Evaluation of a Printable Catalyst for Use in Flex-Circuit & PCB Applications

by A. Kuruganti, K.S. Chen & E.E. Kalu

In this study, a catalyzation technique for electroless deposition was investigated as an alternate method in flex-circuit and printed circuit board fabrications. The method involved a chemical reduction of metal ion in solution on a surface that is made active by printed catalyst. Two commercial electroless copper deposition baths containing formaldehyde as a reductant and EDTA as a complexant were used to study the influence of several deposition parameters. The morphology of the deposited copper, plating rate and adhesion issues are discussed. The nature of copper deposited was studied by scanning microscopy (SEM), atomic force microscopy (AFM) and X-ray diffraction (XRD) techniques. Results obtained from the evaluation of the two electroless copper baths showed that bath temperature and rate of bath agitation are critical issues that affect the adhesion of deposited copper with the new catalyzation technique.

The electroless plating process discussed here involves the fully additive plating technique whereby the required metal thickness is achieved through electroless plating only. The method eliminates etching of deposited or clad-on metal to produce the final circuit pattern. Like the conventional electroless technique, the initiation of the metal deposition process in this technique requires the use of a catalyst. In the conventional technique, the catalyst is applied on the entire substrate surface.[1–4] Following the catalyzation step, a thin conductive metal film is deposited by the electroless method. Subsequently, the metal deposit is built-up to the required film thickness electrolytically. The required circuit pattern is then printed on the substrate using an etching resist, and the unmasked area is etched away to obtain the circuit pattern of interest. The disadvantages of this approach include long processing time, extensive waste and numerous processing steps.

Unlike the conventional method, the method discussed in this work involves the direct printing of circuit pattern on a dielectric substrate using printable catalyst.[5 - 10] The printed catalyst pattern is then selectively metallized to obtain the circuit pattern. The method allows finer lines and spaces on the substrate, and offers better heat and chemical resistance to the high-temperature plating environment since no adhesive layer is used. Further, the method allows thin metallization where it is required, and there are therefore material costs (copper is deposited only where it is required) and waste disposal advantages since the method does not involve removal of plating resist and etching of unwanted portions of copper deposited on the substrate. It is an attractive method for pattern metallization on a substrate, since there is no underlying conductive path to allow electrolytic plating. Furthermore, no complex equipment is required because the substrates are simply immersed in an aqueous solution containing metal ions and reducing agent. In comparison to chemical vapor deposition (CVD), it is a cheaper metallization technique. From a technological viewpoint, it is a relatively simple technique.

Despite its attractiveness, the method requires printable catalyst ink that must possess good printable characteristics on the substrate and be able to withstand the demanding environment of alkaline/acid bath for the duration of plating. Having demonstrated the feasibility of the method for simple electroless copper baths,[6-8] it is of interest to evaluate the feasibility of the method with

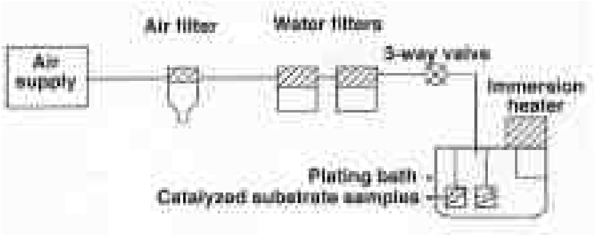

Fig. 1—Schematic diagram of the experimental setup.

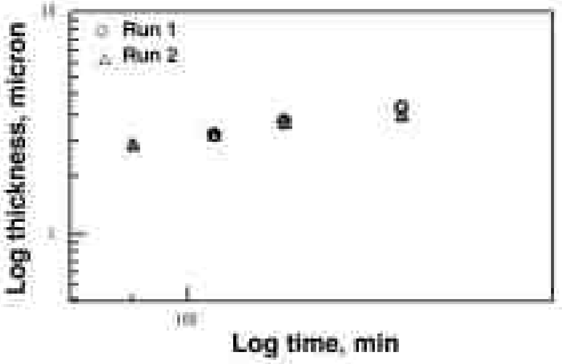

Fig. 2—Plating rate relationship for the high-speed bath (large tank) at 70ºC.

