

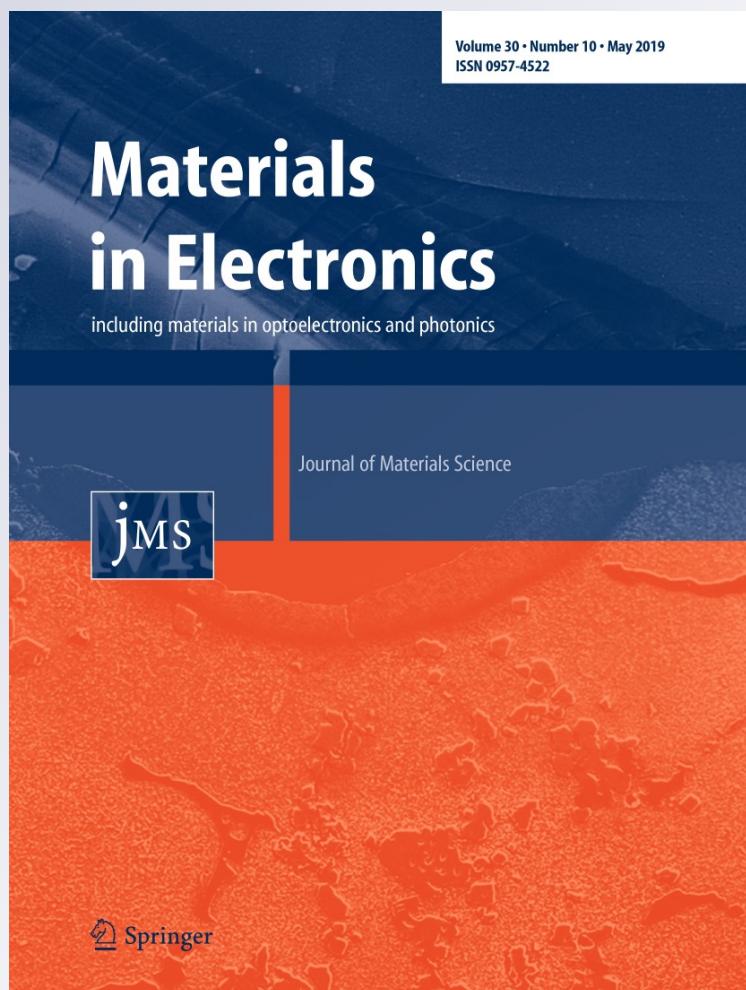
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Characterization of the die-attach process via low-temperature reduction of Cu formate in air

Woo Lim Choi¹ · Young Sung Kim¹ · Ki-Seong Lee¹ · Jong-Hyun Lee¹

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Abstract

For sinter-bonding wide-bandgap power devices to Cu finished metals in air, a low-cost material, i.e., a Cu complex paste containing mechanochemically synthesized Cu(II) formate, was prepared. Characteristics of the die-attach process were analyzed with respect to the bonding conditions. Cu nanoparticles were formed *in situ* by the reduction of Cu(II) formate during heating for the attachment. Sinter-bonding between the nanoparticles and Cu metallization was accelerated by the exothermic heat generated by the Cu complex. As a result, high-speed bonding (1–3 min) was achieved, which prevented severe oxidation of the reduced Cu particles and Cu finish even in air. With the application of an external pressure of 20 MPa, the Cu chips were bonded in only 1 min at 225 °C with a resulting shear strength of 23 MPa. Although the pressure decreased to 13 MPa, bonding occurred within 3 min at 225 and 210 °C, with excellent shear strength exceeding 71 and 39 MPa, respectively.

1 Introduction

Wide-bandgap power semiconductors, including SiC and GaN, are increasingly replacing Si devices in power electronics modules because they exhibit low power loss and high efficiency with high thermal conductivity. To achieve higher module efficiency, the chip junction temperature should be increased to as high as 300 °C, which can be accomplished using bond-line materials with high melting points and thermal conductivities [1–4]. Pb, which is commonly used in high-temperature solder alloys, is toxic and environmentally problematic. On the other hand, Pb-free solder alloys are unsuitable as bonding materials due to remelting and mechanical instability at high temperatures [5, 6]. Transient liquid phase (TLP) sinter-bonding was developed as an alternative to soldering; however, the technique involves long bonding times and suffers from intermetallic-related reliability issues for automotive applications [7–9]. Therefore, owing to the drawbacks of soldering and TLP sinter-bonding, another method to achieve more robust bond lines must be developed using Pb-free materials. The solid-state sinter-bonding technique based on silver, which

exhibits a high melting point and ductile mechanical properties, is promising in this respect [10–12]. However, the use of Ag powder-based sinter-bonding paste as a bonding material is not practical because of the high cost of Ag. In addition, for reducing the long bonding time, the paste must be composed of extremely fine Ag particles, leading to further increased costs [13, 14]. Hence, Cu-based pastes are considered to be the most effective sinter-bonding materials [15, 16]. However, Cu is easily oxidized in air—a tendency which is accelerated with increasing temperature [17]. Hence, the formation of active, pure Cu by *in situ* reduction of Cu complexes in air during semiconductor die bonding and suppression of Cu oxidation by fast sintering are rational strategies to form bond lines comprising Cu. The as-formed pure Cu particles promptly sinter and bond with neighboring Cu particles and metallizations due to their nanoscale features, which can suppress time-dependent oxidation.

