Chemical Mechanical Planarization of copper in alkaline slurry with Uric acid as inhibitor

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Abstract

Chemical mechanical planarization of copper disks in hydrogen peroxide and L-arginine based alkaline slurry was investigated. Various commonly proposed inhibitors were screened by static etch rate experiments and only BTA and uric acid were found to be effective in the alkaline pH range, while potassium sorbate was moderately effective. The combination of arginine, hydrogen peroxide and uric acid leads to a more robust polish rate than can be achieved with glycine, hydrogen peroxide and BTA based slurries. The dissolution in presence of uric acid was further studied by potentiodynamic and cyclic polarization experiments which indicate formation of a passivation layer on the copper surface. Silicon dioxide wafers were polished to determine the Cu/silicon dioxide polish rate selectivity in alkaline pH range and a high selectivity (> 100) was observed. These results indicate that it is possible to conduct a robust copper CMP with alkaline slurries while maintaining an excellent Cu/silicon dioxide polish rate selectivity.

Keywords: Copper; CMP; Uric acid; Inhibitor; Alkaline slurry
1. Introduction

Copper integration when combined with low-k materials, promises much faster devices with less complexity and lower power consumption [1,2]. The dual damascene technique is used for the metallization of copper in multilevel integrated chip fabrication. CMP is the key processing step in the Damascene technique [3]. For efficient planarization of Cu surface, a balance between dissolution rates of the surface using an oxidizing agent, formation of thin passive layer with help of an inhibitor and removal of excess copper with help of complexing agents is needed [4].

Copper CMP can be performed in acidic or neutral or basic medium [5-7]. A high selectivity of Cu/SiO$_2$ polish rate is desirable to minimize erosion losses. In acidic slurry a high Cu/SiO$_2$ polish rate can be easily achieved. Hence, most of the copper CMP studies have focused on acidic slurries [6-19]. However, a disadvantage of the acidic slurries is the possible corrosion of the polishing equipment by the slurry [5]. In the alkaline region, ammonia and ammonium salt based slurries have been investigated but the reported Cu/SiO$_2$ polish rate selectivity is an order of magnitude lower than that achievable in acidic slurries [5].

The combination of hydrogen peroxide as an oxidizer and glycine as complexing agent in Cu CMP slurries has been studied extensively [7-11]. Hydrogen peroxide is a powerful oxidizer and can be effectively used in various solutions in a wide range of pH values. H$_2$O$_2$ is also a very clean oxidizer, which does not require post-CMP cleaning, because its reactions are accompanied only with oxygen and hydrogen evolution, hydroxide-ions and water [3]. CMP slurries require a complexing agent to dissolve the abraded species and a corrosion inhibitor to form a passivative layer to protect the lower regions [6]. Glycine and H$_2$O$_2$ based slurries were also investigated in
alkaline regime where the pH was maintained by buffers [7]. However, the polish rate of silicon dioxide with that slurry is not known.

In peroxide based slurries, apart from glycine few other complexing agents have also been proposed. They include acetic acid [10], ethylene diamine [10], glycolic acid [13], phthalic acid salt [14], citric acid [15], and oxalic acid [16]. L-arginine (Fig 1.a), like glycine, is an amino acid, but arginine based slurries are inherently alkaline, while all the other combinations listed above with peroxide are inherently acidic. Arginine also has the interesting property of inhibiting silicon dioxide polish rate, even in highly alkaline slurries [17].

Benzotriazole (BTA) is a very effective inhibitor of copper corrosion in acidic, neutral and basic media [5, 6]. However, it is reported to be slow in its inhibiting effects [3,18] and toxic [19], besides posing other post-CMP challenges [20-24]. Other additives proposed as copper corrosion inhibitor include citric acid [25], hydrazine [26], amino tetrazole [27], potassium sorbate [28], and imidazole [29].

