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Shao-Yu Chiu, Jia-Min Shieh, Shih-Chieh Chang, Kun-Cheng Lin, Bau-Tong Dai, Chia-Fu Chen, and Ming-Shiann Feng

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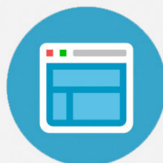

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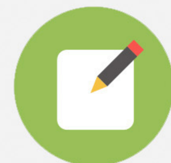

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Characterization of additive systems for damascene Cu electroplating by the superfilling profile monitor

Shao-Yu Chiu
Institute of Materials Science and Engineering, National Chiao Tung University, Hsinchu 30050, Taiwan

Jia-Min Shieh[a)]
National Nano Device Laboratories, 1001-1 Ta-Hseuh Road, Hsinchu 30050, Taiwan

Shih-Chieh Chang and Kun-Cheng Lin
Institute of Materials Science and Engineering, National Chiao Tung University, Hsinchu 30050, Taiwan

Bau-Tong Dai
National Nano Device Laboratories, 1001-1 Ta-Hseuh Road, Hsinchu 30050, Taiwan

Chia-Fu Chen and Ming-Shiann Feng
Institute of Materials Science and Engineering, National Chiao Tung University, Hsinchu 30050, Taiwan

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Gap-filling dynamics of several different species of additives for copper electrodeposition was investigated by monitoring the cross section of a partially filled copper profile on the scanning electron microscopy photo. The filling ration $\Delta y/\Delta x$ between ''bottom-up'' with ''sidewall shift'' was found to be proportional to the filling power of additives. The adsorption-diffusion model combined with cathode polarization and cyclic voltammetric stripping measurements was employed to explain the attribution of additives in superfilling phenomena. The superfilling dynamics was achieved under behavior of additives providing selective inhibition gradient within the damascene feature. By means of those analyses, we have optimized the appropriate amount of additives and achieved the superfilling performance for 0.15 μm vias with aspect ratio 6 by an acid-copper electrolyte with polyethylene glycol, $C1^-$, and 2-mercaptopyridine (2-MP). Due to the additive of 2-MP, chelate formed which enhanced adsorption ability on Cu^0 surface, and the concentration gradient between side-wall shift and bottom-up in the damascene became high enough to attend superfilling electroplating. © *2000 American Vacuum Society.* [S0734-211X(00)13506-1]

I. INTRODUCTION

In the future, copper is going to replace aluminum in ultralarge-scale-integrated circuits (ULSI) metallization because of its low resistivity and high electromigration resistance. In 1997, IBM published their results on fully integrated devices with copper interconnects. Concurrently, a paper discussing six levels of planarized copper interconnects was also published by Motorola.[1–3] Gap-filling requirement becomes the key issue because the damascene process is necessary for copper metallization. The major reason for the damascene process is that traditional process flow for aluminum metallization cannot be directly adopted for copper. Several different deposition techniques could be used to deposit copper films. These include physical vapor deposition, chemical vapor deposition, electroless plating, and electroplating.[4–7] Among these techniques, electroplating emerges as a viable copper-filling technique for damascene processing due to its low deposition temperature, high throughput, high quality and, excellent via/trench filling capability.[8,9]

For the three commonly used electroplating baths, i.e., cyanide, pyrophosphate, and acidic baths, the acidic bath is the most suitable approach for copper wiring in ULSI because of its low cost, ease of control, low toxicity, and high deposition rate.[10] Acid copper plating solutions generally contain copper salt as source of copper ions, a support electrolyte to provide conductivity of the solution, and the addition agents for modifying kinetics of electroplating process and regulating the properties of deposition films. The addition agents commonly include brighteners and levelers.[10] Typically, the overall concentration for addition agents is in the order of parts per million. Nevertheless, both the quality of deposited copper films and gap-filling ability during electroplating are controlled by those minute amount of additives.