A pioneering study on the low-temperature fabrication of pure Cu films by thermal decomposition under a nitrogen atmosphere using a copper–amine–complex paste was reported by Yabuki et al. [18, 19]. Because the oxidation of reduced Cu species was effectively suppressed for 30–60 min via sintering under an inert atmosphere, excellent electrical resistivities ranging from 5.0×10^{-6} to $2.0 \times 10^{-5} \Omega \text{ cm}$ were obtained. Lee et al. reported the fabrication of pure Cu films using a single paste formulation containing Cu complexes [20–22]. They observed a low resistivity value of $8.2 \times 10^{-6} \Omega \text{ cm}$ and good adhesion properties on polyimide

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after heating at 250 °C for 30 min under a nitrogen atmosphere. Although the authors only formed Cu films instead of chip bonding, the results indicate the potential use of pastes containing Cu complexes as effective sinter-bonding materials for high-temperature operations. However, the films adopt an inevitably porous microstructure by gas generation during in situ reduction, which is problematic in bond-line structures. Moreover, the long sintering time and introduction of an inert atmosphere severely affect the efficiency of processes using Cu complex pastes. The most effective method to overcome these issues is to employ a moderate external pressure during the die attachment. The external pressure is expected to increase the bond-line density, reduce bonding time, and eliminate the requirement for an inert atmosphere. Therefore, verification of the effectiveness of external pressure for outgassing during the in situ reduction of a Cu complex paste is required.

Furthermore, paste-based die-attach technology demands silver-finish metallization between the paste and direct bonded copper (DBC) substrate to prevent the oxidation of the copper metallization and to obtain a robust bond. However, the formation of an additional layer will increase costs because of the use of expensive silver and extra processing steps. Consequently, a die-attach process using the simple DBC substrate with a copper finish is more ideal. A previous study using a copper-finished DBC showed that long bonding in air induced the oxidation of the copper finish, leading to decreased bonding strength [23]. However, studies regarding die attachment using Cu-based fillers on copper finishes are very rare. A promising method for reducing the oxidation of copper finishes during bonding in air is to complete the bonding rapidly.

Therefore, in this study, die bonding on Cu metallization was performed in air using a Cu complex paste. To characterize die bonding after the decomposition of Cu formate, the microstructure and shear strength were evaluated with respect to the external pressure, bonding temperature, and bonding time.

2 Experimental

2.1 Paste preparation

Cu-complex particles as a filler material were synthesized via the reported mechanochemical method [24, 25]. First, 23 g of Cu₂O powder (95%, Daejung Chemical Co., Ltd.) as a precursor and 103.5 mL of formic acid (HCOOH, 85%, OCI) were added to a zirconia jar (500 mL capacity) along with 86.3 mL of zirconia balls (1 mm diameter). The ingredients were then subjected to intense mechanical milling for 7 h using a planetary mill at 300 rpm. The resultant slurry was separated using a filter and washed five times with ethanol. Finally, the

filtered Cu-complex sludge was dried in a low-vacuum oven at 50 °C for 2 h. Subsequently, a paste containing the Cu complex was prepared by mixing the complex in α-terpineol (98.5%, Samchun Pure Chemical Co., Ltd.) at a weight ratio of 5.9:4.1 with a spatula.

2.2 Die-attach

The prepared paste was screen-printed on a Cu-metallized alumina substrate over an area of 3 × 3 mm², followed by drying at 150 °C for 2 min to remove the solvent by evaporation. A 3 × 3 × 1 mm³ dummy Cu chip was laid to exact fit on the printed pattern to form a sandwich structure. The Cu chip is beneficial for acquisition of the exact bonding strength without measurement of the lower strength via breaking of a brittle chip during the shear test. For bonding, the sandwich-structured combination was placed on a heating stage of a thermal compression bonder set at the specific bonding temperature of 210 or 225 °C and held for 10 s to heat the combination to the set temperature. The combination was subsequently compressed in air by applying an external pressure of 13 or 20 MPa with a ramp, and the bonding time was measured.

2.3 Characterization

The bonding strength of the formed bond line was defined as the maximum stress measured during shearing at 200 μm/s using a shear tester (DAGE-series-4000, Nordson Corp.). Each sample was measured 5 times, and the average strength values were noted.

