Copper electroplating formulas composed of CuSO₄, H₂SO₄, chloride ions, polyethylene glycol (PEG), bis (3-sulfopropyl) disulfide (SPS), and different levelers for microvia filling of a printed circuit board (PCB) were studied. The influence of the copper electroplating parameters, accelerator and leveler concentrations and cathodic current density on the microvia filling performance was explored using the Taguchi experimental design method. An L9 orthogonal array with four controlling factors at three levels was employed in the experimental design method. Variance analyses of the mean filling performance and the signal-to-noise ratio of the controlling factors showed that the most significant factor for the microvia filling performance was the leveler concentration. The other factors exhibited little to no effect on the microvia filling performance. The contribution of levelers to the filling performance was characterized using galvanostatic measurement and linear sweep voltammetry (LSV) with a working electrode at different rotational speeds. The experimental results indicated that the inhibiting effect of the levelers on the copper deposition was related to forced convection, which is a key physicochemical interaction for exhibiting good filling performance.

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Optimization of the Copper Plating Process Using the Taguchi Experimental Design Method

I. Microvia Filling by Copper Plating Using Dual Levelers

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German), leveler A and leveler B were additionally added into the base electrolyte for all electrodeposition experiments and electrochemical analyses. Leveler A contains nitro functional groups, while leveler B is a quaternary ammonium compound. The temperature of the plating solution was maintained at 28 °C for all of the plating tests and analyses.

**Electrochemical analysis.** Various formulas were characterized with galvanostatic measurements (GMs) and cyclic voltammetry (CV) at different rotational speeds of a rotating disk electrode (RDE). All of the electrochemical analyses were performed in a glass vessel containing 150 mL of electrolyte using a PGSTAT 302 potentiostat (AUTOLAB) with a three-electrode cell system. A platinum RDE with a 3 mm diameter was employed as the working electrode. A phosphorus-containing copper rod was chosen as a counter electrode (CE), and a saturated mercuric sulfate electrode (SMSE) was used as a reference electrode (RE). To characterize the polarization behavior of various formulas, a thin copper layer of 1 µm thickness was predeposited onto the platinum RDE in a preposition bath that contained 0.88 M CuSO4 (Riedel-de Haen, ACS) and 0.54 M H2SO4 (Merck, 96%, Ultrapure) before galvanostatic measurements.

100 rpm and 1000 rpm of the Cu RDE were chosen to simulate the fluid flow inside and outside a microvia, respectively. The cathodic current density was fixed at 1.94 A·dm⁻² in the GMs. After GMs were performed for 400 seconds, 1 ppm SPS was injected into the base electrolyte. Following the injection of SPS, 1 ml·L⁻¹ leveler was injected into the electrolyte. After 800 seconds, another leveler was injected into the electrolyte.¹⁸ The filling performance of various formulas was characterized by a mean potential difference (Δη) between the two polarization curves measured with the Cu RDE that was individually operated at 100 rpm and 1000 rpm. It was showed that if the copper-plating formulas can perform bottom-up filling of a microvia, then a positive potential difference (i.e., Δη = E₁00rpm - E₁000rpm > 0) occurs.²⁵,²⁷

To characterize the impact of forced convection on the filling performance, CV was carried out using four rotational speeds (i.e., 100, 400, 700 and 1000 rpm). The CV was started from an open circuit potential (OCP) to −0.7 V vs. SMSE, followed by a positive sweep from −0.7 V to 0.9 V vs. SMSE for five cycles. The scan rate was 100 mV·s⁻¹ for all of the CV measurements.