Copper electroplating for damascene interconnection of submicron vias and trenches by adding leveling agents in the electrolyte to enhance the filling capability has been investigated.[3,11–14] Most previous studies of copper electroplating with similar bath chemistries utilized either unidentified additives or commercial electrolytes. In our study, we researched additives from the patent literatures[15] that were typical for electroplating unpatterned substrates and found the recipe of the composition and concentration of electrolyte for submicron copper metallization application. The filling capability is a key issue for superfilling in deep submicro and high aspect ratio damascene structure. Accordingly, how to get highly efficient leveling agents is an important work for copper electroplating in ULSI. Several methods for de-

[a)]Author to whom correspondence should be addressed; electronic mail: jmshieh@ndl.gov.tw

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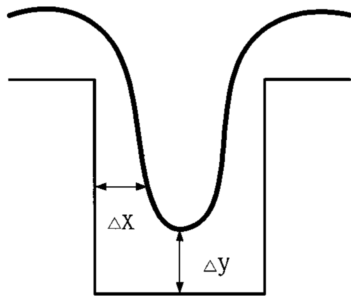

FIG. 1. $\Delta y/\Delta x$ ratio is taken as the characteristic for comparison of various additives and feature scales to measure the filling capability and judge the effectiveness of the plating process.

fining quantitative leveling in electrochemical deposition in the presence of an inhibiting additive have been reported.[16–21] As early as 1957, Watson and Edwards quantified the leveling ability of various additives based on polarization curves measured at different bulk leveling agent concentrations.[16,17] They derived leveling power of an additive for low amplitude features and high electrolyte conductivity, as shown in the following equation:

$$\text{leveling power} = \frac{C_a}{i}\frac{(\partial\eta/\partial C_a)_i}{(\partial\eta/\partial i)_{C_a}} = -\frac{C_a}{i}\left(\frac{\partial i}{\partial C_a}\right)_\eta, \tag{1}$$

where C_a is the concentration of additive in the bulk, i is the current density, and η is the overpotential. Further, Watson and Edwards were able to predict roughening or smoothing on copper deposition obtained from an electrolyte containing variable amounts of thiourea. Additionally, in Aroyo's papers,[19–21] the definition of the leveling power in an electrodeposited coating on the surface of a nonregular profile was defined as

$$\text{LP} = \frac{R_{a,\phi} - R_{a,c}}{d}, \tag{2}$$

where $R_{a,o}$ and $R_{a,c}$ are the roughness of the substrate and the plated surface, respectively, and d is the actually obtained coating thickness. The LP value that calculated from Eq. (2) is independent of the coating thickness. The degree of leveling with different coating thickness could be compared. As previously mentioned, the leveling effect on the gap-filling ability specified for damascene feature, and not large structures, was more important for very-large-scale-integrated application. Renewed definition of leveling power for additives in copper electroplating damascene structure is fundamental. We demonstrated a novel approach to monitor the filling profile changes during deposition as shown in Fig. 1. Through calculating the filling ratio $\Delta y/\Delta x$ of ''bottom-up'' to ''sidewall-shift'' from the cross section of a partially filled copper profile on the scanning electron microscopy (SEM) photo, the leveling power of different additives could be graded. This method could be employed to seek a proprietary additive enhancing superfilling performance. In a previous paper,[13,14] Kelly showed the leveling of 0.2 μm trenches by an acid copper electrolyte with polyethylene glycol (PEG), Cl^-, bis(3-sulfopropyl) disulfide, and Janus Green B. Furthermore, they concluded that the gap-filling capability strongly depended on the activation overpotential of additives in electrolytes. However, in our study, we achieved the superfilling performance in 0.15 μm vias with aspect ratio 6 and found that the activation overpotential is not the only factor influencing gap filling. In this work, we employed adsorption-diffusion model combined with cathode polarization and cyclic voltammetric stripping (CVS) measurements to explain the attributes of additives in superfilling phenomena. By means of those analyses, we have optimized the appropriate amount of additives and achieved the superfilling performance for 0.15 μm vias with aspect ratio 6 by an acid-copper electrolyte with leveler of 2-mercaptopyridine (2-MP) and several other typical additives. Due to the additive of 2-MP, chelate formed which enhanced adsorption ability on Cu^0 surface, and the concentration gradient between sidewall with bottom in the damascene became high enough to attend superfilling electroplating. Concluding unitary effects of additives, there are three indispensable requirements for achieving superfilling property; they are low surface tension of electrolyte, adequate activation overpotential, and sufficient inhibition gradient. If those conditions were present simultaneously during electroplating, then superfilling could be achieved.

TABLE I. Composition of electrolyte.