The morphology and size of the synthesized Cu-complex particles, as well as the microstructures and fracture surfaces of the bond lines, were examined by high-resolution scanning electron microscopy (HR-SEM, SU8010, Hitachi High-Technologies Corp.). Furthermore, to estimate the thermal behavior of the Cu complex during heating, thermal analysis of the particles was performed from 25 to 300 °C at a heating rate of 10 °C/min by thermogravimetry—differential scanning calorimetry (TG-DSC, SDT Q600, TA Instruments).

Phase transformation in the bond line after bonding was verified by X-ray diffraction (XRD, DE/D8 Advance, Bruker). For the XRD analysis, the bond lines were exposed by removing the dummy Cu chip after printing (over an area of 10 × 10 mm²) and bonding.

3 Results and discussion

3.1 Characteristics of the prepared Cu-complex particles

Figure 1 shows the SEM image of the Cu-complex particles obtained after 7 h by the mechanochemical method.

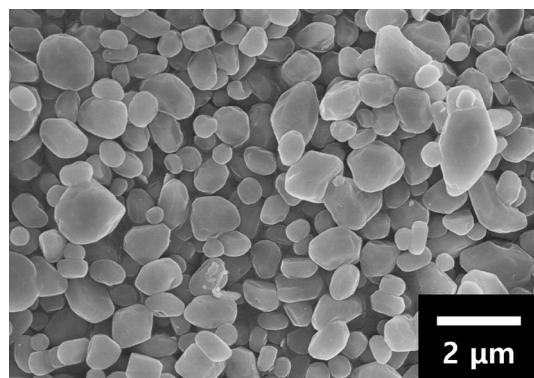


Fig. 1 SEM image of the Cu complex particles obtained using a mechanochemical method after 7 h

The particles are sky-blue with an average particle size of approximately 700 nm (standard deviation of 276 nm). However, the particle shape and size are irregular. As previously reported [25, 26], small amounts of Cu and Cu(COOH)₂ phases were detected in the XRD pattern, but the initial Cu₂O phase was absent. The presence of the pure Cu phase in the sample can be attributed to remnant Cu resulting from the incomplete oxidation of Cu (Eq. 2) originally formed by the reduction of Cu₂O (Eq. 1). The hydrogen generated during Cu oxidation (Eq. 2) fills the finite interior of the sealed jar. Consequently, the pressure inside the jar increases, thereby hindering the oxidation of Cu (Eq. 2) [16].

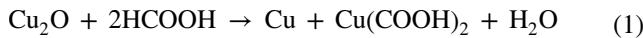
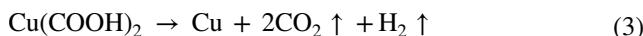


Figure 2 shows the TG-DSC spectra of the synthesized Cu-complex powder. Pyrolysis of Cu(II) formate in the powder began at approximately 200 °C (Eq. 3) [16, 27]:



This is an essential reaction for the formation of a pure Cu film from the Cu complex. The formation of CO₂ and H₂ abruptly reduced the total weight of the specimen by up to 41 wt% during the final heating to 240 °C for 4 min. The weight reduction occurred on the Cu component of the Cu-complex powder. The initial Cu content was 30 wt %, which was higher than that reported in other studies, where an initial Cu content of 25 wt% (and corresponding weight reduction ~44 wt%) was more commonly used [16]. After the rapid weight decrease, the weight of the specimen slightly increased due to oxidation of the as-formed Cu particles, which occurred due to the increased temperature. In addition, exothermic peaks corresponding to three distinct steps were observed in the DSC spectrum during the weight-loss test. The number of steps was different from those reported

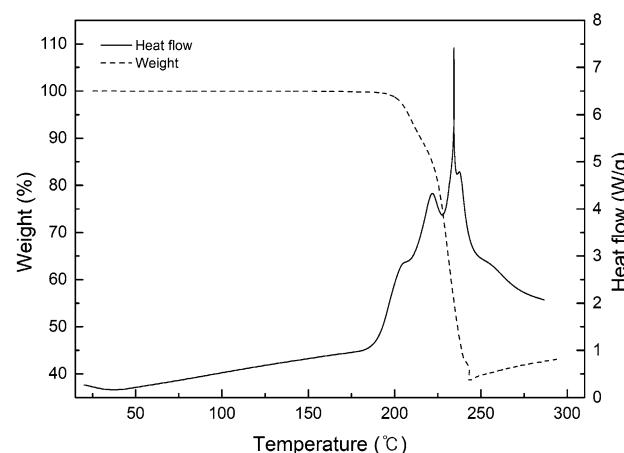


Fig. 2 TG-DSC data of the as-synthesized Cu-complex powder

in other studies, wherein the samples were prepared using a similar mechanochemical method [16]. However, the final sharp peak was similar to that observed for the samples prepared by the precipitation method [16].