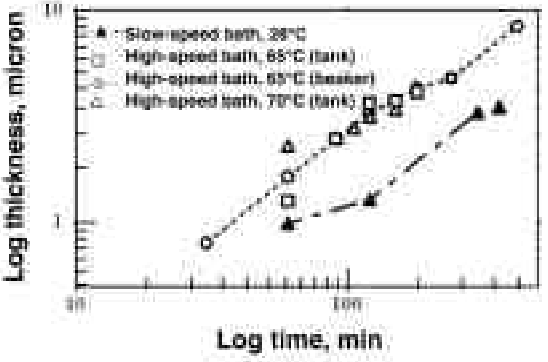

Fig. 3—Comparison of plating rate relationship under different plating conditions.

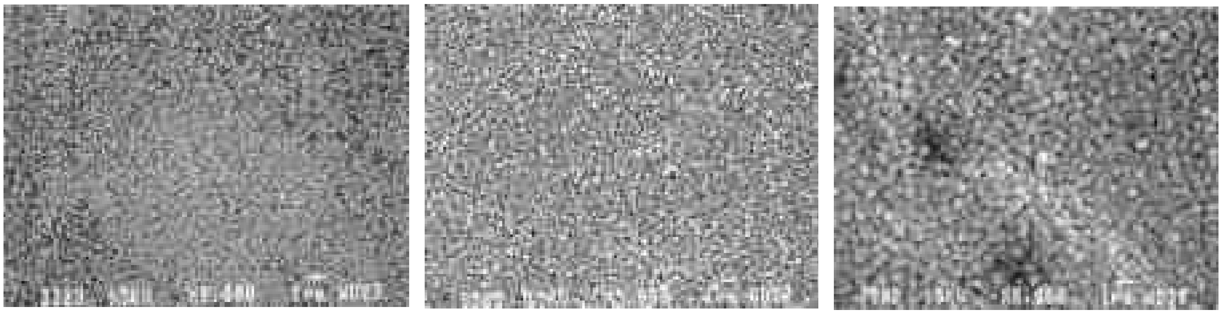

Fig. 4—SEM images of copper deposits at different immersion times for the high-speed bath (large tank) at 65ºC: (a) 30 min; (b) 1 hr; (c) 2 hr.

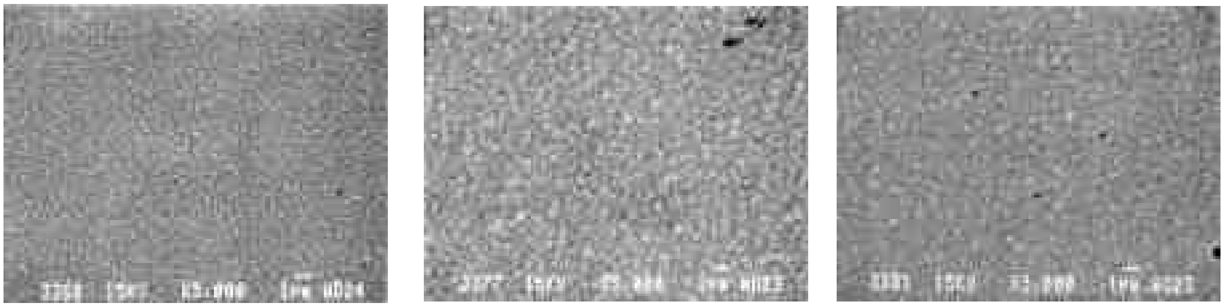

Fig. 5—SEM images of copper deposits at different immersion times for the slow-speed bath (large tank) at 28ºC: (a) 30 min; (b) 1 hr; (c) 2 hr.

a commercial bath. The objective of this study is, therefore, to examine the feasibility of using the aforementioned catalyzation technique with a commercial electroless bath that is designed for the fabrication of flex-circuit and printed circuit boards. The effect of plating bath temperature on the adhesion of the printed catalyst and plated copper is of interest. In addition to the evaluation of the rate of copper deposition, there is also interest in the knowledge of the microstructural quality of copper deposited by the present catalyzation technique.

Experimental Procedure

The experimental apparatus used in this investigation consisted of a 28-L polypropylene tank (Nalgene Industrial Products). The heating system used was a 300 series stainless steel (Cole-Parmer Model 7305) electric immersion heater. A schematic diagram of the experimental setup is as shown in Fig. 1. The immersion heater was clamped to the side of the tank and operated at a low speed of 7 L/min. Its rotatable pump outlet nozzle was pointed along the side of the tank wall to produce best fluid agitation. The heater was also equipped with over temperature protection (OTP) thermostat feature that prevented the unit from burning out when a low liquid level condition existed in the tank. The temperature controller in the heater maintained the set bath temperature within ±0.5ºC. The bath was continuously filtered using 5-micron polypropylene bags and continuously aerated with the help of air spargers. The bath was partitioned using an inert polypropylene board that was fairly resistant to high temperatures in the bath. The purpose of the polypropylene board was to provide a quiescent area within the plating bath where the samples were placed.