**Numerical analysis.** The microvia fillings were plated in a Harvard cell with a continuous air bubble flow. To understand the influence of the fluid flow on the function of these additives, the flow field around the microvia was simplified using a two-dimensional model.³³–³⁶ The continuity equation and the Navier-Stokes equation for an incompressible fluid are as follows, with a steady-state assumption:

\[
\nabla \cdot \mathbf{u} = 0
\]

\[
-\mu \nabla^2 \mathbf{u} + \rho (\mathbf{u} \cdot \nabla) \mathbf{u} + \nabla p = 0
\]

where \( \mathbf{u} \) denotes the velocity vector, \( \mu \) is the viscosity of the fluid (i.e., 1.6 × 10⁻³ Pa·s), \( \rho \) is the density of the fluid (i.e., 1158 kg·m⁻³) and \( p \) is the pressure of the fluid.

Figure 1 shows the boundary conditions. Boundary 1 depicts the average velocity of the continuous air bubble flow at 10 m·s⁻¹. The lengths of boundaries 2 and 8 represent the distance of 1 cm between the air bubble flow and the cathodic surface; a fully developed velocity profile is assumed on these boundaries. Boundaries 3–7 depict the surface of a microvia and are regarded as non-slip surfaces. The fluid dynamics model was solved numerically using the COMSOL software.

**Microvia filling by copper electrodeposition.** The PCB samples for the plating tests were drilled by CO₂ laser ablation to form identical microvias with a diameter of 95 µm and a depth of 85 µm. Following the laser drilling, the PCB samples were subjected to a desmearing process for smear removal, copper electroless deposition for sidewall metallization of the microvias, and copper electroplating for thickening the sidewall copper to prevent it from oxidation. The dimensions of the samples were 4.5 cm × 6 cm.

Two phosphorus-containing copper plates were employed as anodes and placed directly in the plating bath with a working volume of 700 mL. The electrolyte was constantly agitated by continuously flowing air bubbles with a flow rate of 1.5 L·min⁻¹ during the electroplating. The temperature of the plating electrolyte was maintained at 28 °C during the electroplating. The filling performances of the various plating formulas were defined by \( H_2/H_1 \times 100\% \), as illustrated in Fig. 2.³⁷ The filling performance was assessed according to the cross-section of the microvia, which was examined using an optical microscope (OM, Olympus BX51).

**Taguchi experimental design method.** The Taguchi experimental design method⁴⁰ was employed to analyze the influence of the experimental parameters on the copper filling performance. Table I shows the controlling factors and levels designed for the microvia filling experiment. An L₉(³⁴) orthogonal array with four factors at three levels, as listed in Table II, was used in this study. The S/N ratio was calculated according to the “nominal-the-best” case. The ANOVA of

| Table I. Control factors and levels. |
|---------------------------------------|
| Factor                  | Level 1 | Level 2 | Level 3 |
|-------------------------|---------|---------|---------|
| A: Leveler A (ml·L⁻¹)   | 1       | 2       | 4       |
| B: Leveler B (ml·L⁻¹)   | 1       | 1.5     | 2       |
| C: SPS (ppm)            | 1       | 2       | 3       |
| D: Current density (A·dm⁻²)| 1.29    | 1.62    | 1.94    |

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The mean filling performance and the S/N ratio were used to determine the significance and contribution of each control factor.