3.2 Bonding properties

The backscattered-electron image (BEI) image of a representative bond line formed using the Cu-complex paste is shown in Fig. 3. The bond line has formed by heating at 225 °C for 3 min under a pressure of 20 MPa. Because of volume shrinkage due to the conversion from Cu(II) formate to pure Cu and density increase due to the elimination of initial voids, the thickness of the bond line decreases to ~7 μm after chip bonding. The nanoparticles formed from the Cu formate grow by coarsening and, subsequently, pronounced sintering of the grown particles is observed. Moreover, sinter-bonding between the as-grown particles and dummy Cu chip or Cu metallization of the DBC substrate is clearly observed, indicating a robust bonded state without any striking weak points.

Figure 4 shows the XRD patterns of the middle region of the bond lines in the samples bonded at 225 °C for different bonding times under an external pressure of 20 MPa. Peaks indexed to the Cu(II) formate phase are observed even after heating for 1 min, indicating that the Cu formate does not completely decompose (Eq. (3)) after 1 min in our system. However, after heat treatment for 3 min, the formate peak completely disappears and the pure Cu phase as well as a slight amount of CuO are detected, apart from the Al₂O₃ substrate peaks. Hence, it is expected that the formation of the pure Cu phase via pyrolysis of Cu(II) formate is possible even in air and the oxidation of the Cu particles in air begins immediately after pure Cu formation. The formation of pure Cu even in air may be attributed to the in situ formation of a reducing atmosphere envelope that shields the substrate

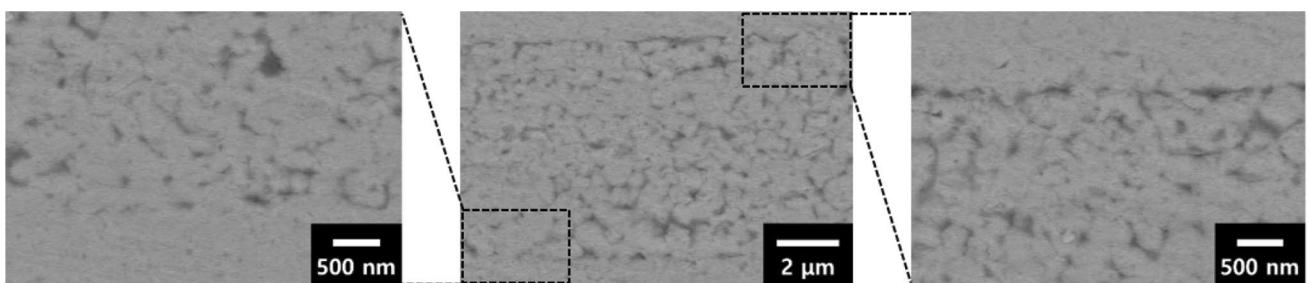


Fig. 3 BEI showing a representative bond line formed using the Cu-complex paste

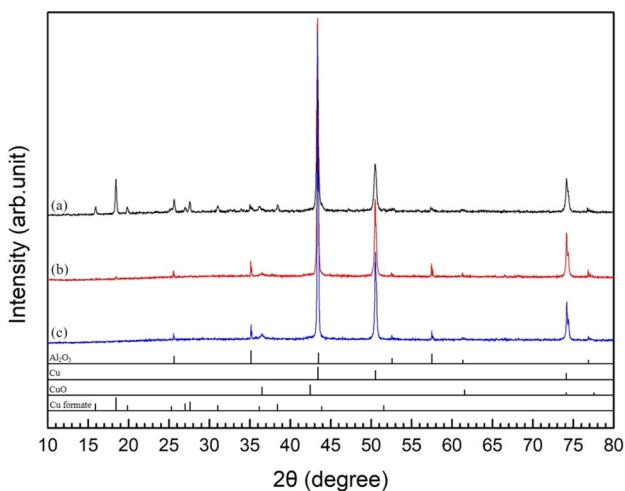
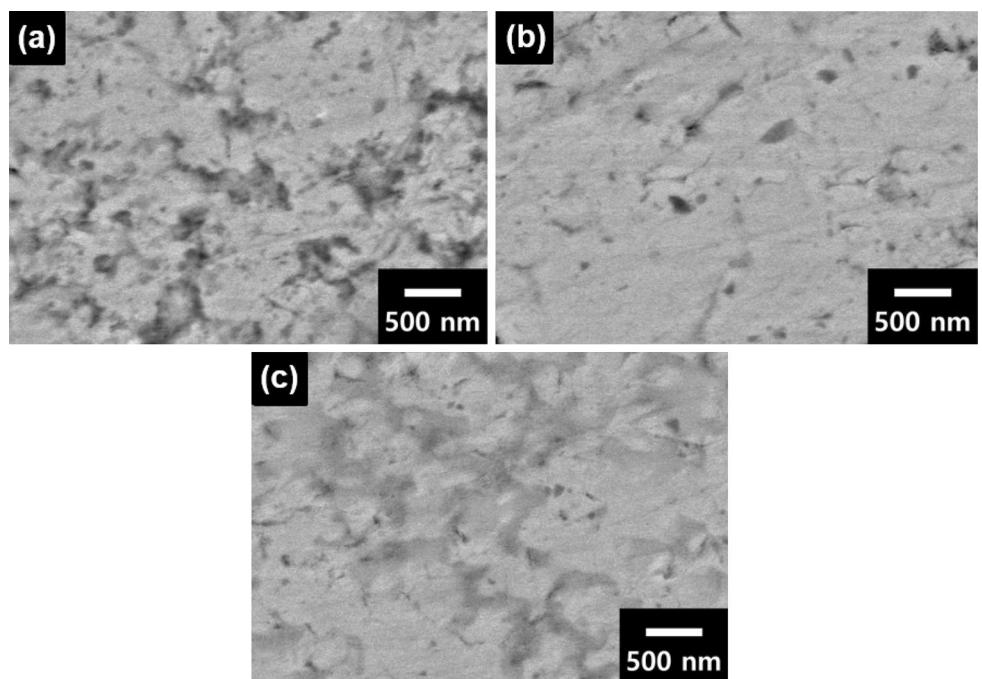