After immersing dummy boards for about 1.5–2 hours in the bath, the electroless bath was activated. For each run, about eight catalyzed polyimide samples were immersed in the bath. These polyimide samples were printed with simple circuit patterns. The catalyst formulation is described in Reference 6. Enough space was provided between the polyimide circuit panels to avoid physical contact between samples and also provide for the release of gas bubbles generated during plating. Circuit panels were handled with gloves to prevent finger oil and skin acid on the surface of the substrates. Two proprietary baths were studied: (1) a high-speed bath[*] and (2) a slow-speed bath.[**] Some runs were conducted in a 3000 mL beaker.

During plating, samples were checked for peeling, blistering and similar undesirable effects every half to one hour. It was observed that the deposition of copper on the catalyzed substrates occurred after an initiation period of 10-15 minutes. A sample was removed from the bath every hour. It was cleaned with distilled water and then dried. The last sample was removed from the bath after it had been immersed in the bath for 8 hours. The thickness of copper deposited on each of the samples was determined using the alpha step 200 surface profiler (Tencor Instruments). The bath ingredients were also monitored every three hours.

The electroless deposited samples were studied using the scanning electron microscope (SEM) (JEOL - JSM 840A). To obtain detailed information about the surface morphology, such as grain structure and roughness of the copper deposited, the samples were analyzed using a tapping/contact mode Atomic Force Microscope (Digital Instruments, Inc., Model: Dimension 3000). All the samples were studied in air, and the surface of the copper was scanned with etched silicon tapping probes. The crystal structure of the copper deposit was studied with a Philip's X-ray diffractometer (Philips, X Pert) using Cu Kα radiation (λ=1.5406 Å). The operating voltage was 45 kV and the current was 40 mA.

Results & Discussion

Evaluation of the Electroless Copper Bath

The rate of copper deposition has been shown to be dependent on the concentrations of chemicals in the bath, level of bath agitation, bath temperature and the type of substrate on which copper was deposited.[11] In this study, the influence of plating temperature of a commercial Cu/EDTA/ formaldehyde/NaOH bath on catalyzed polyimide substrates was evaluated. A high-speed bath with a suggested optimal operating temperature of 70ºC and a slow-speed bath operated at room temperature were evaluated. Plating experiments were

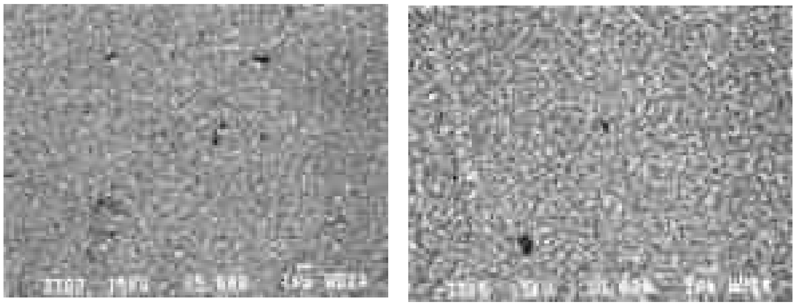

Fig. 6—SEM images of copper deposits at long immersion times in the slow-speed bath (large tank) at 28ºC: (a) 6 hr; (b) 8 hr.

Roughness Values at Different Deposition Times for the High-speed Bath—65°C, Large Tank

Time, hr	R_{rms}, µm	Z_{range}, µm
0.5	0.04	0.205
3.0	0.046	0.276
6.0	0.067	0.686

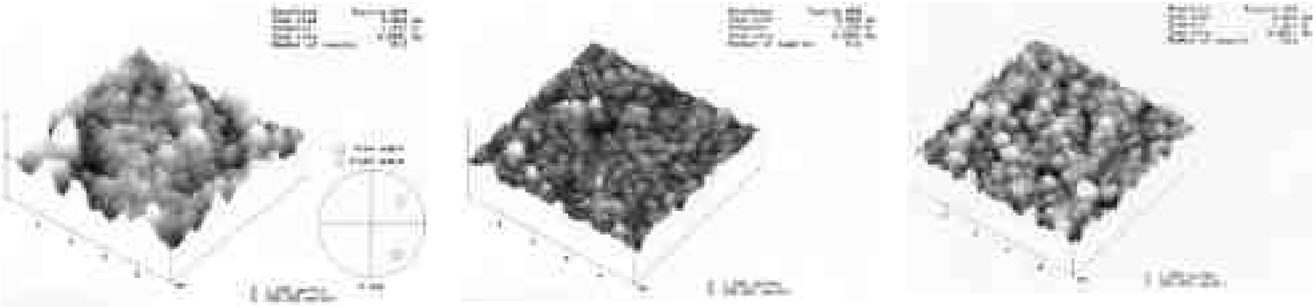

Fig. 7—Three-dimensional TMAFM images of copper surfaces at different immersion times (high-speed bath, 65ºC, large tank): (a) 30 min; (b) 1 hr; (c) 3 hr.

