a result, an improvement of the nitrogen supply will be part of our further investigation. Additionally, as can be seen from the results that different copper powders achieved different bonding strength of copper joint. Hence, sintering behavior of different copper powders will be further investigated in our study. Furthermore many investigations on mechanical and electrical properties of sintered copper material as well as studies on environmental simulations by aging will be done to evaluate copper sintering as a die attach technology.

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