Fig. 4 XRD patterns of sample bond lines obtained by heating at 225 °C under 20 MPa for: (a) 1 min, (b) 3 min, and (c) 5 min

Fig. 5 BEIs of bond lines formed by heating at 225 °C at 20 MPa for bonding times of: **a** 1 min, **b** 3 min, and **c** 5 min



from the oxygen in air, owing to the generation of hydrogen with carbon dioxide (Eq. (3)). In addition, the CuO peak intensity increases with increasing heating time up to 5 min. Consequently, it was estimated from the XRD results that the time required to acquire pure Cu from Cu(II) formate at 225 °C is approximately 3 min in air and that the oxidation of the formed Cu particles proceeds after 5 min because the oxidation degree increases proportionally with increasing heating time in air.

Figure 5 shows the BEIs of the prepared bond lines corresponding to different bonding times for bonding at 225 °C under 20 MPa of external pressure. The bond line formed after processing for 3 min (Fig. 5a) contains many bulk-like microstructures and fewer voids compared with the bond line formed after 1 min (Fig. 5b). The carbon dioxide and hydrogen produced from the pyrolysis of Cu(II) formate present in the sample obtained after heating for 1 min (Fig. 4)

would have hindered the sintering of the particles. This is likely the reason for the presence of the large number of voids. In addition, because of the increased oxygen content resulting from the enhanced oxidation, the surfaces of several sintered particles in the bond line formed after heating for 5 min are dark grey (Fig. 5c).

The bond lines formed at a lower pressure of 13 MPa show surface morphologies similar to those formed at 20 MPa (Fig. 6). However, larger voids between the sintered particles are observed in the bond lines formed at 13 MPa with bonding times of 1 and 3 min.

The BEIs of the bond lines formed at the lower bonding temperature of 210 °C and at different pressures and bonding times are shown in Fig. 7. The images of the bond lines formed after 3 min do not indicate any bulk-like microstructures; instead, the contours of tiny particles are observed, irrespective of the applied pressure. Thus, the microstructural analysis results indicate that a small amount of Cu-complex particles is present even after heating for 3 min. The additional bonding for 2 min, under both 13 and 20 MPa conditions, leads to microstructural evolution in the samples, wherein the grown particles sinter together. However, voids are clearly observed between these particles. These results indicate that the bonding temperature can greatly influence the microstructure of a bond line and can determine the mechanical properties of the joint.

Figure 8 shows the shear strength of the Cu dummy chip bonding as a function of bonding temperature and time under different bonding pressures. The bonding formed after 1 min under 20 MPa (Fig. 8a) at 225 °C exhibits a sufficient bonding strength of 23 MPa. However, the strength could

not be optimized because of the presence of the Cu-complex particles in the bond line. With increasing bonding time to 3 min, the shear strength increases to \sim 70 MPa, which could be attributed to the complete elimination of Cu-complex particles via complete pyrolysis. However, the shear strength notably decreases to 63 MPa with further increased bonding time (additional 2 min), although the value remains considerably high. When the bonding occurs at 210 °C, chip bonding is not accomplished within 1 min. However, considerably high shear strengths of 40 and 53 MPa are measured for the longer bonding times of 3 and 5 min, respectively.