initially carried out in the large plating tank at a temperature of 70ºC to evaluate the high-speed bath. At the end of two hours, it was observed that the adhesion of deposited copper film to the substrate was poor and there was high internal stress (evident on the surface of deposit) on the deposits. These problems shortened the deposition time at 70ºC to only two hours in the large tank and to about 3.5–4 hours in the beaker (with minimal agitation). Figure 2 shows typical plating rate relationship obtained at 70ºC in the tank for two experimental runs carried out at identical conditions of temperature, concentration of bath ingredients and bath agitation. As expected, the plots show an increase in the deposit thickness with time. The thickness of copper deposited on the substrate as a function of time is plotted on a log-log scale because the plating rate relationship is expected to follow the general relation,

$$\text{Plating thickness} = \alpha(\text{Time})^{\beta} \qquad (1)$$

where constants α and β depend on the experimental conditions. Therefore, the values of α and β are obtained from the slope and intercept of the plot on a log-log scale.

Because of the adhesion problems encountered at the bath's optimal operating temperature of 70ºC, a lower operating temperature was evaluated. Experimental runs were conducted at a temperature of 65ºC in both the large tank and beaker. The agitation for the beaker scale was accomplished with a magnetic stirrer that was operated at a low speed. At 65ºC, the plating time was extended to about three hours in the large tank and to six hours in the beaker before blistering and signs of poor adhesion were observed. The long plating time achieved in the beaker before blistering set in was attributed to the stirring effects. For the present catalyzation method, the results suggest that although stirring of the solution ensures an improved mass transport from the bulk of the solution to the substrate, it also has a shearing action on the surface of the copper deposit. As a result, it leads to poor adhesion between the copper deposited and the underlying substrate. The agitation in the bath, therefore, is a critical factor in determining the quality of the copper deposit.

Figure 3 presents the plating rate relationship under different plating conditions on a log-log scale. The figure shows that the plating thickness achieved at short immersion time in the high-speed bath at 70ºC is greater than the thickness achieved in the other bath conditions. The observed differences between the 65ºC and 70ºC runs at short immersion time in the tank is due to the differences in the rate of reactions occasioned by the temperature difference. This is true because it is known that many reactions double their rates for each 10 Cº rise in temperature.[2] At long immersion times however, the deposit thickness obtained when the high-speed bath is operated at 70ºC in the tank is comparatively lower than that obtained when the bath is operated at 65ºC in either the beaker or tank. The observation indicates that in addition to chemical kinetics, several other factors affect the achievable plating thickness. In the catalyzation method used in this study, it appears such factors may include adhesion, peeling and stirring, or agitation effects. Such undesirable effects seem to increase with bath temperature for the catalyzation method used in this work.

Early in the deposition, it appears that the rate of copper deposition is higher in the beaker than in the large tank at 65ºC (Fig. 3). However, at long deposition times, the plating thickness achieved at 65ºC in both the beaker and large tank are essentially equal. The differences observed early in the deposition can be due to a combination of several factors including stirring effects, catalytic activity of substrates and measurement errors. Below a temperature of 65ºC, the bath was inactive and could not plate in either large tank or beaker.

Plating experiments were carried out at room temperature (28ºC) with moderate agitation to evaluate the low temperature bath. Deposition times of eight hours were achieved without signs of poor adhesion, peeling or blistering. The plating rate relationship of the copper deposited at this temperature is also shown in Fig. 3. As expected, Fig. 3 indicates that

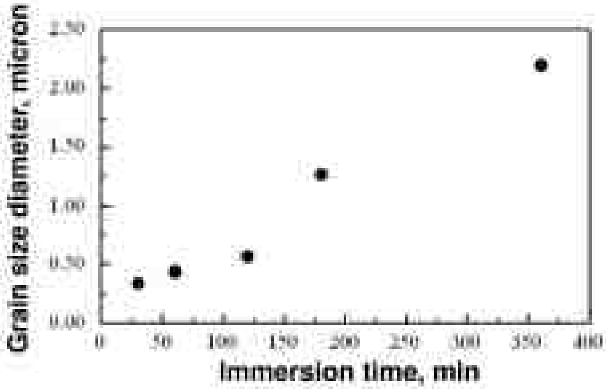

Fig. 8—Variation of grain size with immersion time (high-speed bath, 65ºC, large tank).