$CuSO_4\cdot 5H_2O$	30–90 g/l
H_2SO_4	100–400 g/l
Cl^-	50–100 ppm
PEG	20–2000 ppm
Leveling agents	10–2000 ppm

II. EXPERIMENT

Our copper electroplating experiments were carried out in hull cell. The hull cell is a trapezoidal box of nonconducting material with one side at a 37.5° angle.[22] The copper anode is laid against the right angle side and the wafer used as cathode is laid against the sloping side. Contact to the electrode was implemented outside of the electrolyte with an alligator clip. Agitation air was introduced into the solution from a compressor. In our experiment, the component of electrolyte was listed as Table I. The electrolyte was composed of copper sulfate (30–90 g/l), sulfuric acid (50–200 ml/l), chloride ion (50–100 ppm), wetting agent (80–200 ppm), and leveling agent (10–2000 ppm). All electroplating works were proceeded at room temperature. The direct current (dc) power supply and the sample size utilized in this work was Keithley model 2400 and 1×3 cm^2. The patterned wafers were composed of a 30-nm-thick ionic metal plasma (IMP)–TaN layer as the diffusion barrier and a 200-nm-thick IMP–Cu film as the seed layer. Results of both dc polarization and CVS measurements were used to analyze the attributes of additives in acid copper electrolytes. dc polarization analyses were performed on an EG&G Potentiostat/Galvanostat Model 273A. The counter electrode was platinum and the working electrode was copper foil with constant surface area 1 cm^2. The scanning rate was 2 mV/s

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and the condition time was 30 s. The initial potential was started at +250 mV and the final potential was completed at −1.5 V from open circuit potential (OCP). All potentials were reported relative to a double-junction saturated calomel electrode. A typical CVS technique was applied for evaluating levels of organic additives such as suppressing and brightening agents.[23,24] The CVS measurements were operated at room temperature by EG&G PARC Model 616 RDE. The indicator electrode was 1 cm^2 platinum electrode mounted in the rotating disc configuration and the Ag/AgCl electrode served as the reference electrode. Typically, operating parameters were 50 mV/s sweep rate and 0–3500 rpm on the working electrode. The scanning range was from −400 to 800 mV. Furthermore, the cross-section profiles of deposit films were examined by using field emission scanning electron microscope.

III. RESULTS AND DISCUSSION

Various organic additives have been typically adopted in acid copper electrolytes to control the current distribution on the macro- and microscale. The inhibitors in electroplating baths with fewer concentrations could yield smoother electrodeposits on the microscopic scale. The effect on leveling might be explained through the classical diffusion-adsorption theory.[3,16–21] According to the diffusion-adsorption mechanism, due to the diffusion distance to the peaks being shorter than the distance to the valleys, more inhibitors arrive on the peaks. Thus, the electrodeposition reaction is more inhibited on the peaks. Obviously, less metal is deposited and the profile becomes smoother. In our study, we developed a well-tuned and simple electroplating solution to fill defect-free structures and investigate the optimized recipe of each additive for gap-filling performance. All components in our electroplating solution were found from the patent literature or chemical handbooks. Typically, those additives were for electroplating unpatterned or larger-scale-feature substrates. Their mechanisms on a submicron scale within deep vias/trenches, for our purposes, should be quite different from dynamics on the blanket substrates. In order to distinguish the efficiency of different leveling agents more easily, we adopted the larger dimension patterns (about 1 μm, AR:1) and calculated the $\Delta y/\Delta x$ ratio of a partially filled trench profile to quantify the leveling power of additives. PEG as wetting agent and four different leveling agents were investigated. They were ammonium peroxydisulfate, thiourea, 4-mercaptopyridine (4-MP), and 2-MP. The concentration of all additives was adjusted to the operative concentration for promoting the filling ability. The average $\Delta y/\Delta x$ values of each leveling agent in electrolytes containing PEG were 2-MP (1.83)>thiourea (1.69)>4-MP (1.49)>ammonium peroxydisulfate (1.17) as shown in Fig. 2. The results showed that the filling power of 2-MP was higher than other additives. In Fig. 3(c), we observed that the superfilling effect was achieved in vias by using 2-MP as the leveler. This resulted because the copper deposition rate on the bottom was higher than the rate on top corners and sidewalls. As seen in Fig. 3(b), thiourea as the leveler produced the conformal phenomenon with a seam. This resulted because the copper deposition rate on top corners and sidewalls were almost equal to the rate on the bottom. In Fig. 3(a), the anticonformal deposited trench profile with a void was found for 4-MP as the leveling agent. In this case, the copper deposition rate on top corners and sidewalls were higher than the rate on the bottom. The consistent results also could be seen in Fig. 4 for 0.35 μm vias with AR 4. According to the earlier results, the $\Delta y/\Delta x$ ratio is actually proportional to the filling power of additives.