The bonding formed under a lower external pressure of 13 MPa at 225 °C and bonding time of 1 min exhibits a low shear strength of 14 MPa (Fig. 8b). However, with increasing bonding time to 3 min, the shear strength drastically increases to \sim 70 MPa. When the bonding time is further increased by 2 min, the shear strength again decreases to 56 MPa. At 210 °C, the chip cannot bond within 1 min, and with increasing bonding time to 3 and 5 min, the shear strength increases to 39 and 51 MPa, respectively.

With increased bonding time, rapidly increasing shear strength is observed in this study, unlike that observed in other studies. This could be attributed to the formation of nanoscale Cu from Cu(II) formate. The considerable sinterability of nanoscale metal particles has been reported in similar studies [28–33]. Although the Cu nanoparticles were rapidly oxidized in air, hydrogen and carbon dioxide generated during pyrolysis might have hindered the oxidation of the initially formed Cu nanoparticles. In addition, the heat generated from the Cu complex during heating could also contribute to the fast sinter-bonding.

Fig. 6 BEIs of bond lines formed by heating at 225 °C at 13 MPa for bonding times of: **a** 1, **b** 3, and **c** 5 min

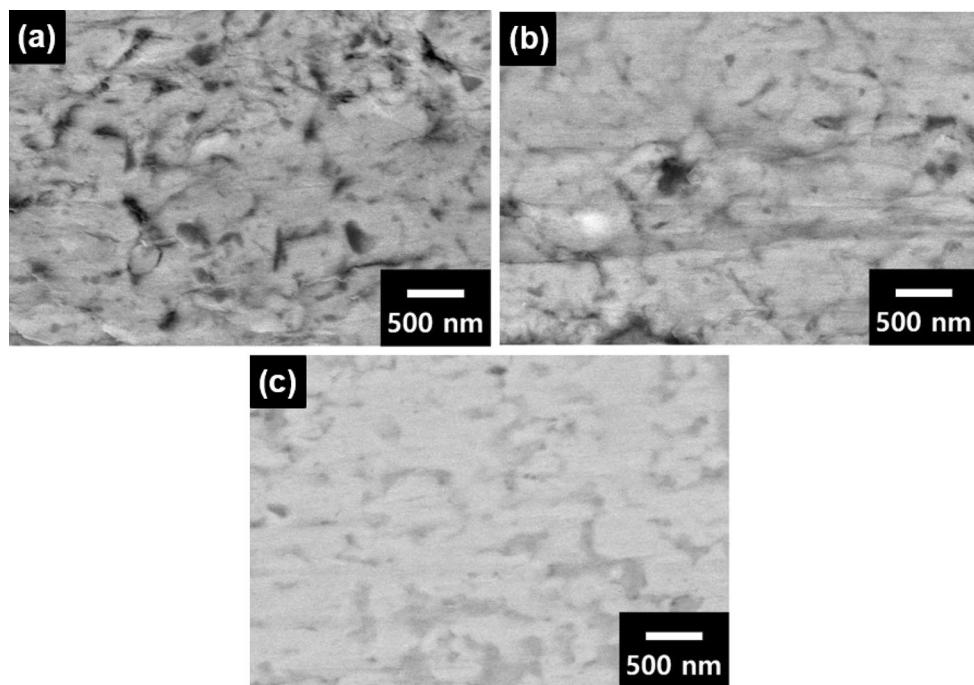


Fig. 7 BEIs of bond lines formed by heating at 210 °C under the following pressures and bonding times: **a** 20 MPa and 3 min; **b** 20 MPa and 5 min; **c** 13 MPa and 3 min; and **d** 13 MPa and 5 min