Results and Discussion

Electrochemical analysis.— According to CDA theory, the mass transfer of chloride ions is convection-dominant, rather than diffusion- and migration-controlled and the functions of the suppressor, leveler and accelerator depend on the surface coverage of chloride ions, so the adsorption of the suppressor, leveler and accelerator used for the microvia filling is convection-dependent. The suppressor and leveler dominate copper deposition at a high surface coverage of chloride ions due to a strong convection; on the other hand, the accelerator dominates copper deposition at a low surface coverage of chloride ions due to a weak convection. These phenomena lead to a different copper deposition rate between the mouth and the bottom of a microvia. The CDA mechanism can explain that if a copper plating formula can lead to good filling performance in microvias, then it also exhibits a positive cathodic potential difference ($\Delta \eta = E_{1000rpm} - E_{1000rpm > 0}$) in GM, as was defined in previous works. The GM analyses with additives injection are shown in Fig. 3. When 1 ppm SPS was injected into the basic plating electrolyte, the CDA behavior was not observed (i.e., $\Delta \eta = 0$). After adding leveler A or B of 1 ml to the electrolyte, the $\Delta \eta$ was significantly positive (i.e., $\Delta \eta_1 = 15$ mV and $\Delta \eta_2 = 13$ mV). These results show that leveler A or B interacts with SPS and PEG-Cl to exhibit CDA behavior. When both levelers A and B of 1 ml were simultaneously present in the electrolyte, the $\Delta \eta$ became larger (i.e., $\Delta \eta_1 = 18$ mV and $\Delta \eta_2 = 25$ mV) than $\Delta \eta_1$ and $\Delta \eta_2$. The CDA behavior was obviously enhanced due to the simultaneous addition of levelers A and B.

According to the GM analysis, the copper deposition rate decreases upon increasing the forced convection in the presence of levelers. To evaluate the correlation between the copper deposition rate and the convective strength, CV measurements of various plating electrolytes were carried out. The charges of the copper stripping peaks in the CV measurements are shown in Fig. 4. Formula A, containing only PEG, Cl$^-$, and SPS, did not exhibit CDA behavior because the charge of the copper stripping was almost independent of the rotational speed of the Cu-RDE (i.e., $\Delta C = 0.752$ mC). After adding 1 ml of leveler A or B into the electrolyte (i.e., formula B and formula C), these plating formulas became sensitive to the rotational speed of the Cu-RDE, showing that stronger forced convection resulted in less copper deposition. Hence, the deposition rate at the bottom of the microvia was greater than that outside of the microvia.

In contrast with formula B, the variation in the copper deposition rate caused by various rotational speeds in formula C ($\Delta C = 1.173$ mC) was smaller than that using formula B ($\Delta C = 2.103$ mC). Therefore, the acceleration of the copper deposition at the bottom of the microvia using formula C was weaker than that using formula B, leading to a flat copper profile at the upward-moving copper surface, whereas a copper bump profile was formed with formula B. When 1 ml of both levelers A and B were simultaneously added to the plating electrolyte (i.e., formula D), the best filling performance was achieved, as shown in Fig. 4. A flat copper profile combined with a thin copper layer was deposited on the board surface, because formula D resulted in the largest $\Delta C = 2.526$ mC.

Numerical analysis.— Figure 5 illustrates the velocity profile in the proximity of the RDE. The flow behavior of the electrolyte on the surface of the RDE is characterized as

$$V_r = 0.5\omega^{1/2}v^{-1/2}rY_h$$

where $Y_h$ denotes the thickness of the moment boundary layer (i.e., from $V_r = 0$ to $V_r = V_c$ max), $v$ is the kinetics viscosity of the electrolyte, $\omega$ is the rotational speed of the RDE, $V_r$ is the tangential velocity on the rotating disk, and $r$ is the distance from the center of the rotating disk. Therefore, if one knows the ratio of $V_r/Y_h$, one is able to theoretically calculate $\omega$.

The velocity distribution in the microvia as a function of the plating time was simulated as shown in Fig. 6. In the simulation model, the velocity profile on the copper surface of the microvia was assumed to be linear. Additionally, the velocity profile shown in Fig. 5 is linear from the surface of the RDE to the maximum velocity. To correlate the velocity profile on the RDE shown in Fig. 5 with that on the surface of
Figure 5. Vector representation of the fluid velocity near a rotating disk and microvia.

The PCB during plating shown in Fig. 6, the ratio $m = V_{x \text{ max}}/Y_h$ on the RDE at various rotational speeds was compared with the ratio $m = V_{x \text{ max}}/Y_h$ on the PCB that was obtained from the simulation model and shown in Fig. 7a. The simulation result shows that 100 rpm and 1000 rpm used in the GMs may represent the flow velocities at the via bottom and mouth, respectively.

