# Electrical Properties of Conductive Adhesives as Affected by Particle Compositions, Particle Shapes, and Oxidizing Temperatures of Copper Powders in a Polymer Matrix

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**ABSTRACT:** The effects of particle compositions, particle shapes, and oxidation temperatures on the electrical properties of conductive adhesives have been investigated. Silver-coated copper powders and uncoated copper powders with spherical and flake-shaped particles are oxidized at temperatures such as 30, 175, 240, 300°C and 350°C for 2 h and dispersed in an epoxy matrix. The results of this study indicate that the electrical properties of the conductive adhesives are strongly affected by the particle compositions and oxidation temperatures and only slightly affected by the particle shapes. Silver-coated copper powders show significantly greater oxidation resistance than uncoated copper powders. To understand how silver-coated copper powders show such oxidation resistance, they are analyzed by the techniques of thermogravimetric analysis (TGA), X-ray diffraction (XRD), and Auger spectroscopy to observe how metal oxides such as AgO, $Cu_2O$, and CuO affect the electrical properties of the conductive adhesives. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 2045–2053, 2004

**Key words:** adhesives; ESCA; fillers; oxidation; copper powder

## INTRODUCTION

As demand for smaller, lighter, thinner components and higher performance in electrical and electronic appliances continues to grow, especially in mobile devices, more effort goes into downsizing semiconductor devices. This increases the input/output (I/O) pin count of the devices and decreases the I/O pin pitch. For those devices, printed wiring boards (PWBs) with higher wiring density are essential. To enhance the packaging efficiency of PWBs, the electrical routes within a PWB are filled with a conductive adhesive to provide electrical interconnection between metal layers.[1–3] A number of studies have examined the electrical properties of metal-filled polymers and the mechanisms involved in the formation of conductive composites of polymer/metal systems.[4–9] Various conductive powders such as Al, Ag, Pd, Cu, carbon black, carbon fiber, and various graphitic powders are applied to polymers to create electrically conductive adhesives.[10–25] Silver powder with flake and spherical-shaped particles is the most widely used metallic filler for conductive adhesives.[26] Silver has the disadvantage of being relatively expensive. Aluminum powder is widely used in the adhesives industry as reinforcing filler and as a decorative, but aluminum powder can not be used to make electrically conductive plastics because of the oxide film that insulates the particle contact points. Gold powder is sometimes used for special electronic assembly operations. Gold-filled adhesives are much more expensive and show a much lower electrical conductivity than silver-filled adhesives. Palladium is limited in quantity; no powders of pure palladium are manufactured. Some copper powders are mixed into epoxy to manufacture low-cost conductive adhesives. Metallic fillers generally suffer from the oxidation of the metallic particles and the deterioration of the electrical properties of the composite because of the nonconductive nature of such oxide layers.[24] Unlike the oxidation of aluminum, copper is readily oxidized at a low temperature and has no self-protective layer to prevent further oxidation.[27–29] Therefore, the study of copper oxidation and the development of a means to prevent copper oxidation are crucial to the application of electrically conductive copper fillers. Silver coating has been used to enhance the oxidization resistance of copper fillers to air.[30–33] In this study, the silver coating to enhance the oxidization resistance of copper fillers to air and improve the electrical properties of conductive adhesives is investigated. Silver-coated and uncoated copper fillers are oxidized at 30, 175, 240, 300, and 350°C for 2 h and are studied to see how they affect the electrical properties of conductive adhesives.

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The electrical properties of the composites are dependent on the concentration, size, and shape distribution of the conductive particles.[34–39] In this study, the electrical properties of conductive adhesives are studied to see if there is any relationship with the particle shape of silver-coated copper powders either flake or spherical. In this study, thermogravimetric analysis (TGA), X-ray diffraction (XRD), and Auger spectroscopy are used to study the particle compositions and indicate how the specified metal oxides on the surfaces of copper powders affect the electrical properties of conductive adhesives.

## EXPERIMENTAL

### Materials and sample preparation

In this study, copper powders (20.4% in volume and 70% in weight) are mixed into epoxy in readiness for preparing copper conductive adhesives, which are prepared by mixing epoxy resin/novolac, curing agent anhydride, accelerator, and copper powder and blending through EXAKT "Triple Roller Mills" (E. J. Payne Ltd., Longton, UK). Three copper powders such as uncoated copper powder 800 and silver-coated copper powders 106 and 107 are used to investigate the electrical properties of conductive adhesives. In this study, the electrical properties of cured conductive adhesives are measured by the volume resistivity of the adhesives. The lower the volume resistivities of cured conductive adhesives are the better the electrical properties of cured conductive adhesives. The conductive adhesives are casted on a glass slide to make a film 1.5 cm long, 0.8 cm wide, and 0.012 cm thick and cured at 175°C for 2 h. A four-points probe is applied to the film of cured conductive adhesives to determine their resistance by applying a current. The volume resistivity of a cured conductive adhesive is calculated by

$$\text{volume resistivity } (\Omega\text{-cm}) = [\text{resistance } (\Omega) \times \text{area } (\text{cm}^2)]/[\text{length (cm)}]$$

### Scanning electron microscope observation

A Hitachi model *S*-2700 scanning electron microscope (SEM) (Hitachinaka, Japan) apparatus was used to observe the morphology of the copper powders in the conductive adhesives. A 15-keV accelerator was used to scan the sample surfaces.