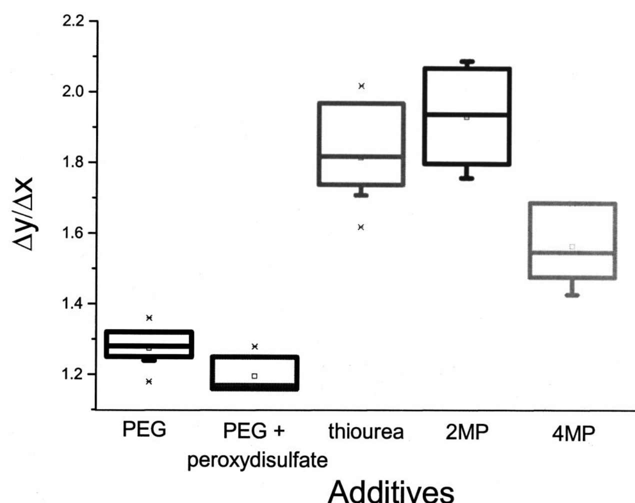

FIG. 2. $\Delta y/\Delta x$ ratio of different additives from the cross-section of a partially filled copper profile on the SEM photo.

Although leveling phenomena on anisotropic deposition rates has responded on the transient electroplating contour and resultant of gap-filling within the damascene structure,

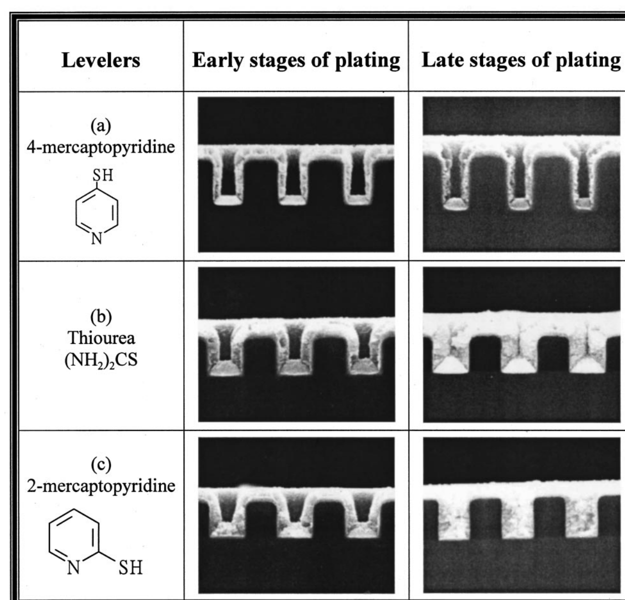

FIG. 3. 0.8 μm, AR: 1 cross-section profiles of wafers on SEM photo. (a) Anticonformal deposited trench profile within a void was found by using 4-MP as the leveling agent, (b) the conformal phenomenon with a seam in a via was produced by using thiourea as the leveler, and (c) the superfilling effect was achieved by using 2-MP as the leveler.

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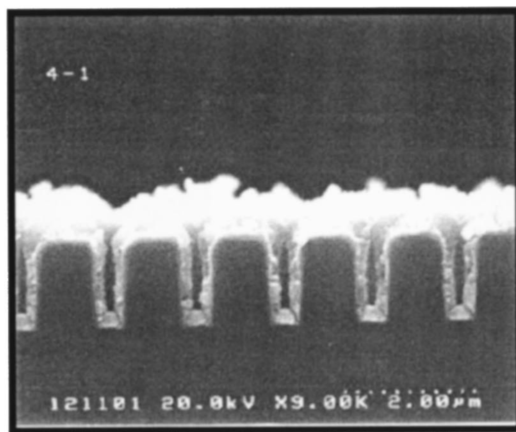
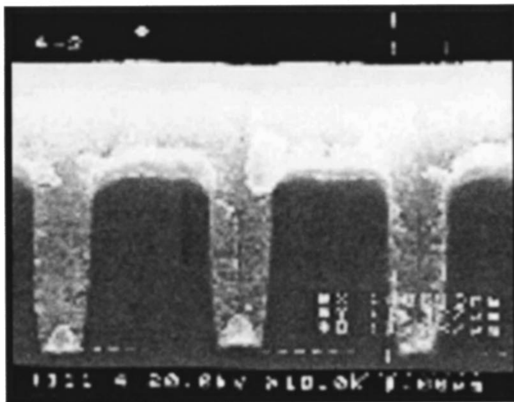
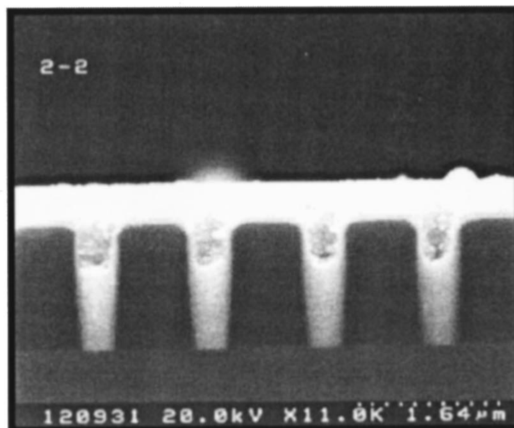


