Selection and Optimization of Corrosion Inhibitors for Improved Cu CMP and Post-Cu CMP Cleaning

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Corrosion inhibitors play a key role in obtaining global planarization and protecting against corrosion during copper CMP. However, these inhibitors leave organic residues and increase particle contamination after the CMP process, which can directly affect the device yield. Cu CMP is usually performed with a slurry containing silica particles and a BTA corrosion inhibitor. High levels of organic defects and particle contamination are produced due to the high concentration of BTA used to meet CMP requirements. In this work a suitable corrosion inhibitor, 5-methyl-benzotriazole (MBTA), is proposed and used at an optimized concentration to remove organic residues and particle contamination effectively during the post-CMP process. The optimum inhibitor concentration was estimated for BTA and MBTA. Based on etching and EIS studies, it was found that a lower concentration of MBTA (relative to BTA) is needed to provide the conditions required for CMP. The passivation layer formed by MBTA can be removed easily during the post-CMP process. Thus, our results indicate that it is possible to reduce the concentration of the inhibitor while maintaining the same corrosion inhibition efficiency, thereby reducing the particle and organic defect levels.

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Copper (Cu) is commonly used as an interconnecting material in microelectronic applications due to its outstanding properties, including its high electrical and thermal conductivities, high electromigration resistance, and low cost.1,2 Chemical mechanical planarization (CMP) is a key step in achieving both local and global planarization. Generally, during the metal CMP process, material is removed by a balance between dissolution, passivation, and abrasion. Hence, the slurry used for copper CMP consists of different components, such as an oxidizer, complexing agent, corrosion inhibitor, and abrasive particles.3,4 After the CMP process, several types of contaminations are produced, including particle contamination and organic residues.5 These contaminants should be removed by using a post-CMP process. Hence the slurry components should produce effective performance during the CMP process and can also be easily removed by the post-CMP process. One of the most critical components is the corrosion inhibitor.

Benzotriazole (BTA, C₆H₅N₃) is a prominent organic corrosion inhibitor used for protecting Cu surfaces from corrosive environments in the semiconductor industry. BTA is an aromatic heterocyclic compound that contains three nitrogen atoms on its surface, as shown in Figure 1a. The BTA acts as a weak acid by releasing a proton or as a base by accepting a proton to one of the nitrogen lone pairs of electrons. These chemicals can also be utilized to form bonds covalently or coordinately to the Cu surface.6 Subsequently, this chemisorbed insoluble stable complex on the Cu surface acts to prevent Cu corrosion.7,8 The composition and thickness of BTA films vary depending on the pH, electrode potential, solution composition, and amount of dissolved oxygen.9,10 Furthermore, the BTA layer can form as one of two film types; the first is a monolayer of adsorbed BTA molecules on the reduced Cu surface and the second is a much thicker layer of Cu(I)-BTA molecules that formed by the dissolution of Cu in the presence of an oxidizer or at anodic potentials.9 The formation of BTA molecules on the Cu surface occurs via the triazole rings, with one or two of the nitrogen atoms interacting with Cu, and the molecular plane is perpendicular to the surface. An electron will be donated by the Cu surface, resulting in the dissolution of Cu and the subsequent formation of a stable polymeric film of Cu(I)-BTA on the Cu surface. The formation mechanism of the Cu-BTA complex is proposed and described by the following reactions:11

\[
\text{Cu}^+ + \text{BTAH}^+ \rightarrow \text{Cu}^2+ + \text{BTA}^- \quad [1]
\]

**Figure 1.** Chemical structures of (a) 1H-benzotriazole and (b) 5-methyl benzotriazole.
rates were correlated with the EIS results. Based on the experimental results, optimized concentrations were estimated for these two inhibitors. It is observed that the concentration of the inhibitor influences the organic residual removal and also the particle removal during the post-CMP process.