### TGA analysis

To evaluate the oxidation of Cu powders, the weight gained by Cu powders was increased by the use of rising temperatures measured by TGA (Perkin Elmer TGA7; Perkin Elmer, Wellesley, MA). The temperature range for Cu powders was set from 30–480°C and heated at a rate of 20°C/min, and air was used for purging at 30 mL/min.

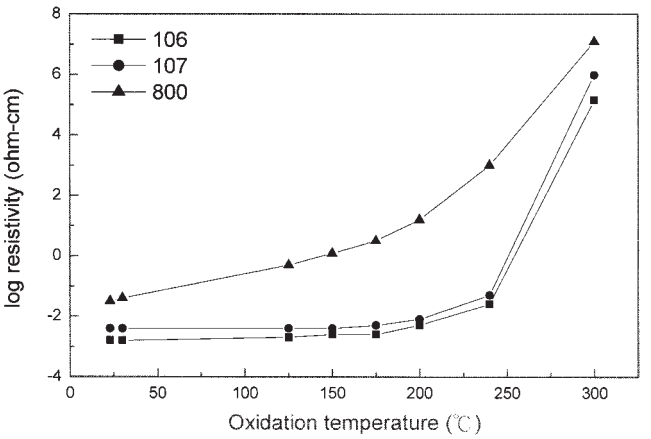



**Figure 1** The volume resistivity of cured copper conductive adhesives filled with silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 that were oxidized at various oxidation temperatures for copper powders for 2 h.

### Auger spectroscopy analysis

Auger spectroscopy exhibits a high resolution for carbon, oxygen, silver, and copper. Auger data were collected by surveying on the surface and using an in-depth X-ray high-resolution mode examination of the samples by using a VG Scientific 310 D (VG Scientific, Beverly, MA), a system equipped with an Mg anode (X-ray source) with an operational voltage of 3 kV and an operational current of 8 mA. Data were recorded at 45° collecting angles resulting in an analysis sputtering rate of 1Å/1 sec.

### XRD analysis

The XRD data of the disc-shaped specimen were collected in a Sintage X-4000 (California). The X-ray generator with $CuK_{\alpha}$ radiation was operated at 35 kV and 30 mA. The diffractometer scanning speed was 4°Å/min. The Bragg angle 2 $\theta$ was operated from 20 to 80°.

## RESULTS AND DISCUSSION

### Electrical properties of conductive adhesives

The volume resistivity of cured conductive adhesives filled with silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 is shown in Figure 1. The volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 after oxidization at room temperature (23°C) is the