Uric acid (Fig 1.b), a heterocyclic nitrogen compound similar to BTA, is known to protect copper from corrosion in amino alcohol during the removal of photo resist [19] and is a non-carcinogenic and an environmentally friendly chemical. Uric acid is only slightly soluble in water but it dissolves more in alkaline solution. Thus it is a suitable candidate for inhibiting copper corrosion in alkaline slurries. In this report, the copper removal with alkaline slurry containing hydrogen peroxide and arginine was investigated by potentiodynamic and cyclic polarization experiments and CMP. The suitability of uric acid as inhibitor for this slurry system was also studied using electrochemical methods, static etch rate and CMP at various inhibitor concentrations. In order to ascertain the inhibition of SiO\textsubscript{2} polish rate by arginine in presence of peroxide and uric acid, oxide polish rate was measured.
2. Experimental

2.1. Polishing Experiments

The polishing experiments were carried out using a bench top Labpol-5/Laboforce-3 polisher (Struers), with SUBA IV pads (Rodel Inc). Electrochemical grade copper disks of 25.4 mm diameter were used for all the polishing and static etch rate experiments. The slurry contained 1 (vol./vol)% fumed silica abrasives (Cab-o-sil, M-5 with a surface area of 200 m$^2$/gm, from Cabot-Sanmar, India), 1 wt% L-arginine, various concentrations of hydrogen peroxide (0 to 5 vol %) and various concentrations of uric acid (0 to 75 mM). The pH of the slurry varied between 10.3 (for slurry with 1 wt% arginine) and 7.2 (for slurry with 1 wt% arginine, 5 (vol/vol)% H$_2$O$_2$ and 0.075 M uric acid). The polishing runs were carried out for 3 minutes and the slurry flow rate was maintained at 100 ml/min. The applied down force was 30 N (2.15 psi). The sample was rotated at 250 rpm while the turn table with the pad was rotated at 150 rpm. The relatively higher speeds are employed to obtain better accuracy for the removal rates. Static etch rate experiments were carried out for a period of 30 min. BTA, citric acid hydrazine and potassium sorbate were also studied as possible inhibitors with the static etch rate experiments. All the chemicals were of AR grade (Merck India, India) and MilliQ(Millipore) water was used for the experiments.

The polish rates and the etch rates of copper were calculated by measuring the weight loss of the disks using Citizen Balance (0.1 mg accuracy). Silicon wafers with thermal oxide (SWI, Taiwan) were cut into square pieces of approximately 5 cm$^2$ area and polished under the same conditions as copper samples. The polished oxide samples were cleaned by ultrasonication for 60 seconds and dried in Argon. The thicknesses before and after polish were measured using
Filmetrics UV-20 thin film thickness measuring system. At least three identical runs were conducted and the average removal rates and etch rates are reported in nm/min.

2.2. Electrochemical Studies

Potential Dynamic polarization tests were carried out by using Electrochemical Instrument PARSTAT 2263 (Princeton Applied Research). The Cu Electrode (working electrode) used in these experiments of 11 mm diameter (99.9999% purity, Aldrich) was attached to a Teflon rod. Ag/AgCl (3.5M KCl) and a platinum electrode (CH Instruments) were used as the reference and counter electrodes respectively. The cell was placed in a Faraday cage to minimize interference from electrical noise. Prior to each experiment, the working electrode was mechanically polished with successively finer grades of alumina (Buehler Inc) down to 0.05 micron in diameter, followed by rinsing and sonication in ultra pure water to remove any attached alumina particles.

Potentiodynamic polarization curves were obtained by scanning the potential at a rate of 1 mV/sec from -250 mV to 500 mV versus open circuit potential (OCP). The corrosion potential ($E_{corr}$) and the corrosion current density values ($I_{corr}$) were calculated by linear extrapolation of the cathodic and anodic branches of the Tafel plots, using the Powercorr software.

3. Results and Discussion

Fig. 2 shows the polish rate of copper as a function of hydrogen peroxide concentration in slurry containing 1 wt% silica and 1 wt% of arginine with and without uric acid. The addition of H$_2$O$_2$ to 1 wt% arginine slurry increases the polish rate up to 0.1 vol% of H$_2$O$_2$. Further increase in H$_2$O$_2$ concentration reduced the polish rate by about 42% (from 390 nm/min to 225 nm/min). For glycine and H$_2$O$_2$ based slurries, a similar trend has been reported [7,8]. When copper samples were polished with 0.01 M glycine and various H$_2$O$_2$ concentrations at a pH of 4 and 9,
the peak polish rate was reported to occur at 0.25 wt% H\textsubscript{2}O\textsubscript{2} concentration [7]. The polish rate at higher H\textsubscript{2}O\textsubscript{2} concentration decreases by more than 50% (from 140 nm/min at 0.25 wt% H\textsubscript{2}O\textsubscript{2} to about 60 nm/min at 5 wt% H\textsubscript{2}O\textsubscript{2}, at a pH of 4). Seal et.al.[8] reported that with 0.1 M glycine based slurry, the polish rate of copper vs H\textsubscript{2}O\textsubscript{2} concentration shows a peak at approximately 2.5 vol % of H\textsubscript{2}O\textsubscript{2}. Again a further increase in H\textsubscript{2}O\textsubscript{2} concentration decreases the polish rate by more than 50% (from 360 nm/min at 2.5 vol % of H\textsubscript{2}O\textsubscript{2} to 120 nm/min at 7.5 vol % of H\textsubscript{2}O\textsubscript{2}).