Materials and Methods

For experiments, a 1-μm-thick electroplated 300-mm Cu wafer was used, and Cu coupons were sampled from the full wafer. All the Cu coupons were pre-treated with acetic acid (99.7%, Sigma Aldrich) without dilution to remove the native oxide from the Cu surface.\textsuperscript{21} Additionally, dipping (for static etch rate studies; 2 cm\textsuperscript{2} coupon wafers, 1 min) and polishing tests without abrasive particles (for dynamic etch rate studies; 4 cm\textsuperscript{2} coupon wafers, 1 min) were carried out for these experiments. Since dynamic etch rate and removal rates provides similar trends, only dynamic etch rates were measured to understand the inhibitor role clearly without interference of abrasive particles. The laboratory-prepared slurry consisted of 10 mM citric acid, 5 wt% hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}), and 10 mM inhibitors (BTA or MBTA) with and without abrasive particles. Polishing tests were performed using a polisher (Poli-500, GnP Technology, Korea) with the prepared Cu slurry without abrasives to measure dynamic etch rates and the following polishing conditions were maintained: Head / platen speed; 40 / 60 rpm; pressure: 3 psi; flow rate: 150 ml/min; time: 1 min. Then, the samples were rinsed with de-ionized water (DIW) and dried with N\textsubscript{2}. The thickness of each Cu coupon wafer was analyzed using a four-point probe (CMT-SR5000, AIT, Korea) before and after polishing to obtain the etch rate. After polishing test, Cu coupon wafers were cleaned by dipping in diluted tetramethylammonium hydroxide (TMAH) for 1 min and inhibitor removal efficiency was evaluated. A potentiostat (VersaSTAT4, Princeton Applied Research, USA) was used for electrochemical impedance analysis of post Cu surface after different chemical treatment. In order to evaluate the particle detectivity and particle removal efficiency, the prepared slurry with silica abrasive particles (100 nm, ACE NANOCHEN, Korea) was contaminated on Cu surface by using a spin dry method. The particle contamination level was analyzed using an FE-SEM (MIRA3, TESCAN, Czech Republic) and calculated with image processing software. The contaminated Cu samples were immersed in the commercial cleaning solution for 1 min and rinsed with DIW followed by N\textsubscript{2} dry. The particles on post Cu surface after cleaning was analyzed again to estimate the particle removal efficiency which is calculated by the following equation:

\[
\text{Particle Removal Efficiency (\%)} = \left( \frac{n_{\text{contam.}} - n_{\text{cleaning}}}{n_{\text{contam.}}} \right) \times 100
\]

where \(n_{\text{contam.}}\) and \(n_{\text{cleaning}}\) is particle number after contamination and cleaning respectively.

EIS is one of the most powerful techniques used to investigate the processes occurring at solid-liquid interfaces.\textsuperscript{16,22–25} In this study, ex situ EIS was performed to characterize the Cu-inhibitor complex formation on Cu surfaces and to analyze the removal of the Cu-inhibitor complex. All the experiments were performed using a potentiostat and a conventional three-electrode system. The working electrode was Cu treated with various chemicals, and the reference and counter electrodes were Ag/AgCl (saturated KCl) and a platinum mesh, respectively. 1 cm\textsuperscript{2} circular areas of post Cu surface after slurry/cleaning treatment was exposed to the supporting electrolyte solution (0.1 M NaClO\textsubscript{4}). An electrical equivalent circuit (EEC) model was employed to estimate the polarization resistance, and the polarization resistance values were used to quantify the Cu-inhibitor complex. The spectrum was obtained in the frequency range of 10 kHz–0.1 Hz at the open-circuit potential (OCP) with an amplitude of 10 mV rms. Commercial software (ZSimpWIN, EChem Software, USA) was used to retrieve the EEC parameters.

Figure 2. (a) Static and (b) dynamic etch rates of Cu in a solution mixture of citric acid (10 mM), H\textsubscript{2}O\textsubscript{2} (5 wt%), and an inhibitor (10 mM).

Results and Discussion

Figure 2a and 2b shows the static and dynamic etch rate results of copper in the presence of citric acid, which is a well-known complexing agent, at pH 3 under different conditions (e.g., with and without adding H\textsubscript{2}O\textsubscript{2} and an inhibitor (10 mM)). In both graphs, etch rate of copper is too low when H\textsubscript{2}O\textsubscript{2} was not added, irrespective of the inhibitor presence because citric acid forms a direct water-soluble compound by reacting with copper oxide (CuO) through the following reaction mechanism:\textsuperscript{26}

\[
\begin{align*}
2\text{H}_2\text{O}_2(\text{aq}) & \rightarrow 2\text{H}_2\text{O}(\text{l}) + \text{O}_2(\text{g}) \quad [3] \\
2\text{Cu}(s) + \text{O}_2(\text{g}) & \rightarrow 2\text{CuO}(s) \quad [4] \\
3\text{CuO}(s) + 2\text{Cu}_3\text{H}_5\text{O}_7(\text{aq}) & \rightarrow \text{Cu}_3(\text{Cu}_2\text{O}_7)\text{L}_2(\text{aq}) + 3\text{H}_2\text{O}_2(\text{aq}) \quad [5]
\end{align*}
\]

In case of dynamic etch rate by polishing, abrasion by pad asperities was not effective because the exposed copper surface during the polishing is pure copper in the absence of H\textsubscript{2}O\textsubscript{2} and it is harder than copper oxide. Therefore, the role of the inhibitor was not clearly observed without H\textsubscript{2}O\textsubscript{2}.