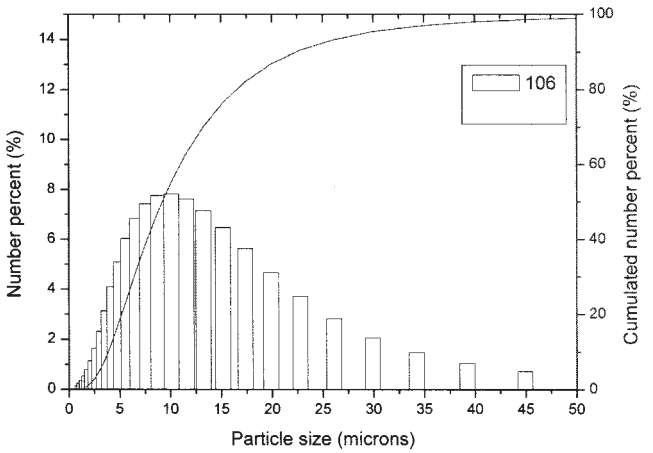



**Figure 2** The particle size distributions of silver-coated copper powder 106.

highest at $1 \times 10^{-1.5}$ Ω-cm (0.0316 Ω-cm), exhibiting the least-desirable electrical properties. The volume resistivity of cured conductive adhesives filled with silver-coated copper powder 106 after oxidization at room temperature (23°C) is $1 \times 10^{-2.8}$ Ω-cm (0.00158 Ω-cm). The volume resistivity of cured conductive adhesives filled with silver-coated copper powder 107 after oxidation at room temperature (23°C) shows that their electrical properties lie between those filled with silver-coated copper powder 106 and those filled with uncoated copper powder 800 with a volume resistivity of $1 \times 10^{-2.4}$ Ω-cm (0.00398 Ω-cm), that is between 0.00158 and 0.0316 Ω-cm. As the temperature of the copper powders is increased from 23 to 175°C, the volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 also exhibits the least-desirable electrical property as the volume resistivity is gradually increased to $1 \times 10^{0.5}$ Ω-cm (3.16 Ω-cm), thus, increasing volume resistivity 10-fold. The volume resistivity of cured conductive adhesives filled with silver-coated copper powders 106 and 107 is slightly increased to $1 \times 10^{-2.6}$ Ω-cm (0.00251 Ω-cm) and $1 \times 10^{-2.3}$ Ω-cm (0.005 Ω-cm), respectively. Hence, the silver coating provides good oxidation resistance for copper powders at temperatures from 23 to 175°C. However, as the oxidation temperature is increased to 240°C, the volume resistivity of cured conductive adhesives filled with silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 are all gradually increased to $1 \times 10^{-1.6}$, $1 \times 10^{-1.3}$, and $1 \times 10^{3}$ Ω-cm, respectively. By further raising the oxidation temperature to 300°C, the volume resistivities of cured conductive adhesives filled with silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 are all rapidly increased to $1 \times 10^{5.15}$, $1 \times 10^{5.98}$, and $1 \times 10^{7.08}$ Ω-cm, respectively.

Figures 2 and 3 show the particle size distributions of silver-coated copper powders such as 106 and 107. A particle size of 10 microns is the most frequent for silver-coated Cu powders 106 and 107. However, cured conductive adhesives filled with silver-coated copper powder 106 show a slightly lower than cured conductive adhesives filled with silver-coated copper powder 107 as shown in Figure 1. Particle shape is a possible factor in the volume resistivity of cured conductive adhesives filled with silver-coated copper powders 106 being slightly lower than that of cured conductive adhesives filled with silver-coated copper powder 107. Figures 4 and 5 show SEM photographs of cured conductive adhesives filled with silver-coated copper powders 106 and 107. Silver-coated copper powder 106 has flake-shaped particles and silver-coated copper powder 107 has spherically shaped particles. Silver-coated copper powder 106 with flake-shaped particles provides a greater contact area for the copper powders in conductive adhesives than silver-coated copper powder 107, with spherically shaped ones resulting in a better electrically conductive path.

Figure 6 shows the particle size distributions of uncoated copper powder 800. Figure 7 shows an SEM photograph of the cured conductive adhesives filled with uncoated copper powder 800. Uncoated copper powder 800 has spherically shaped particles. Compare this with the SEM photograph of the cured conductive adhesives filled with silver-coated copper powder 107 as shown in Figure 5. The contact area of uncoated copper powder 800 in conductive adhesives is lower than the contact area of silver-coated copper powder 107 in conductive adhesives. The lower contact area of the uncoated copper powder 800 in conductive adhesives is because of the smaller particle size of uncoated copper powder 800 than the silver-coated copper powder 107. Five microns is the most-frequent size for uncoated copper