Amino acids along with cupric ions enhance the dissociation of H\textsubscript{2}O\textsubscript{2} and generate OH radicals [9]. Hydroxyl radical is a much stronger oxidant than H\textsubscript{2}O\textsubscript{2} and hence would enhance the oxidation of copper. The oxide is dissolved by the complexing action of the amino acid, which in turn will catalyze the dissociation of H\textsubscript{2}O\textsubscript{2}. This is similar to the reported behavior of glycine and hydrogen peroxide based slurries [10]. The peak in the polish rate vs H\textsubscript{2}O\textsubscript{2} concentration is normally explained as follows: [7,8,14,30] At low concentrations, H\textsubscript{2}O\textsubscript{2} increases the oxidation rate and the copper etch rate also increases. Higher H\textsubscript{2}O\textsubscript{2} concentration leads to passivation by the formation of a thick oxide layer on top of the metal layer and the diffusion of ions is restricted leading to reduced removal rate. The exact location of the peak will depend on the balance between the growth rate of the oxide film and the removal by mechanical and chemical actions. A thicker oxide film at higher concentration of H\textsubscript{2}O\textsubscript{2}, if present, may reduce the corrosion rate slightly, but does not offer passivation [3]. Thus the film formed on the surface in slurries without uric acid does not offer good protection from corrosion. At neutral or alkaline pH range, copper will tend to form an Cu\textsubscript{2}O, CuO and Cu(OH)\textsubscript{2} [3,31] while at very low pH values, stable oxide does not form on copper [7]. The oxide and hydroxide layers however do not form a protective layer, but are rather in the form of coarse insoluble precipitates[3]. Besides, even at alkaline pH regime, the film may not exist in presence of effective complexing agents
such as ammonia \[3, 31\] or glycine or EDTA\[10\]. In ammonium hydroxide slurries, copper actively dissolves and stable passive oxide is not found on the copper surface and similar hypothesis was proposed for glycine and peroxide slurry in alkaline pH \[10\]. We propose that arginine may also complex the copper similarly and thus stable oxide would not be present. Even if a film forms, the extent of growth of the film will be controlled by the kinetics of growth rate and the effectiveness of passivation will depend on the porosity of the film. The removal of the film by mechanical abrasion is controlled by various parameters including the hardness of the film formed while the removal by chemical etching is controlled by parameters including the porosity of the film and the film thickness.

It was reported that arginine increases the generation of hydroxyl radicals only slightly while glycine increases the generation by an order of magnitude \[9\]. However, the polish rates reported here are comparable to those reported in literature for glycine and H\(_2\)O\(_2\) based slurries \[7, 8\]. The hydroxyl radical studies \[9\] were conducted at a pH of 8.4, while the polish rate experiments for glycine and H\(_2\)O\(_2\) based slurries are typically conducted in the acidic pH range. Besides, the type and concentration of abrasives and polishing conditions such as the pad type, polish pressure and velocity also play a key role in determining the overall polish rate and that may explain the good polish rate obtained with arginine and peroxide based slurries.

One of the requirements of copper CMP slurry is that it should passivate the material being planarized so that the metal lines which are not in direct contact with the polishing pad region remain passivated \[3\]. From Fig.2, it is clear that the slurry without uric acid has a high polish rate and there is a need to add an inhibitor to induce passivation. The addition of 0.05 M uric acid reduces the polish rate of copper and the overall polish rate trend is similar with and without uric acid. However, the peak polish rate occurs at 0.5 vol % of hydrogen peroxide
concentration. The formation of an inhibiting film may modify the chemical as well as mechanical removal rate of copper and hence the location of peak polish rate would change with the addition of uric acid. Uric acid is effective inhibitor throughout the peroxide concentration range studied here. In presence of uric acid, the variation in copper polish rate with \( \text{H}_2\text{O}_2 \) concentration is also very minimal. This proves to be an advantage since it would lead to more robust process conditions. Hydrogen peroxide is not a very stable compound and it decomposes in the presence of light and heat. Thus the concentration of \( \text{H}_2\text{O}_2 \) in slurry may decrease if it is stored for a long time. A slurry combination which provides a high and more or less constant polish rate over a wide range of \( \text{H}_2\text{O}_2 \) concentration will lead to a robust process. Thus, from the process control point of view, arginine and uric acid based slurries would be preferable to glycine based slurries.