Figure 6. Cross-sections of microvias after copper filling at various plating stages and the corresponding fluid flow simulations. The electroplating time was (a, b) 15 minutes, (c, d) 30 minutes, (e, f) 45 minutes, (g, h) 60 minutes. The diameter and depth of the microvia were 95 $\mu$m and 85 $\mu$m, respectively. The plating solution contained 0.88 M CuSO$_4$, 0.54 M H$_2$SO$_4$, 200 ppm PEG, 60 ppm Cl$^-$, 1 ppm SPS, 1 ml$^{-1}$ leveler A, and 1 ml$^{-1}$ leveler B.

Figure 7. (a) The relationship between the m values of a microvia and the corresponding rotating speeds on the Cu-RDE, which was obtained according to Eq. 3. (b) the variations of the m values of a microvia and the corresponding rotating speeds on the Cu-RDE. The dash lines, indicated by (△) and (○), are the m values at the via mouth and via bottom, respectively. The solid lines, indicated by (◦) and (□), are the corresponding rotating speeds simulated at the via mouth and the via bottom, respectively.
In contrast with the via mouth, the flow velocity at the bottom first decreased with the decreasing bottom area when the plating time proceeded from 15 minutes to 30 minutes, as shown in Figs. 6a–6d. The corresponding rotational speed of the Cu-RDE at the via bottom, estimated by simulation and calculation, was 73 rpm at the plating time of 15 minutes and 17 rpm at the plating time of 30 minutes, as shown in Fig. 7b. The flow velocity on the PCB surface was almost fixed at 1180 rpm during plating, as shown in Fig. 7b. A significant enhancement in the filling performance was not observed at the bottom center from 15 minutes to 30 minutes, but occurred at the bottom corner, as shown in Fig. 6c. This result was attributed to the lowest coverage of chloride ions occurring at the corner of the bottom area because both the strongest electrostatic repulsion and the weakest convection occurred there, as confirmed in Fig. 6d. Hence, the suppressor (i.e., PEG-Cl\(^{-}\)) was not easily adsorbed there,\(^37\) and the SPS easily replaced the PEG and adsorbed at the corner to form the V-shaped copper profile in Fig. 6c.

The convection strength at the via bottom continued to weaken with the bottom area shrinkage from 15 minutes to 30 minutes, as predicted in Figs. 6b, 6d and Fig. 7b. The weakest inhibition and strongest acceleration that was caused by the weakest convection and the strongest electrostatic repulsion of chloride ions interacted at the via bottom, so that the fastest copper deposition rate occurred, as shown in Figs. 6c and 6e. When the convective strength between the via mouth and the via bottom approached similar after the fastest copper deposition rate occurred, as shown in Figs. 6c and 6e. When the convective strength between the via mouth and the via bottom rapidly approached to that of the via mouth after 30 s, as predicted in Figs. 6f, 6h and Fig. 7b. The flow velocity on the PCB surface was almost fixed at 1180 rpm during plating, as shown in Fig. 7b. A significant enhancement in the filling performance was not observed at the bottom center from 15 minutes to 30 minutes, but occurred at the bottom corner, as shown in Fig. 6c. This result was attributed to the lowest coverage of chloride ions occurring at the corner of the bottom area because both the strongest electrostatic repulsion and the weakest convection occurred there, as confirmed in Fig. 6d. Hence, the suppressor (i.e., PEG-Cl\(^{-}\)) was not easily adsorbed there,\(^37\) and the SPS easily replaced the PEG and adsorbed at the corner to form the V-shaped copper profile in Fig. 6c.