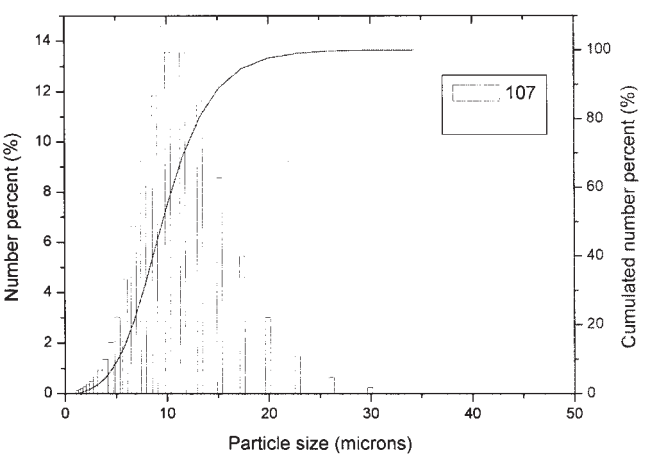



**Figure 3** The particle size distributions of silver-coated copper powder 107.

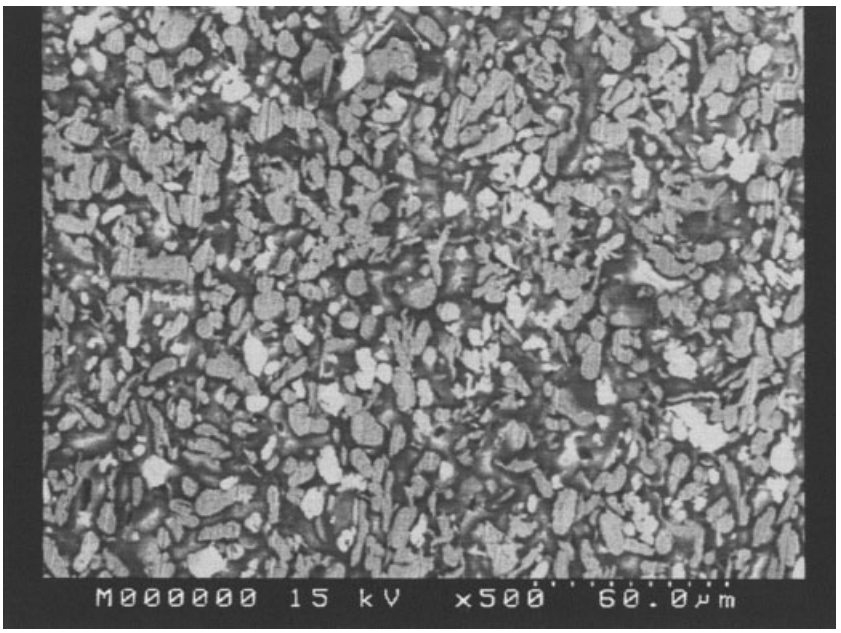



**Figure 4** An SEM photograph (×500) of cured copper conductive adhesive filled with 20.4% by volume of silver coated-Cu powder 106.

powder 800 (see Figure 6) and is much smaller than the particle size of 10 microns for silver-coated copper powder 107 (see Figure 3). However, the volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 is much higher than that of the adhesives filled with silver-coated copper powder 107, as shown in Figure 1. Not only the particle compositions but also their sizes affect the volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 and cured conductive adhesives filled with silver-coated copper powder 107, as shown in Figure 1. Because copper is less conductive than silver and because of the smaller contact area, the result is in a higher volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 compared

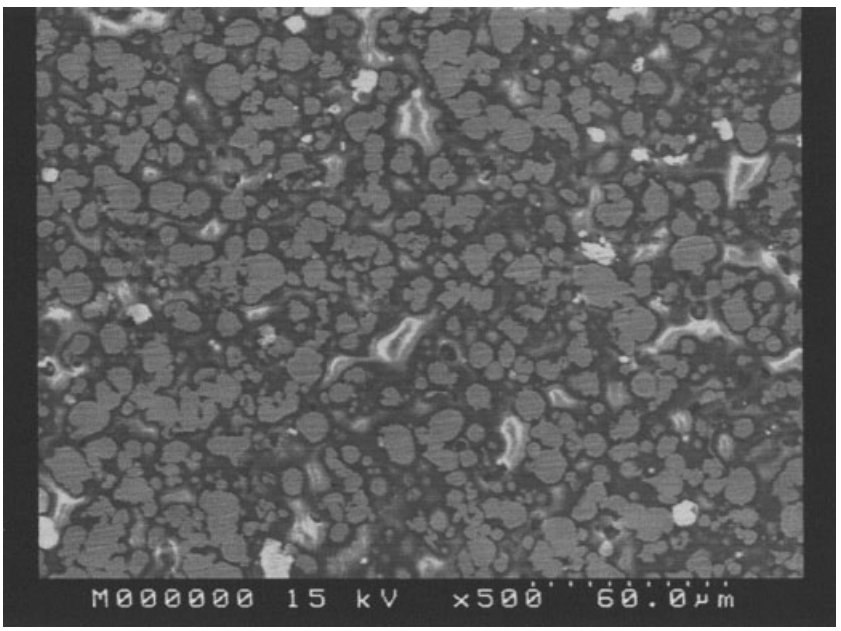



**Figure 5** An SEM photograph (×500) of cured copper conductive adhesive filled with 20.4% by volume of silver-coated Cu powder 107.