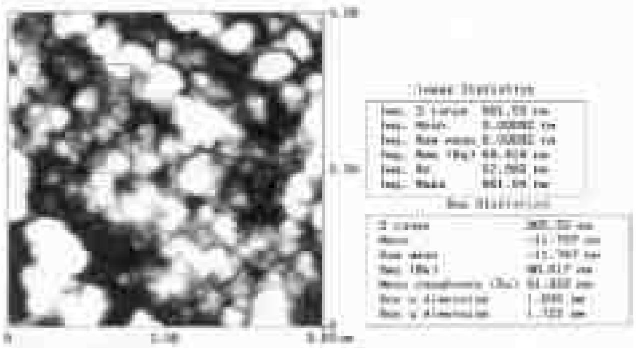

Fig. 9—Roughness analysis for the copper deposit obtained from the high-speed bath (0.5 hour immersion time, 65ºC, large tank).

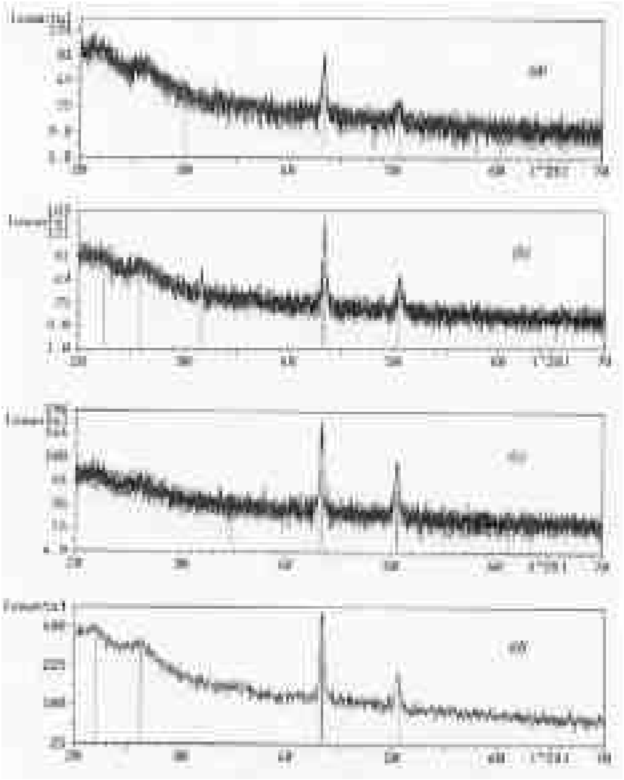

Fig. 10—XRD patterns for the copper deposited at 65ºC in the large tank with the high-speed bath. (a) 0.5 hr; (b) 2 hr; (c) 2.5 hr; (d) 5 hr.

the plating rate is lower at the lower bath temperatures. For instance, the average plating rate was 1.5 µm/hr at a temperature of 65ºC (moderate agitation) while an average plating rate of 0.99 µm/hr was obtained for the low temperature (28ºC) bath. Because of the substantial immersion time and thickness achieved in the high-speed bath at 65ºC with moderate agitation, further studies on copper deposits from the bath were conducted with samples obtained at this temperature.

As seen in Fig. 3, when the high-speed bath is operated in a beaker at 65ºC with slow agitation, long deposition times and substantial film thickness values are obtained. It then suggests that for the particular catalyzation technique used in this work, a bath temperature of 65ºC is preferred over the suggested 70ºC for the proprietary high-speed bath. The above results indicate that to obtain the necessary adhesion between the plated copper and the polyimide substrate and a copper thickness of 10 µm (an important thickness required for flex-circuits) within four to five hours, the commercial high-speed bath must be operated at a temperature of 65ºC with moderate agitation. Evaluation of both baths indicates a strong influence of temperature on film adhesion, thickness and plating time.