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Table III shows that the experiment number 1 exhibited the best S/N ratio; that is, it had the highest FP (%) and the smallest absolute value of the S/N ratio. Figure 9 shows the plots of the S/N ratio versus the various factor levels. The analysis shows that the characteristic quality (i.e., FP %) decreases with increasing leveler A concentration. The other factors result in the best characteristic quality at intermediate levels. The contribution and significance of the variable factors was calculated using variance analysis (ANOVA), as listed in Table IV. Table IV shows that the leveler A, SPS, and current densities are significant factors in the filling performance. In contrast, leveler B has a less-significant contribution to the filling performance. From Fig. 9, the optimum level of each factor is 1 ml (A1) for leveler A, 1.5 ml (B2) for leveler B, 2 ppm (C2) for SPS, and 16.2 A dm\(^{-2}\)(D2) for the current density. The combination of the optimal levels, A1-B2-C2-D2, was checked with a plating experiment to confirm its performance. The predicted filling performance of the optimal factor combination of A1-B2-C2-D2 was calculated from Eq. 7:

$$y_{predicted} = y_{avg} + \sum_{i=1}^{7} (y_{opt} - y_{avg})$$

Figure 9. S/N graph of the filling performance.
Table IV. ANOVA results of filling performance in micropores.

| Factor        | Sum of squares (S) | Degree of freedom (F) | Variance (V) | Contribution (%) |
|---------------|-------------------|-----------------------|--------------|-----------------|
| Levealer A    | 18900.92          | 2                     | 9450.461     | 38.608          |
| Levealer B    | 3642.816          | 2                     | 1821.408     | 7.441           |
| SPS           | 13149.79          | 2                     | 6574.896     | 26.859          |
| Current density | 13148.42        | 2                     | 6574.21      | 26.857          |
| Error         | 116.0389          | 63                    | 1.841887     | 0.237           |
| Total         | 48957.99          | 71                    |              | 100             |

Table V. Confirmation and Comparison of the optimal conditions.

| Optimal Process parameters | Optimal condition | Optimal condition |
|----------------------------|-------------------|-------------------|
| Level                      | A1B2C2D2          | A1B2C2D2          |
| S/N ratio (db)             | −7.996            | −9.909            |

where y_{predicted} is the estimated S/N ratio, y_{avg} denotes the average S/N ratio of each experiment, y_{i,opt} is the average S/N ratio of the optimal level of each factor and j is the number of parameters affecting the multiple response.

The confirmation result is listed in Table V. If the predicted S/N ratio obtained using the optimal level of each factor is very close to the result of the confirmation experiment, then the Taguchi experimental method has succeeded. From Table V, the predicted S/N ratio of the optimal level combination (i.e., A1-B2-C2-D2) is −7.996 db, which is very close to the experimental value of −9.909 db (see Fig. S2).

Conclusions

In this study, the correlation between the rotational speed of the Cu-RDE and the velocity profile of the copper plating solution near a micropore during the plating process was established by combining an analytical solution and a numerical solution of fluid dynamics. The simulation result can reasonably explain the bottom-up filling mechanism in a micropore. The fluid velocity profile determines the chloride ion distribution inside the micropore during plating, such that the suppressor and accelerator unevenly act inside the microvias, which is referred to as convection-dependent adsorption (CDA). The CDA behavior results in a coverage gradient of chloride ions along the sidewall of the microvias due to the gradient of forced convection. Hence, copper is preferentially deposited at the bottom corner in the beginning because SPS-Cl\(^-\) easily works there, but the PEG-levealer-Cl\(^-\) does not easily function there due to the small number of adsorbed chloride ions.

When the filled copper profile is turned into a V-shape, the weakest fluid velocity appears again in the micropore, but it moves from the bottom corner to the bottom center of the microvias, such that the CDA mechanism starts up again to strongly enhance the copper deposition at the via center until complete filling. The Taguchi experimental design method identified the contributions of levealer A, SPS and current density as 38.6%, 26.9%, and 26.9%, respectively, whereas levealer B had no significant effect on the filling performance, but levealer B can modify the copper surface profile on the via top area, resulting in a flat copper profile.

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