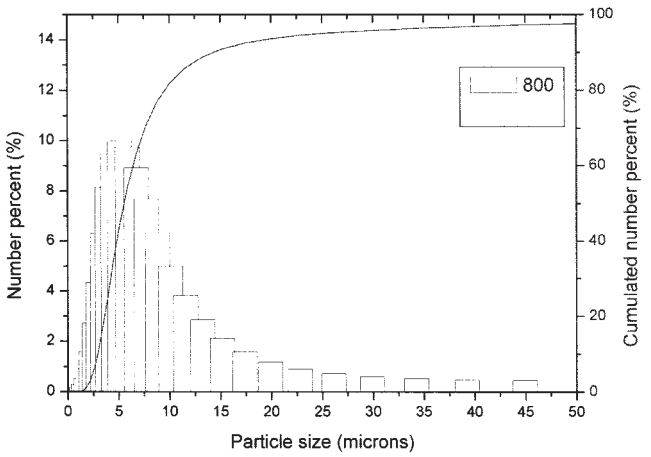



**Figure 6** The particle distributions of uncoated copper powder 800.

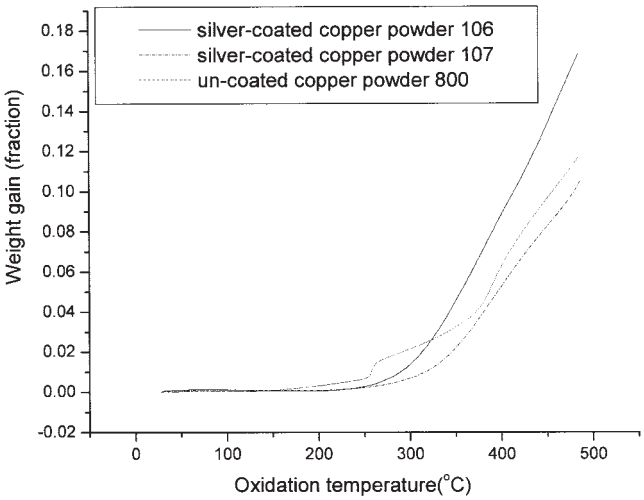



**Figure 8** The TGA curves of silver-coated copper powders such as 106 and 107 and uncoated copper powder 800.

with the volume resistivity of cured conductive adhesives filled with silver-coated copper powder 107.

### Oxide growth of copper powders

The oxide growth of silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 was investigated using TGA. The TGA curves of silver-coated copper powders 106 and 107 and uncoated copper powder 800 are shown in Figure 8. Figure 8, indicating the uncoated copper powder 800, is oxidized gradually from 30 to 260°C and oxidized rapidly from 260 to 480°C. The gradual oxidation from 30 to 260°C of uncoated copper powder 800 results in the volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 gradually increasing in rising temperatures from 30 to 260°C as shown in Figure 1. The rapid oxidation from 260 to 300°C of uncoated copper powder 800 results in the volume resistivity of cured conductive adhesives filled with uncoated copper powder 800 rapidly increasing in rising temperatures from 260 to 300°C as shown in Figure 1.

Figure 8 indicates silver-coated copper powders such as 106 and 107 are not oxidized from 30 to 175°C, are slightly oxidized from 175 to 240°C, are gradually oxidized from 240 to 340°C, and are rapidly oxidized

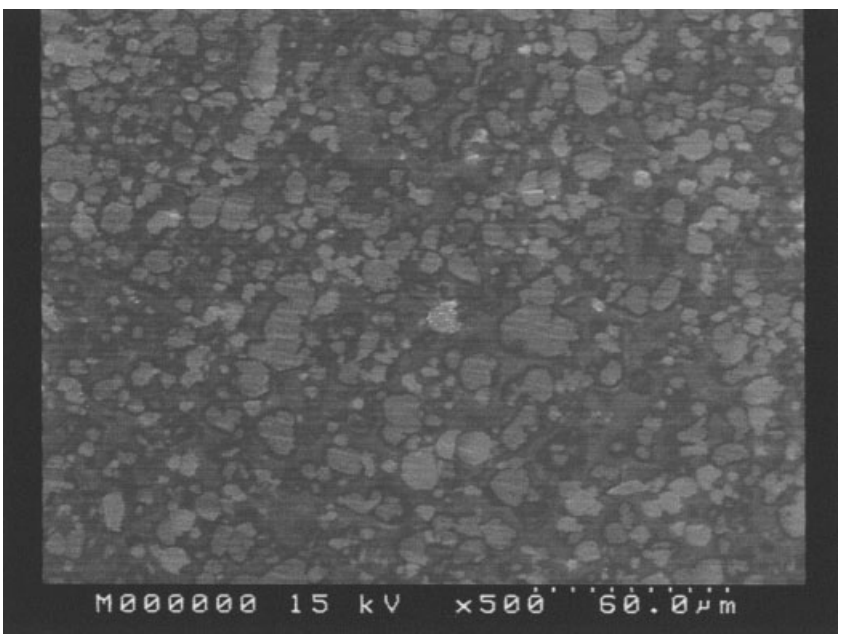



**Figure 7** The SEM photograph (x 500) of cured copper conductive adhesive filled with 20.4% by volume of uncoated copper powders 800.

from 340 to 480°C. The no oxidation from 30 to 175°C of silver-coated copper powders such as 106 and 107 results in the volume resistivity of cured conductive adhesives filled with silver-coated copper powder such as 106 and 107 not increasing in rising temperatures from 30 to 175°C as shown in Figure 1. The slight oxidation from 175 to 240°C of silver-coated copper powders such as 106 and 107 results in the volume resistivity of cured conductive adhesives filled with silver-coated copper powder such as 106 and 107 slightly increasing in rising temperatures from 175 to 240°C as shown in Figure 1. The gradual oxidation from 240 to 300°C of silver-coated copper powders such as 106 and 107 results in the volume resistivity of cured conductive adhesives filled with silver-coated copper powders such as 106 and 107 rapidly increasing in rising temperatures from 240 to 300°C as shown in Figure 1.