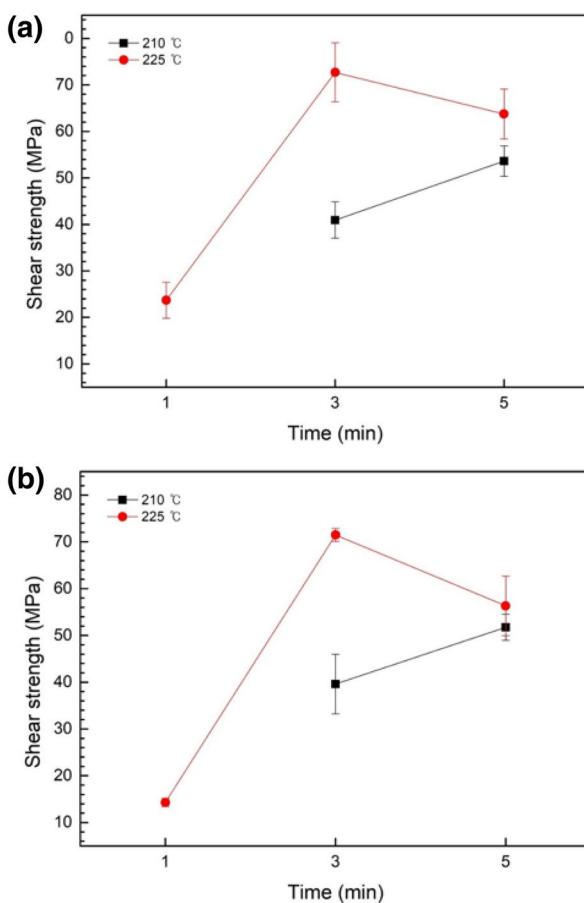
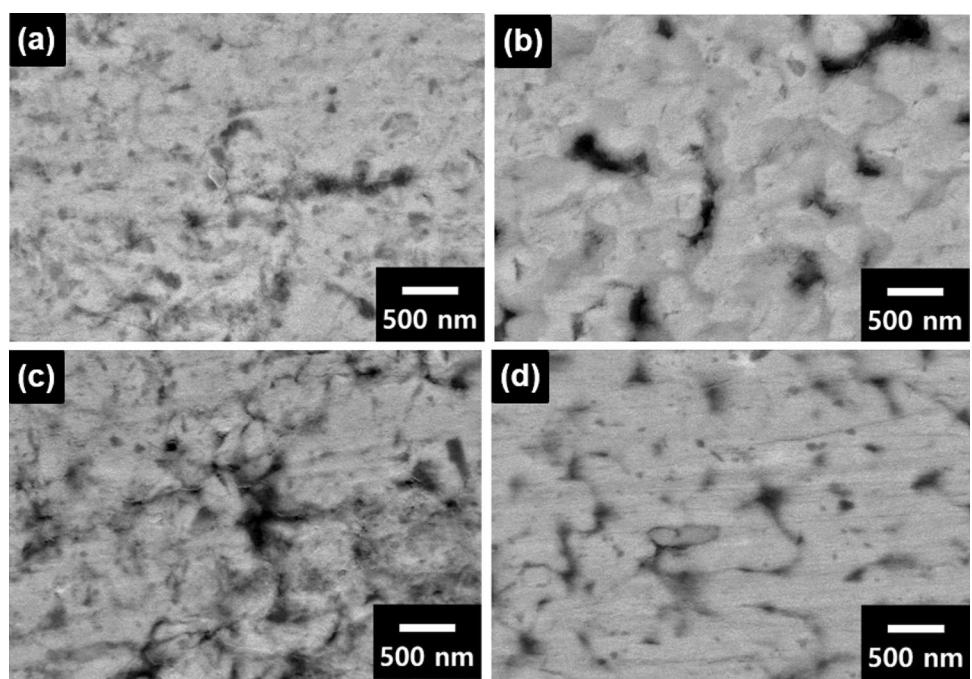


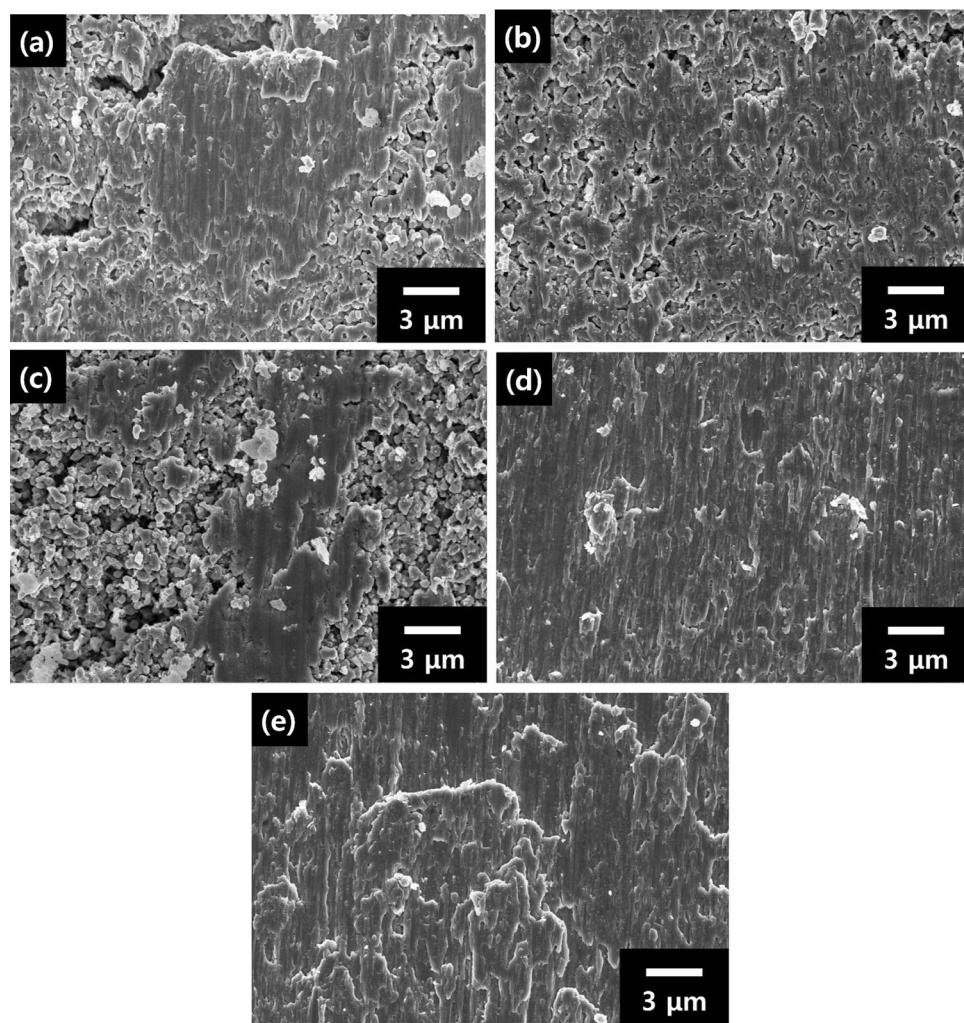
Fig. 8 Shear strengths of dummy Cu chips bonded by heating at 210 and 225 °C for different times under pressures of: **a** 20 MPa and **b** 13 MPa