Figure 3 shows that the plating rate data obtained from the experimental results fit the general relation given by equation (1). The constants α and β are found to have values of 0.044 and 0.870, respectively, for the high-speed bath (65ºC) with slow stirring. On the other hand, when the slow-speed bath is operated at room temperature with minimal agitation, a similar plating rate relationship with α and β taking values of 0.040 and 0.770, respectively, are obtained. Although limited in scope, the results suggest that the value of the exponent β is related to the level of bath agitation. Figure 3 also indicates that at long deposition times, the growth rate of the copper film seems to slow down considerably, though the concentrations of the bath ingredients are maintained at optimum values. The evaluation of the baths therefore indicates the significance of bath temperature and agitation for the particular catalyzation method used. Whereas the present catalyzation technique seems disadvantaged by its unsuitability for high plating bath temperature and high plating rate, it seems suited for intermediate plating temperatures and its good activation conditions.

Morphology of Deposited Copper Samples

Scanning Electron Microscopy

SEM images that show the growth of copper deposits at different immersion times in the high-speed bath at a temperature of 65ºC are shown in Fig. 4. As expected, an increase in grain size with deposition time is observed. In terms of grain distribution, Fig. 4a shows a dispersed surface of copper particles (at a deposition time of 0.5 hour), while Fig. 4c shows a uniformly covered copper surface (at a deposition time of 2 hours). The increase in coverage with time is expected as self-catalyzation of the copper deposit progresses with time. The cracks observed in Fig. 4b may be connected with the dehydration of the carrier polymer used for substrate catalyzation. Cracks are not present in the other samples. When compared to Fig. 4a, Fig. 4b shows both an increase in grain size and surface coverage uniformity. In Fig. 4b, differences in grain size are observed between the grains formed earlier compared to the most recently-initiated grains. The recently-initiated grains are seen on the uncovered areas of an "earlier time period," *e.g.*, Fig. 4a. The SEM results give a qualitative analysis of the growth phenomenon, but an accurate quantitative grain size analysis with time was done using atomic force microscopy (AFM) as shown later.

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[*] *Shipley Circuposit 71®, Shipley Company, Newton, MA.*

[**] *Shipley 328Q®, Shipley Company, Newton, MA.*

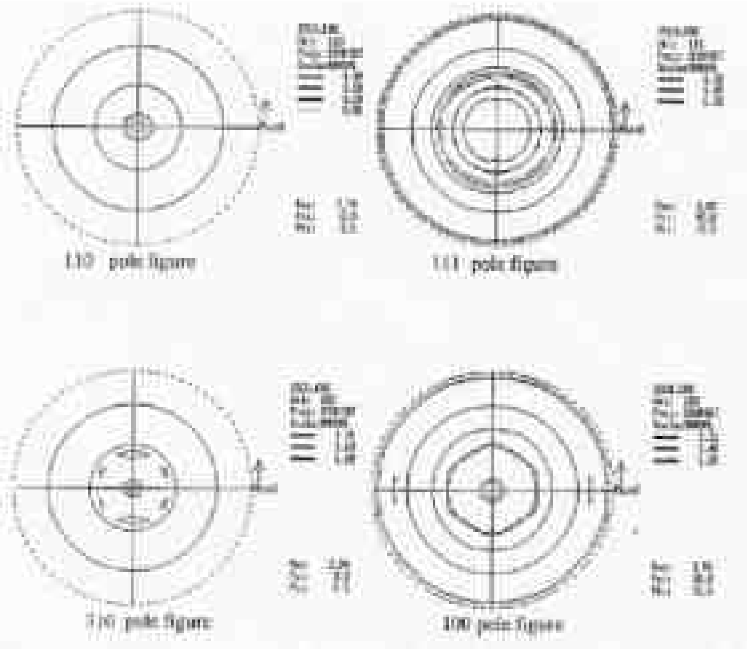

Fig. 11—Pole figure analysis of copper samples (immersion time = 2.5 hr, high-speed bath, large tank, 65ºC).

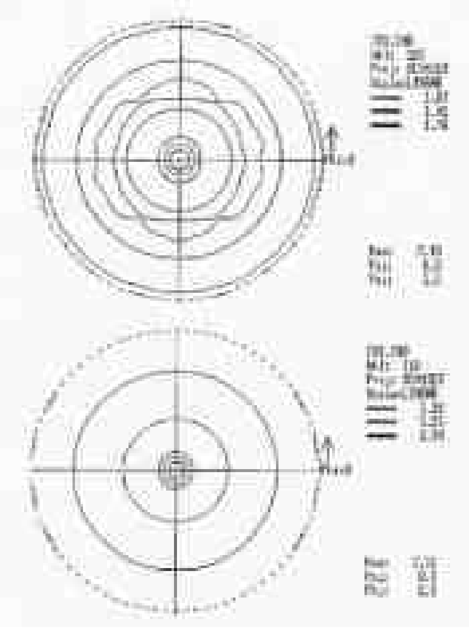

Fig. 12—Pole figure analysis of copper samples deposited in 5 hr (large tank, 65ºC, high-speed bath).