Figure 8 shows the oxidation on uncoated copper powder 800 gradually increasing but silver-coated copper powders such as 106 and 107 show negligible oxidation as the temperature rises from 30 to 240°C, indicating the silver coating on the surfaces of copper powders can provide extra oxidation resistance in temperatures from 30 to 240°C.

Figure 8 shows silver-coated copper powder 106 is oxidized more easily than 107 if the temperature rises higher than 240°C. As silver-coated copper powders such as 106 and 107 were oxidized at 300°C for 2 h, the cured conductive adhesives filled with silver-coated copper powder 106 achieved lower volume resistivity than cured conductive adhesives filled with 107, as shown in Figure 1. Silver-coated copper powder 106 with flake-shaped particles (see Figure 4) provides a greater contact area than silver-coated copper powder 107 with spherically shaped particles (see Figure 5), resulting in a lower volume resistivity of cured conductive adhesives filled with silver-coated copper powder 106 (see Figure 1). This indicates an even higher oxidation rate than silver-coated copper powder 107 (see Figure 8).

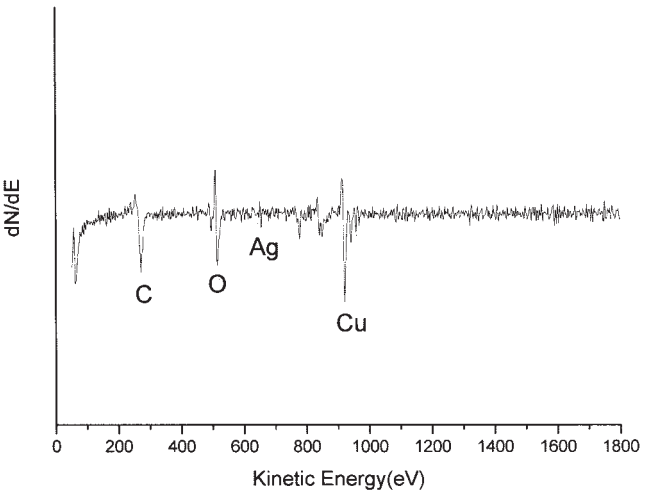



**Figure 10** The Auger spectrum of the surfaces of particles of silver-coated copper powder 106 after oxidation at 240°C for 2 h.

### Metal oxides of copper powders

The metal oxides of silver-coated copper powders such as 106 and 107 and uncoated copper powder 800 were investigated by Auger spectroscopy and XRD. The Auger curves of the surfaces of particles of silver-coated copper powder 106, after oxidation at 175°C and 240°C for 2 h, are shown in Figures 9 and 10. Figure 9 indicates only silver, oxygen, and carbon are

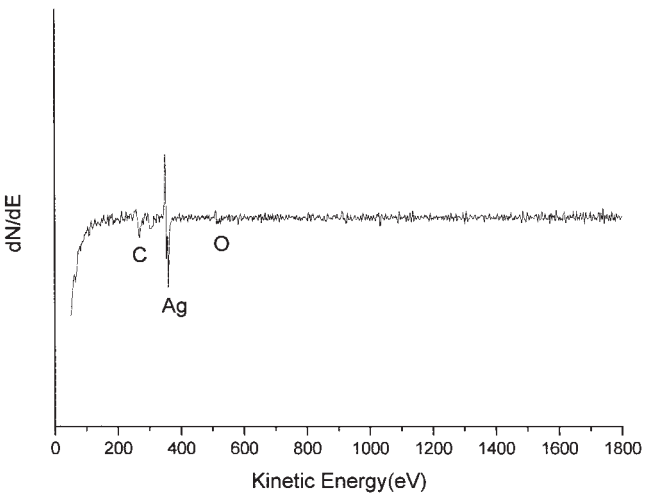



**Figure 9** The Auger spectrum of the surfaces of particles of silver-coated copper powder 106 after oxidation at 175°C for 2 h.

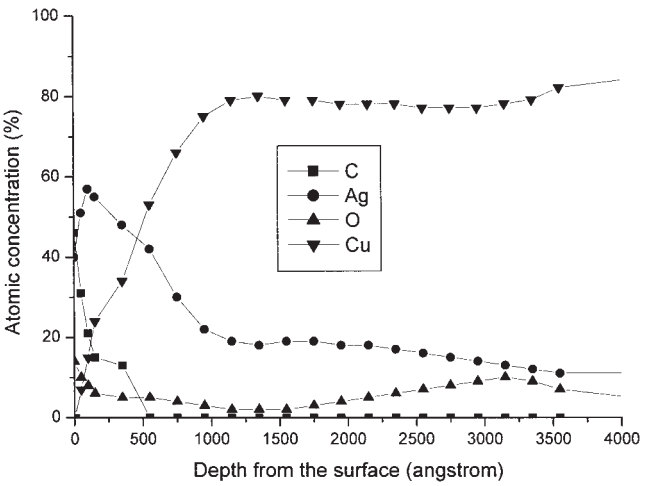



**Figure 11** The atomic concentrations of Cu, O, Ag, and C for silver-coated copper powder 106 after oxidation at 175°C for 2 h plotted as a function of the depths from the surface.