(a)



(b)



(c)

FIG. 4. 0.35 μm, AR:4 cross-section profiles of wafers on a SEM photo. (a) Anticonformal deposited trench profile within a void was found by using 4-MP as the leveling agent, (b) the conformal phenomenon with a seam in a via was produced by using thiourea as the leveler, and (c) the superfilling effect was achieved by using 2-MP as the leveler.

the mechanisms of chemical-electrical reaction, interlayer near seed layer, and matter adsorption-diffusion of additives in the sub-0.2 μm space, are necessary for purchasing categorical certification. In electrochemical deposition, the cathode polarization measurements provided comparison about inhibition ability between different additives. The higher overpotential is producing by additive, and the higher inhibition effect presents on copper deposition, as shown in Fig. 5. Also, it was found that the activation overpotentials of different additives at equal current density (0.001 A = 1 A/dm^2) in electrolytes containing PEG were thiourea (−0.253 V)>2-MP (−0.186 V)≈4-MP(−0.188 V)> peroxydisulfate (−0.132 V). This was because the added thiourea provided the highest suppression ability on copper deposition. In addition to dc polarization analysis, the CVS method would be employed to make the precise registration of electrochemical reaction changes on the cathode and anode, influenced by different organic additives, to be possible.[23,24] Our investigations with the aid of CVS show a single peak in the anodic area corresponding to copper dissolution that responded from the deposition of copper in the cathodic area. The equal current density during the cathodic and anodic processes, as well as the existence of a single peak in the cathodic area, suggest that the current density during the cathodic period was used singularly for the reduction of copper ions. In Fig. 6, the effects on the copper stripping area of various additives in solution containing PEG at constant electrode rotation rate 2500 rpm were no leveling agent (6258.72)>2-MP (1312.78)≈4-MP (1236.07) >thiourea (281.42). CVS measured on the condition of rotating electrode is advantageous for easily observing the diffusion-adsorption effect of additives on leveling. The lower areas around the peaks in the anodic region for different additives suggest that the effective concentration of copper ions involved electrodeposition in the cathodic cycle and was decreased by the leveler. The inhibition effects of leveling agents in copper electroplating baths with PEG were thiourea>2-MP≈4-MP>no leveling agent. The results of CVS analyses were consistent with the results of cathode polarization measurements. But CVS and polarization measurements showed some difference from filling ratio $\Delta y/\Delta x$.

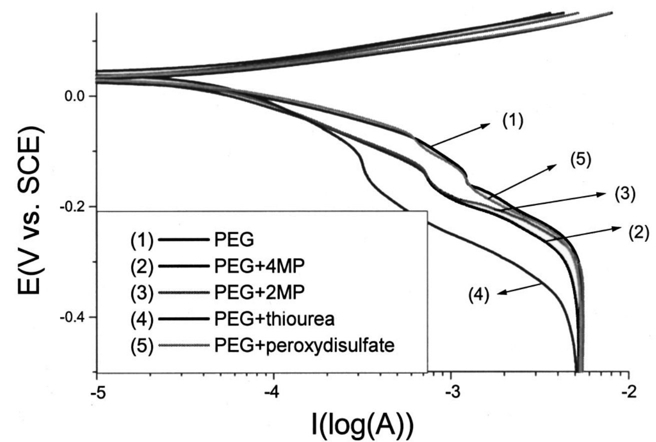


FIG. 5. dc polarization curves of various additives in the electrolyte. The scanning rate was 2 mV/s and the condition time was 30 s. The initial potential was started at +250 mV and the final potential was ended at −1.5 V from OCP.