Active copper etching was observed when H\textsubscript{2}O\textsubscript{2} was added and it indicates that the formation of copper oxide (CuO) by H\textsubscript{2}O\textsubscript{2} and its dissolution by citric acid occurs simultaneously. Gorantla et al.,
proposed that citric acid would be active HX\(^2^-\) and H\(_2\)X\(^-\) (where X = -OOCCH2HOOC\(^-\)-CH2COO\(^-\)) at lower pH range and they would adsorb on CuO and react faradaically to form soluble complexes as shown in below Equations 6 and 7.\(^{25}\) Lee et al., also observed that there would be great increase of removal rate in the presence of H\(_2\)O\(_2\) compared to without its presence when Cu was polished with citric acid at pH 4.\(^{26}\)

\[
2\text{CuO} + 4\text{H}^+ + 2\text{HX}^2^- = 2(\text{HXCu}^+) + 2\text{H}_2\text{O} \quad [6]
\]

\[
2\text{CuO} + 4\text{H}^+ + 2\text{H}_2\text{X}^- = 2(\text{H}_2\text{XCu}^+) + 2\text{H}_2\text{O} \quad [7]
\]

When the inhibitor was added, a clear reduction in the etch rate of copper was observed. It can be seen that MBTA strongly inhibits the copper etching rate compared to BTA when the same concentration was used. A similar trend was also observed for the dynamic etch rate result, as shown in Figure 2b. The dynamic etch rate is much higher than the static etch rate due to the favorable conditions for higher etching caused by the increased diffusion rate of the reacting ions.\(^{28}\) With the addition of an inhibitor, the dynamic etch rate was not decreased significantly due to the continuous removal of adsorbed inhibitor molecules resulting from the abrasive action between the polishing pad and copper surface during the polishing. This shows that inhibition at dynamic condition (polishing) is less effective than static condition (dipping).

The characteristics of nanometer-thin Cu complex layers are very difficult to analyze, but they influence the removal rate and amounts of defects on polished Cu surfaces. EIS data were analyzed using EEC modeling to qualitatively and quantitatively compare the results obtained under different chemical conditions. Figure 3 shows the schematic of the EEC used for modeling. In the circuit model, \(R_{\text{sol}}\) represents the solution resistance; \(\text{CPE}_c\) and \(R_c\) are the capacitance and resistance associated with the passive film, respectively; and \(\text{CPE}_{\text{dl}}\) and \(R_{\text{ct}}\) represent the capacitance and resistance associated with the electrical double layer and the charge transfer resistance at the surface-electrolyte solution interface, respectively. Since the presence of a Cu-inhibitor complex film affects both parameters, the polarization resistance (\(R_p\)) was calculated by summing \(R_c\) and \(R_{\text{ct}}\).\(^{30,31}\)

\[
R_p = R_c + R_{\text{ct}}
\]

Figure 4 shows the polarization resistances of post copper surface after dipping and polishing using the abrasive-free slurry with and without inhibitors. For both the static and dynamic states, the polarization resistance increases with the formation of the Cu-inhibitor complex when inhibitors were added. In addition, the polarization resistance also increases when H\(_2\)O\(_2\) was present without an inhibitor; this is due to the generation of a Cu oxide passivation layer. On the other hand, the polarization resistance is observed to decrease when inhibitors were used with H\(_2\)O\(_2\); this is the case because H\(_2\)O\(_2\) and the inhibitor react with Cu together. Thus, the polarization resistance value in this case is between the values of Cu oxide and the Cu-inhibitor complex, as shown in Fig. 4. The Cu-MBTA complex exhibits a higher polarization resistance value than the Cu-BTA complex. Compared to the dipping conditions, the polarization resistances for the CMP process are lower, which can be attributed to the dynamic abrasion action on the Cu-inhibitor complex layer. This result indicates that carrying out CMP with abrasives may be highly unfavorable for corrosion protection due to the more aggressive removal of the Cu-inhibitor complex layers. Both the etch rate and EIS results show that MBTA has a higher inhibition efficiency than BTA. The introduction of a nonpolar methyl group increases the film hydrophobicity, thereby improving Cu corrosion prevention.\(^{26}\) Also, the solubility of BTA is higher than MBTA; thus, MBTA is more strongly adsorbed on the Cu surface.\(^{32}\) Furthermore, the methyl group has an electron-releasing characteristic; this phenomenon is referred to as the “positive inductive effect” (+I effect). However, alkyl groups also tend to release electrons. If a molecule has a negative charge (e.g., BTA\(^-\)) and is attached to a +I group (e.g., a methyl group), its charge becomes amplified and the molecule readily donates electrons to the metal to form a metal-inhibitor complex. In other words, MBTA is more active than BTA because of the stronger +I effect induced by the methyl functional group.