Electrochemical experiments provide valuable insight into the dissolution and passivation characteristics of metals [3]. Polarization curves for the copper electrode in 1 wt% Arginine + 5 vol% \( \text{H}_2\text{O}_2 \) mixture and various concentrations of uric acid are shown in Fig. 3. The corrosion potential (\( E_{\text{corr}} \)) where the passive to active transition occurs, is in the range of 0.15 V to 0.25 V vs Ag/AgCl. Again, it is seen that there is no sign of passivation in the curve corresponding to the solution without uric acid. Thus, it is clear that copper oxide layer in these slurries, if present, does not offer good corrosion protection. This finding is similar to the reported behavior of copper in other hydrogen peroxide based slurries [3]. The addition of Uric acid increases the value of \( E_{\text{corr}} \), possibly due to the presence of inhibiting film on the copper surface which may offer higher resistance to the movement of ions leading to an increase in \( E_{\text{corr}} \). Similar increases in \( E_{\text{corr}} \) have been reported for copper electrode with the addition of BTA to ammonia slurries [5], citric acid to nitric acid slurries [25] and \( \text{H}_2\text{O}_2 \) to blank slurries [12] and glycine slurries [8].
Organic compounds with Nitrogen can form coordinate bonds with copper and protect it from corrosion [19]. In this system, an inhibiting film may be formed by the preferential adsorption of the uric acid on the copper surface. The corrosion currents ($I_{\text{corr}}$) were estimated by extrapolating the cathodic and anodic branches of the polarization curves. $I_{\text{corr}}$ also decreases with uric acid concentration as shown in Fig. 4. In general, a reduction in $I_{\text{corr}}$ may be due to suppression of anodic or cathodic reaction. For concentrations higher than 0.025 M of uric acid, the anodic branch of polarization curve shows a clear decrease in current indicating the formation of passivating film. This decrease occurred at a potential of about 0.5 V vs Ag/AgCl. Further increase in potential resulted in decreased current density probably due to increased adsorption of the uric acid. For lower concentrations of uric acid, the anodic branch does not show any kink which would indicate passivation and only a reduction in the current is observed. This may indicate that the film formed at lower uric acid concentrations offers some resistance to corrosion but does not completely passivate the surface.

For effective planarization, it is desirable to have high removal rate of protruding region, typically by a combination of chemical and mechanical action and low or no removal rate of lower region by forming a passivation layer. Static rate is a measure of chemical removal in CMP. Ideally, the dissolution should be small and the combined chemical and mechanical removal should be large. The effect of Uric acid on the static etch rate of copper in $\text{H}_2\text{O}_2$ and arginine system is shown in Fig 4. The $I_{\text{corr}}$ is also shown in the same plot for comparison. Both $I_{\text{corr}}$ and the static etch rate decrease with the addition of uric acid. Here it is observed that even with the addition of 0.01 M uric acid, the etch rate and the $I_{\text{corr}}$ show significant reduction. Hence it can be concluded that uric acid is effective in reducing copper corrosion rate during CMP. It is also noted that the reduction tends to saturate at a concentration of 0.05 M. If the static etch rates
are high, then the planarization efficiency would be low. The actual etch rate during polishing may be much higher than the ones measured at room temperature since the local temperature can raise by about $10^\circ$ C during CMP[32-34]. Thus it is necessary to maintain a very low etch rate for effective planarization. Hence even corrosion inhibition provided by the uric acid in this slurry may not be sufficient for direct industrial application and the slurry composition and operating conditions will have to be optimized further.

Cyclic potentiodynamic curves performed at various reverse scan potentials can be used to determine the stability of the passivation in a system [3]. Presence of hysteresis with increased anodic current would indicate breakdown of the protective layer, as was found for BTA[35]. Fig 5 shows the cyclic potentiodynamic curves of copper in 5 vol % hydrogen peroxide, 1 wt% arginine and 0.05 M uric acid. The scans were reversed at various potentials and it was found that the anodic current decreased during the reverse scan, indicating enhanced passivity. The $E_{corr}$ obtained during the reverse scan was also found to be slightly higher than the $E_{corr}$ during the forward scan. This behavior is similar to that of copper in solutions containing potassium carbonate which provides stable passivity [3].