Figure 5 shows the SEM images of copper deposited at various immersion times in the low speed bath. As shown, no significant difference exists (with respect to the growth phenomenon) between the SEM images of the high-speed bath and the slow-speed samples. In terms of grain distribution, the SEM image of the copper deposit obtained at a short immersion time (*e.g.,* 30 min) in the high-speed bath looked similar to the SEM image of deposit obtained at the same immersion time in the slow-speed bath. The similarity in appearance may be because of the catalyst nucleation sites on the substrates. The nucleation sites on the substrates are independent of plating bath temperature. However, as immersion time increased, we observed that the size of the grains deposited by the high-temperature bath differed from those deposited by the low-temperature bath. This is probably due to the differences in the plating rate of the baths. The grains at an immersion time of 2 hours in the high-speed bath therefore appear better defined in shape, larger in number and probably in size than the grains obtained in the slow-speed bath at the same immersion time. It appears that the grains obtained at a residence time *t*, in the low-temperature bath will equal the size of grains deposited at a fraction of immersion time *t* in the high-temperature bath. In terms of shape, we observe that the morphology of the high-temperature samples differed from those of the low-temperature runs. However, for both runs, uniform deposits were obtained at immersion times greater than 1.5 hours. After 8 hours of deposition in the slow-speed bath, a dense copper covered surface was obtained. However, the copper thickness obtained after 8 hours of immersion in the low speed bath was still lower than the deposit obtained in 4 hours at 65ºC.

Figure 6 compares the deposits obtained in the slow-speed bath at long immersion times. The figure shows that at long immersion times, the grains tended to elongate in size. This contrasts with the morphology of rounded grains observed at 0.5 and 1-hour deposition times (*c.f.*, Fig. 6 to Figs. 5a and b). The reason for this elongation can only be attributed to a possible two-dimensional growth with time for surface coverage. For some samples, evidence of hydrogen evolution from the surface of the deposit was shown by the presence of voids. Internal stresses are thought to be responsible for the blistering on the surface of the deposits.

Atomic Force Microscopy

Figure 7 shows the three-dimensional TMAFM images of the copper surface at different immersion times. The figures show random distribution of copper particles on the surface of the catalyzed substrate. Sectional analysis of the copper deposit provided information on the grain size. As expected, a steady increase in grain size was observed with deposition time. Figure 8 is a plot of the grain size diameter with time. During the first two hours of deposition, the hourly increase in grain size was about 1.3 times its previous value. During the next three hours of deposition, the magnitude of increase in grain size per hour more than doubled. This observation can be explained on the basis of the phenomenon of migration of the copper particles. Initially, the copper particles are tightly bound to the surface of the substrate, but soon the self-catalytic process of copper deposition results in loosely bound particles on the surface that migrate and agglomerate forming mounds of copper that are huge by comparison. This explains the two-fold increase in grain size.

We can conclude that during the initial stages of electroless copper plating, fine copper particles (diameter < 0.35 µm) deposit on the polyimide substrate. Subsequently, copper crystals filled in the numerous pores with diameters < 2.2 µm leaving no apparent gaps. The TMAFM analysis for the grain sizes confirmed the observation from the SEM images (Fig. 4) that the copper particles gradually grow in size to form a thin uniform layer on the substrate.

Figure 9 indicates the roughness values on a selected area of the copper deposit. The root mean square (rms) roughness value was calculated using the formula

$$R_{rms} = \{(Z_i - Z_{ave})^2 / N\}^{0.5} \qquad (2)$$

where Z_i is the height of an individual pixel, Z_{ave} is the average height within a frame area, and N is the total number of pixels. The Z_{Range} value is the total height range analyzed (highest to lowest points). As can be seen from the figure, at a thickness of 0.8 to 1.0 µm obtained at a deposition time of 0.5 hour, a smooth surface with a root mean square (rms) roughness value of 40.6 nm was obtained. This is expected since an earlier work[12] shows that at the end of nucleation stage (a few nanometers in thickness) in the growth process of an electroless film, a smooth film forms. As can be seen from the table, both the rms and Z_{Range} roughness values increase with deposition time. This trend can be explained by the formation of aggregates of copper particles on the thin layer of copper film occasioned by the self-catalytic behavior of the copper deposition process.