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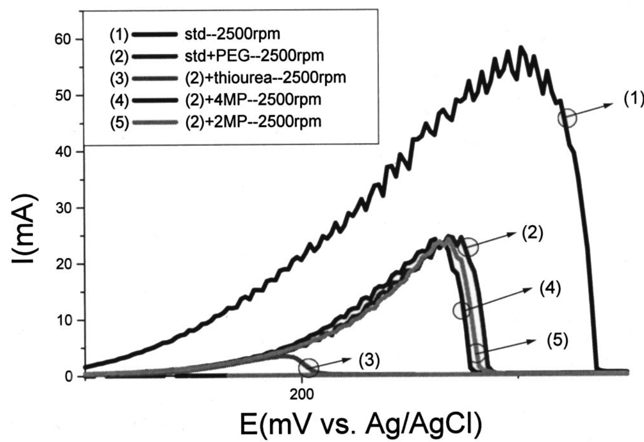

FIG. 6. Effects of various additives on copper stripping. Operating parameters were 50 mV/s sweep rate and 2500 rpm on the working electrode.

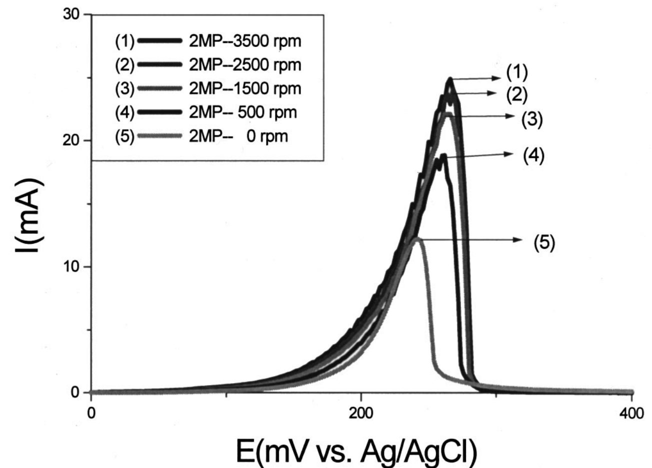

FIG. 8. Effects of 2-MP (12 ppm) on copper stripping with increasing electrode rotation rate (0–3500 rpm). Operating parameters were 50 mV/s sweep rate.

Further evidences for verifying those disagreements became essential.

As a theoretical consideration, transport of the metal ion (in this case, the cupric ion, Cu^{2+}) and of inhibiting additives is dominated by convection except within a concentration boundary layer that extends several tens of microns from the electrode surface.[3] From the adsorption-diffusion theory, the rate of electrodeposition is preferentially inhibited on those portions of the electrode surface that are more accessible to these agents by mass transport. Figure 7 shows the CVS results for the electrolyte-containing thiourea as a function of electrode rotation rate, and shows that the copper stripping area decreased (the inhibition effect increased) when the rotation rate increased from 0 to 3500 rpm. The explanation of this phenomenon is that the enhancement of additives mass transport is higher than the enhancement of cupric ion mass transport. This behavior of quicker transport of additive mass would result in near-equilibrium concentrations of leveler within the whole damascene structure. According to this kind of derivation, we suggested that the added thiourea was unable to produce superfilling because it did not provide selective inhibition gradient within the via, though its activation overpotential is high. However, if 2-MP provided adequate overpotential and selective inhibition gradient, then superfilling could be obtained. As shown in Figs. 8 and 9, the electrolyte containing 2-MP or 4-MP, when the electrode rotation rate increased from 0 to 3500 rpm; the inhibition effect decreased because the copper stripping area increased. For 2-MP or 4-MP, the enhancement of cupric ion mass transport is higher than the enhancement of additives mass transport because their molecular weights are higher than thiourea. This trend was contrary to the case of thiourea. Finally, for superfilling to occur there should be concentration gradient of the leveler within the via.