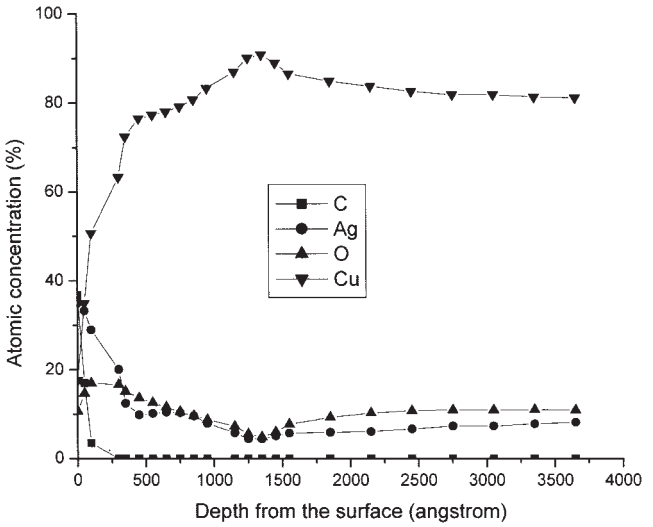
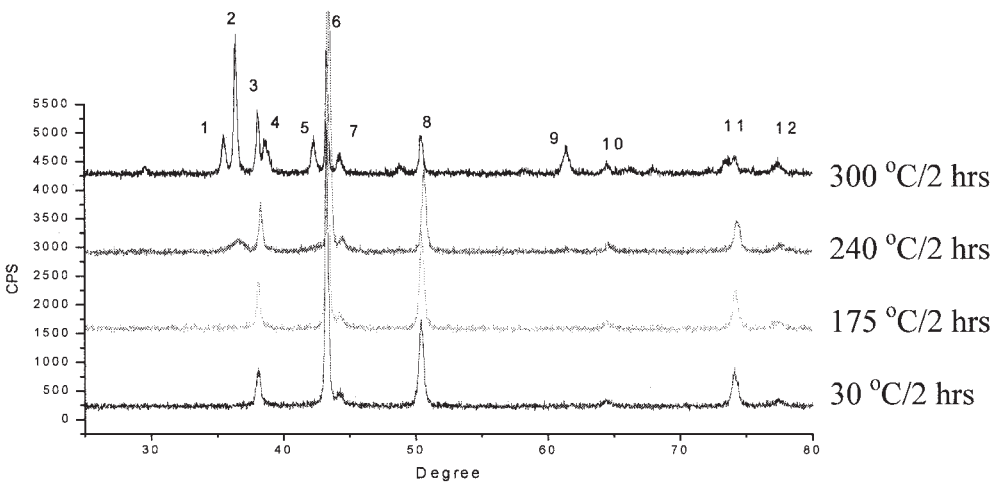



**Figure 12** The atomic concentrations of Cu, O, Ag, and C for silver-coated copper powder 106 after oxidation at 240°C for 2 h plotted as a function of the depths from the surface.

observed in the surface layers of silver-coated copper powder 106 after oxidation at 175°C for 2 h. Figure 10 shows silver, oxygen, carbon, and copper observed in the surface layers of silver coated-Cu powders 106 after oxidation at 240°C for 2 h. For silver-coated copper powder 106, as the temperature increases to 240°C, copper migrates from its interior to its surface layers.

The atomic concentration fractions of Cu, Ag, O, and C for silver-coated copper powder 106, after oxidation at 175 and 240°C for 2 h, are plotted as a function of depth from the surface of its particles as shown in Figures 11 and 12. Figure 11 indicates the atomic concentration of the copper in silver-coated copper powder 106 after oxidation at 175°C for 2 h increases from 0% at the surface to 75% at a depth of 1550 Å and then the atomic concentration of the copper remains constant for depths between 1550 and 4000 Å. For silver-coated copper powder 106, after oxidation at 240°C for 2 h, the atomic concentration of the copper in silver-coated copper powder 106 increases from 17.62% at the surface to 86.51% at a depth of 1550 Å and then the atomic concentration of the copper remains the same from 1550 to 4000 Å. Figure 13 shows XRD curves of silver-coated copper powder 106 after oxidation at various temperatures for 2 h. After oxidation at 240°C, the copper migrates from the interiors of particles of silver-coated copper powder 106 to their surfaces and is oxidized as $Cu_2O$ at 36.49°. In addition, as can be seen in Figure 13, the copper in silver-coated copper powder 106, after oxidation at 300°C for 2 h, migrates from the interior to the surface and is oxidized as $Cu_2O$ and CuO at 36.49 and 35.59° (42.23 and 61.43°). Hence, the volume resistivity of cured conductive adhesive filled with silver-coated copper powder 106 increases as the temperatures rise from 240 to 300°C. This is because of more $Cu_2O$ being