Figures 5a and 5b show the dynamic etch rate and the polarization resistance of Cu as a function of inhibitor concentration. The dynamic etch rate was observed to decrease as a function of BTA/MBTA concentration and BTA showed lower inhibition performance at all concentration than MBTA. It indicates that lower concentration of MBTA can be used than BTA to achieve the same inhibition performance, e.g. 3 mM MBTA showed the similar dynamic etch rate as 10 mM BTA. In the case of EIS, the polarization resistance was found to be directly related to the inhibitor concentration, and 3 mM MBTA showed the similar polarization resistance as 10 mM BTA.
Therefore, these results suggest that 3 mM MBTA produces an inhibition efficiency that is equivalent to 10 mM BTA.

The polarization resistance of the Cu-inhibitor complex after the cleaning process is shown in Fig. 6. A diluted TMAH solution was employed; TMAH is widely used as a base cleaning solution for post-Cu CMP cleaning processes. The cleaning efficiency of the Cu-inhibitor complex was evaluated by EIS. The polarization resistance was increased after polishing with abrasive-free slurry and 10 mM BTA showed similar value with 3 mM MBTA. After cleaning with TMAH solution, the polarization resistance was decreased with increasing TMAH concentration and it became similar with cleaned Cu with 1% TMAH. It indicates that TMAH is effective to remove Cu-inhibitor complex from Cu surface. When BTA and MBTA are compared, it was observed that the Cu-inhibitor complex with 3 mM MBTA is more easily removed than when 10 mM BTA was used below a TMAH concentration of 0.5 wt%. This result can be directly correlated to the amount of the inhibitor adsorbed onto the Cu surface. MBTA molecule is larger than BTA due to the methyl group and it may cause steric hindrance effect. Antonijevic and Petrovic also concluded that as molecule is larger, concentration of inhibitor required to obtain the same degree of surface coverage becomes less due to the efficient surface blocking. Therefore, it can be concluded that adsorbed number of MBTA molecules is lower than BTA even with similar surface coverage and it makes the difference in inhibitor removal efficiency.

To evaluate the effect of inhibitor on particle defect, the prepared slurry with silica abrasive particles (100 nm) were contaminated on Cu surface by using spin dry method. The numbers of particles on the Cu surface before and after cleaning were observed using FE-SEM and counted with imaging software. The results of the particle analysis are shown in Fig. 7. These results indicate that the contaminated particles were removed after cleaning; however, the contamination...
level and cleaning efficiencies varied with the inhibitor conditions and the quantitative analysis is shown in Figure 8. After contamination, slurry without inhibitor showed the lowest particle contamination level. When slurry contains inhibitors, particle contamination was increased due to the hydrophobic layer formation by the inhibitor on Cu surface.35,36 10 mM BTA showed higher contamination than BTA as MBTA contains extra hydrophobic methyl group to increase the hydrophobicity of Cu surface. As we discussed above, it is considered that adsorbed amount of 3 mM MBTA is lower than 10 mM MBTA and it may cause lower hydrophobic surface which can be the reason for lower particle contamination. The adsorbed inhibitor is removed by cleaning solution during the cleaning process and it can be expected that 3 mM MBTA is removed more easily than other cases as discussed earlier. When adsorbed inhibitor is removed, the wet cleaning mechanism becomes effective. The higher PRE with 3 mM MBTA and without inhibitor can be attributed to this reason. Therefore, it can be concluded that smaller amounts of inhibitor with a higher inhibition efficiency is more desirable to reduce or eliminate any inhibitor-related organic/particle contamination for post-Cu CMP cleaning.

Conclusions

In this study, it is found that MBTA shows a higher corrosion inhibition efficiency than BTA at the same concentration. Specifically, the results show that using 3 mM MBTA exhibits an equivalent corrosion inhibition efficiency to 10 mM BTA. The presence of a methyl group in MBTA induces the positive inductive effect, which enhances the reactivity of the lone pair electrons of nitrogen in the triazole group. Therefore, lower quantities of MBTA show better performance than 10 mM BTA. For the cleaning process, it is also easier to remove 3 mM MBTA than 10 mM BTA in TMAH-based cleaning solutions. Similarly, a solution with 3 mM MBTA results in a cleaner surface with much less particle contamination. The etching and lift-off processes are also expected to be more favorable at lower inhibitor concentrations. The results provide evidence that a strong inhibitor at the proper concentration can enhance the particle- and inhibitor-related organic contaminant removal during post-CMP cleaning. Therefore, this study proposes an effective way to optimize the corrosion inhibitor concentration in a slurry to yield a better cleaning efficiency. The particle contamination and removal are found to be highly affected by the Cu-inhibitor complex layer. The Cu-inhibitor complex causes more particle contamination, and these complexes make the particles difficult to remove.

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Figure 8. The number of particles under various inhibitor conditions on Cu surfaces before and after cleaning. Particle removal efficiency (%) is shown together.