Although the diffusion model could explain the thiourea reaction on leveling effect within the vias/trenches, the different behaviors between 2-MP and 4-MP needed more proof. Diffusion speeds of those two levelers should be ap-

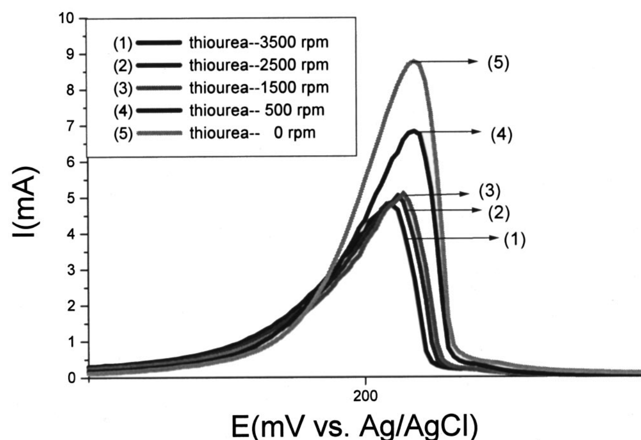

FIG. 7. Effects of thiourea (100 ppm) on copper stripping with increasing electrode rotation rate (0–3500 rpm). Operating parameters were 50 mV/s sweep rate.

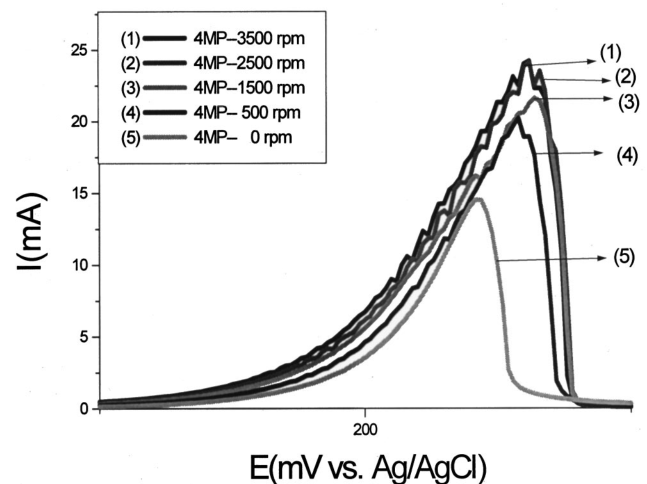

FIG. 9. Effects of 4-MP (12 ppm) on copper stripping with increasing electrode rotation rate (0–3500 rpm). Operating parameters were 50 mV/s sweep rate.

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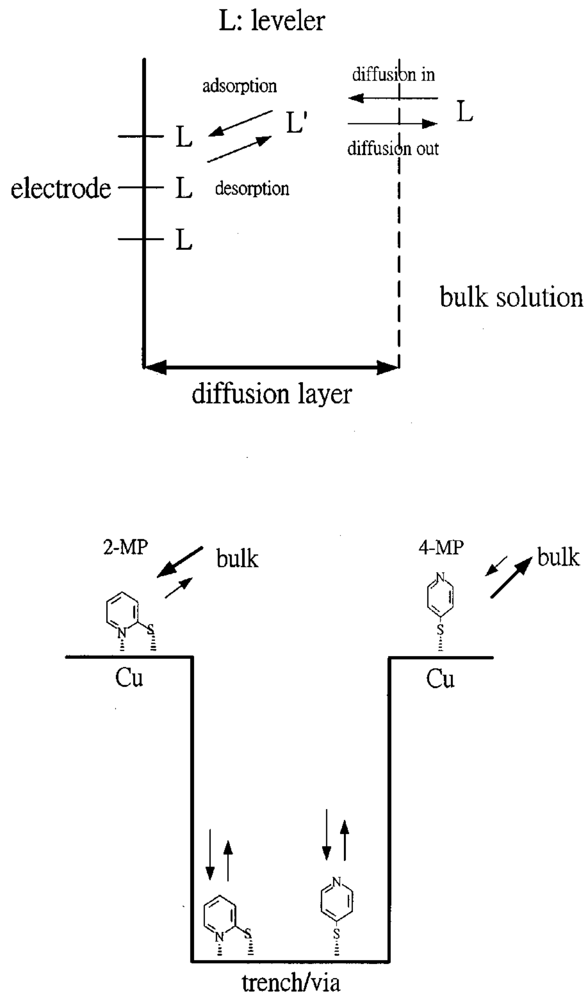

FIG. 10. Adsorption/desorption ability onto Cu^0 surface of 2-MP and 4-MP.