3.3 Fracture properties

Figure 9 shows the fracture surfaces on the DBC substrates after shear testing of the chips bonded under a pressure of 13 MPa at different bonding temperatures and bonding times. All fracture surfaces are formed on the inside of the bond lines, instead of at the interfaces (i.e. the bottom surface of the chip/bond-line or bond-line/top surface of the substrate). Die bonding at 210 °C for 3 min (Fig. 9a) results in a non-uniform microstructure due to the slight sintering of particles on the fracture surface. However, patches of bulk-shaped structures, on which shear bands are formed, are also observed; this is indicative of incomplete sintering. The fine particles are possibly those of the Cu complexes containing the Cu(II) formate phase. With increased bonding time to 5 min (Fig. 9b), a more uniform microstructure is developed due to enhanced sintering. In addition, ductile sheared patterns containing shear bands are formed on the sintered structure after fracturing. The enhanced sintering degree and microstructural homogeneity are directly responsible for the observed increase in shear strength.

For the sample obtained after die bonding for 1 min at 225 °C, the contours of the Cu complex particles and voids between the particles, as well as locally formed bulk structure on the fracture surface are clearly observed (Fig. 9c), indicating an inadequate sintered state. Shear bands are also observed, but only in the local bulk structure. The low shear strength measured for the samples could be attributed to this fractured surface. When bonding was performed for 3 min (Fig. 9d), almost complete sintering of particles occurred, resulting in the disappearance of voids due to the elimination

Fig. 9 SEM images of the fracture surfaces on DBC substrates obtained after shear testing of the Cu chips bonded at different bonding temperatures and times under a pressure of 13 MPa: **a** 210 °C and 3 min; **b** 210 °C and 5 min; **c** 225 °C and 1 min; **d** 225 °C and 3 min; and **e** 225 °C and 5 min



of Cu complex particles. Consequently, highly developed shear bands were formed over the entire fracture surface, resulting in very high strength. When the bonding time was increased to 5 min (Fig. 9e), a similar fracture surface was observed, but with layers. The formation of the layers reflects the brittle nature of the bonding, resulting from the oxidation of the bond-line material. In addition, chips bonded under a pressure of 20 MPa showed similar fracture surface characteristics.

4 Conclusions

High-speed and robust die attachment on Cu-finish metallization was achieved in air using a paste containing Cu(II) formate, due to the formation of Cu nanoparticles by reduction and rapid sintering of the nanoparticles. The fast sinter-bonding was attributed to the heat generated by the mechanochemically synthesized Cu-complex powder, as revealed by the exothermic peaks in the TGA–DSC

spectra during heating (for attachment). With a bonding time of 3 min at 225 °C under an external pressure of 13 or 20 MPa, the shear strength significantly increased to values exceeding 70 MPa due to the completion of the pyrolysis reaction. However, with further increasing bonding time to 5 min, the strength was reduced due to surface oxidation of the sintered Cu particles. At a slightly lower temperature of 210 °C, the strength was significantly decreased due to slow pyrolysis. A bonding time of 3 min resulted in a shear strength of ~40 MPa due to the presence of remnant Cu-complex particles. At the same temperature, the increased bonding time of 5 min led to an increased shear strength (exceeding 50 MPa) due to the complete removal of the Cu complexes. To achieve sufficient bonding within the short bonding time of 1 min, a bonding temperature of 225 °C was required. Under these conditions, application of a high pressure was effective for enhancing the strength, although the high pressure did not significantly influence the strength with increasing bonding time to 3 min.

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