X-ray Diffraction

The crystal structure was studied using X-ray diffraction (XRD). The study confirmed the cubic structure of copper. Figure 10 shows the XRD patterns for the copper-deposited samples with increasing deposition time at high temperature. There are features in the X-ray diffraction spectra that indicate that the films consist of polycrystalline copper. Despite their weak intensities, lines in the diffraction pattern were observed at Bragg angles that corresponded to copper diffraction peaks of (111) and (200). The copper (111) peak ($2\theta = 43.297^\circ$) becomes stronger with increasing deposition time. This trend is expected from an increase in time with the deposition of copper crystals of (111) orientation. The additional peaks appearing in the XRD pattern, for example the (200) peak at ($2\theta = 50.433^\circ$), indicates the polycrystalline nature of the copper film.

To obtain information about the preferred orientation of the copper crystals, the stereographic projections of the samples were studied. Figures 11 and 12 show the stereographic projections at different deposition times of 2.5 and 5 hours, respectively. From the figures, it can be seen that the copper crystals showed a preferred orientation in the (110) direction. This result is in good agreement with earlier work done by Matsuoka, *et al.*[13] In their work, NaOH was the alkali used in the formulation of the electroless copper bath solution. Figure 11 indicates that the intensity in the (110) direction is almost four times that of the background, while the intensity in the (100) direction is almost twice. Figure 12 also indicates that the intensity in the (110) direction is more than twice that of the random orientation while the intensity in the (100) direction is nearly twice that of the random orientation. This result indicates that a large number of crystals were predominantly deposited in the (110) direction.

A study of the stereographic projections of samples plated for 1.5 hours showed that the intensities in all the three directions, (110), (111) and (100) were in the same range. We can conclude that no preferred orientation of the crystals was observed after 1.5 hours of deposition. An analysis of samples plated for 2 hours was found to be similar to the results obtained after 1.5 hours of deposition. For the 2-hour samples, the intensities in the (110), (111), (310), (100) directions were all in the same range. It appears, therefore, that a substantial deposition time of ≥ 2.5 hours is required to analyze the texture of the copper film formed from the present catalyzation method.

The apparent crystallite size of the copper deposited can be determined from the broadening of the diffraction with the help of Scherrer's equation.[13] Matsuoka, *et al.*[13] have shown that the relationship between the mechanical properties and the crystal structure of copper films can be interpreted on the basis of Schmid's law. In order to verify the suitability of the material deposited for printed circuit board applications with respect to its mechanical properties, the approach given by Matsuoka, *et al.* can be adopted. It is, however, not done here since it is outside the scope of the present work.

Conclusions

In the investigation of the feasibility of the catalyzation method described, it was found that the thickness of the copper deposit is limited by the adhesion of copper to the polyimide substrate. Therefore, an interfacial analysis of the copper/catalyst surface and the catalyst/polyimide interface is recommended to establish the feasibility of this catalyzation technique for metallization using commercial baths. The sensitivity of the catalyst to high temperature and shearing is outlined. It is found that a temperature of 65ºC with minimum bath agitation must be maintained to obtain optimum results for the high-speed bath. The low-temperature bath suggests a most likely route to the utilization of the catalyzation method. However, the plating rate at the low temperature is not high enough to economically meet the needs of the flex circuit and PCB industry. A plating temperature of 65ºC is therefore prescribed. Preliminary surface characterization of copper deposits was performed and showed that the copper deposit obtained possessed the requisite property of copper used for flex-circuits. A detailed study of the mechanical and electrical properties of the copper deposits is recommended to further confirm the feasibility of the one-step, fully additive plating technique.

Acknowledgments

This work was partially supported by the United States Department of Energy under contract no. DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the United States Department of Energy. We acknowledge the financial and analytical support from the Materials Research and Technology Center (MARTECH) at Florida State University. The technical assistance of Dr. Eric Lochner of MARTECH is acknowledged. The Chemical Engineering Department, FAMU-FSU College of Engineering assisted in meeting the publication costs of this article.

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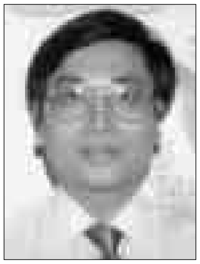

Dr. Ken S. Chen is a principal member of the technical staff at Sandia National Laboratories. He earned his PhD in Chemical Engineering from the University of Minnesota in 1992. He is an author/co-author of two US patents on additive metallization of polymeric substrates via Pd-catalyzation and electroless/electrolytic plating. His current research activities focus on the modeling of electrochemical processes such as electroplating, batteries, and atmospheric metal corrosion.

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