proximate because the molecular weights or chemical organizations were similar. It is presumed that the superfilling effect of 2-MP is due to its strong adsorption, both on the copper seed layer and on the growing clusters of the new phase. In addition to the adsorption effect of additives on seed layer to reduce the growth rate of copper crystals, the concentration gradient of additives in vias or trenches also must be present. Following, when the concentration of levelers on the sidewall is more than on the bottom in vias or trenches, the stronger inhibitor effect appears on the sidewall more than on the bottom. Then, the growth rate of copper crystals on the bottom is higher than on the sidewall and the superfilling is achieved. When comparing 4-MP with 2-MP with no significant difference between 2-MP and 4-MP on diffusion behavior, we suggested that the inhibition effect depends on the density of monolayer of organic species. Additionally, the adsorption/desorption ability onto Cu^0 surface may be the key for their different leveling power. In previous studies,[25] Alonso and co-workers concluded that in CVS measurements, if a platinum electrode is pretreated with 1×10^{-3} M solution of 4-MP in water, the copper deposition exhibits behavior similar to that of 2-MP. The overpotential for 4-MP is identical to 2-MP. Since 4-MP is incapable of

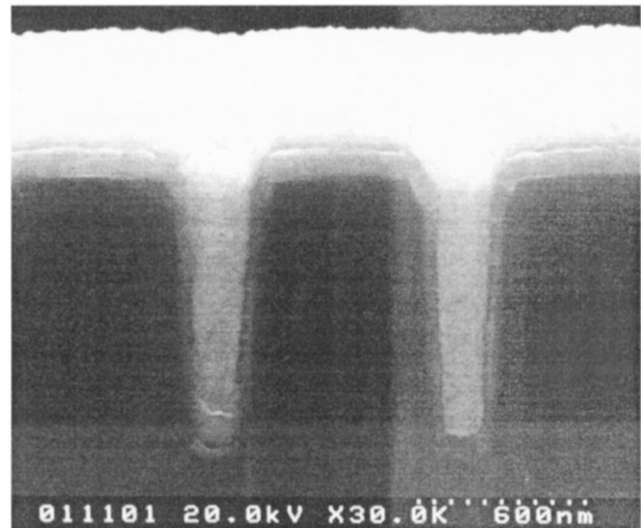

FIG. 11. Superfilling effect was achieved in 0.15 μm with aspect ratio 6 vias by using 2-MP as the leveling agent.

forming a chelate, whereas 2-MP is capable, the fact that the same overpotential is observed in both cases suggests that the interaction is dominated by the sulfur atom. In H_2SO_4 solution (at PH=0), the nitrogen atom in 4-MP is protonated. Since the nitrogen site is located para to the sulfur, such protonation would (by inductive effects) cause a weakening of the sulfur-platinum bond. However, in 2-MP chemical structure, the nitrogen site is located ortho to the sulfur and then forming a chelate on electrode surface. Because the higher adsorption ability of 2-MP, more amount of 2-MP consumed at the top, and less amount to the bottom. According to the earlier reasons, the concentration gradient of 2-MP between top and bottom is more than 4-MP because the adsorption ability of 2-MP is higher than 4-MP, as seen in Fig. 10. For the other additives, either the overpotential was not sufficient enough to suppress copper deposition at the top corners and sidewalls or the selective inhibition gradient could not be supplied, causing the anticonformal profile. Additionally, adding PEG as the wetting agent could prevent pits or pores produced in deposited copper by reducing surface tension and enhancing the filling capability. In our results, we achieved the super-filling performance of 0.15 μm vias with aspect ratio 6 by an acid-copper electrolyte with PEG, Cl^-, and 2-MP as shown in Fig. 11.

IV. CONCLUSIONS

The $\Delta y/\Delta x$ ratio of a partially filled trench profile is proportional to the filling power of additives. Moreover, the results are consistent with the features of filling profiles. The sufficient activation overpotential, lower surface tension of the electrolyte, and selective inhibition gradient within the feature are three indispensable conditions for procuring the superfilling property. Adding PEG in the electrolyte could reduce surface tension and enhance the filling capability. In electrolyte containing PEG, the superfilling phenomenon

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could be achieved in 0.15 μm vias with aspect ratio 6 by using 2MP as the leveling agent, because it provided not only adequate overpotential but also selective inhibition gradient. The adsorption-diffusion model combined with cathode polarization and CVS measurements was employed to explain the attribution of additives in superfilling phenomena. The superfilling dynamics was achieved because the behavior of additives provided selective inhibition gradient within the damascene feature.

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