| Number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| θ | 35.59 | 36.49 | 38.17 | 38.75 | 42.23 | 43.37 | 44.32 | 50.5 | 61.43 | 64.51 | 74.17 | 77.5 |
| Element/Oxide | CuO | $Cu_2O$ | Cu | AgO | CuO | Cu | Cu | Cu | CuO | AgO | Cu | Ag |
| Miller Index | (1 1 1) | (1 1 1) | (1 1 1) | (1 0 0) | (1 1 1) | (1 1 1) | (2 0 0) | (2 0 0) | (2 2 0) | (0 2 3) | (2 2 0) | [1 1 1] |

**Figure 13** The elements and oxides for silver-coated copper powder 106 after oxidation at various temperatures for 2 h determined by XRD.

formed and fewer particles being further oxidized to CuO in the surface layers of silver-coated copper powder 106.

The oxides of copper determined by XRD for uncoated copper powder 800 after oxidation at 30, 175, 240, and 300°C for 2 h are shown in Table I. Table I indicates $Cu_2O$ is formed in uncoated copper powder 800 at temperatures higher than 175°C. As the oxidation temperature increases to 300°C, $Cu_2O$ and CuO are formed in the surface layers of the uncoated copper powder 800. The volume resistivity of cured conductive adhesive filled with uncoated copper powder 800 increases in oxidation temperatures from 175 to 300°C because of a greater amount of $Cu_2O$ forming and little of it being further oxidized to CuO on the surfaces of particles of uncoated copper powder 800.

The electrical performance of cured conductive adhesives filled with silver-coated copper powder 106 is slightly affected by the silver for AgO as shown in Figure 13. Cured conductive adhesives filled with silver-coated copper powder 106 indicate good electrical performance; even silver-coated copper powder 106, oxidized at temperatures from 30 to 240°C, indicates a low volume resistivity as shown in Figure 1.

The oxides of copper determined by XRD for silver-coated copper powder 107 are shown in Table II. Only AgO is formed on silver-coated copper powder 107 after oxidation at 30 and 175°C for 2 h. $Cu_2O$ and AgO are formed on silver-coated copper powder 107 after oxidation at 240°C for 2 h. $Cu_2O$, CuO, and AgO are formed on silver-coated copper powder 107 after oxidation at 300°C for 2 h. The formation of $Cu_2O$, CuO, and AgO on silver-coated copper powder 107 after oxidation at 30, 175, 240, and 300°C for 2 h follows the same pattern as the formation of $Cu_2O$, CuO, and AgO on silver-coated copper powder 106. This results in the volume resistivity of cured conductive adhesives filled with silver-coated copper powder 107 following the same pattern as cured conductive adhesives filled with silver-coated copper powder 106.

## CONCLUSION

The results of this study indicate that electrical properties such as the volume resistivity of cured conductive adhesives filled with silver-coated copper powders such as 106 and 107 and uncoated copper powders 800 are strongly affected by particle composition and oxidation temperatures because of the formation of oxides such as $Cu_2O$ and CuO on the surfaces of copper powders. The silver coating provides good oxidation resistance in temperatures lower than 175°C by hindering the copper diffusing from the interior to the surface of silver-coated copper powders 106 and 107. This avoids the formation of $Cu_2O$ and CuO on the surfaces, resulting in good electrical performance because of the low volume resistivity of cured conductive adhesives filled with silver-coated copper powders 106 and 107. The formation of AgO on the surfaces of silver-coated copper powders such as 106 and 107 does not affect the volume resistivity of cured conductive adhesives filled with silver-coated copper powders 106 and 107.

The results of this study indicate that the electrical properties of the conductive adhesives are slightly affected by the particle shape. The silver-coated copper powder 106, with flake-shaped particles, shows better electrical performance than silver-coated copper powder 107, with spherical-shaped particles, because of the lower volume resistivity of cured conductive adhesives filled with silver-coated copper powder 106.

**TABLE I**
**The Formation of CuO and $Cu_2O$ in Uncoated Copper Powder 800 after Oxidation at Various Temperatures for 2 h as Observed by XRD**

| Oxidation temperature | CuO | $Cu_2O$ |
|---|---|---|
| 30°C | X | X |
| 175°C | X | • |
| 240°C | X | • |
| 300°C | • | • |

X, not formed; •, formed.

**TABLE II**
**The Formation of CuO, $Cu_2O$, and AgO for Silver-Coated Copper Powder 107 after Oxidation at Various Temperatures for 2 h as Observed by XRD**

| Oxidation temperature | CuO | $Cu_2O$ | AgO |
|---|---|---|---|
| 30°C | X | X | • |
| 175°C | X | X | • |
| 240°C | X | • | • |
| 300°C | • | • | • |

X, not formed; •, formed.

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