Fig 6 shows the effect of uric acid on the polish rate of copper. Clearly the removal rate decreases with increase in uric acid concentration. The inhibiting effect also tends to saturate at a concentration of 0.05 M uric acid as was seen in the static etch rate experiments. An ideal inhibitor will reduce the etch rate without affecting the polish rate. It is seen from Fig.2 that uric acid does reduce the polish rate. However, the ratio of polish rate to etch rate increases with the addition of uric acid. BTA, the commonly studied inhibitor also shows similar behavior. It was reported that polish rate decreases by more than 50% with the addition of BTA to glycine and $\text{H}_2\text{O}_2$ slurries [9].
Other potential candidates such as BTA, hydrazine, citric acid and potassium sorbate were evaluated for their corrosion inhibiting effect in the \( \text{H}_2\text{O}_2 \) and arginine slurry and the static etch rates of copper in various slurries are provided in Table 1. BTA is very effective and potassium sorbate is moderately effective in reducing the corrosion, while hydrazine and citric acid actually lead to an increase in the etch rate. The common mechanism of corrosion inhibition is by adsorption of the chemical onto the metal surface. Adsorption behavior of the inhibitors will depend on the pH value and presence of other chemicals in the system. Hydrogen peroxide or arginine may modify the interaction between the potential inhibitors and the copper surface. For example, citric is reported to be an inhibitor of copper corrosion in nitric acid [25], but it increases the polish rate of copper in \( \text{H}_2\text{O}_2 \) based slurries [15]. Thus it is not surprising that additives like hydrazine or citric acid which inhibit corrosion in some slurry actually enhance the corrosion in a different environment. Even though slurries with BTA show very low static etch rates for copper, they provide corrosion protection only in a narrow potential range above corrosion potential and the protective film disintegrates above a threshold potential [3]. The increase in anodic current and the decrease in the \( E_{\text{corr}} \), during the reverse scan of the cyclic polarization experiments confirm this problem [35]. Besides, BTA creates post-CMP challenges such as leaving hydrophobic copper surface [19], increasing particle adhesion [20] and increased etch rate of copper during cleaning [21]. Also, BTA does not eliminate micro scratches [22] or pits [23,24]. BTA can also cause problem in the treatment of used slurry since it is recalcitrant to biodegradation [19, 36].

One of the major concerns with alkaline slurries for Cu CMP is the possible high polish rate of silicon dioxide at high pH, leading to the problem of oxide erosion. However, arginine has been investigated as an additive for CMP slurry for Shallow Trench Isolation (STI) and it
was reported that it suppresses the oxide polishing [17]. For 1% arginine and 5% H$_2$O$_2$ slurry, the polish rate of silicon dioxide at the same polishing conditions as that of copper was found to be 1 nm/min with out uric acid and 2 nm/min with 0.05 M uric acid. In some cases, the difference in oxide thickness before and after polish was within the measurement accuracy of the instrument (1 nm). Thus arginine and H$_2$O$_2$ slurries provide Cu/SiO$_2$ polish rate selectivity in the range of 60 to well over 300. However, polishing experiments must be conducted with patterned wafers and slurry parameters such as the composition and pH and other parameters such as rotational speed and pressure must be optimized further to minimize the roughness and dishing and erosion losses.

4. Conclusions

A slurry with H$_2$O$_2$ as oxidizing agent and L-arginine as complexing agent provides a robust polishing performance for Copper CMP. The polish rate peaks at 0.1 vol% H$_2$O$_2$ and decreases moderately with further increase in H$_2$O$_2$ concentration. In the absence of an inhibitor, the static etch rate is high. The etch rate as well as the polish rate decrease with the addition of 0.01 M of uric acid and the inhibiting effect saturates at a concentration of 0.05 M of uric acid. Electrochemical experimental results are in line with the etch rate data. The increase in the $E_{corr}$ values with addition of uric acid suggests the formation of passivative layer and it is supported by the presence of kinks in the anodic branch of polarization curves. Cyclic polarization experiments indicate that the passivity provided by uric acid is stable. BTA is also very effective and potassium sorbate is moderately effective in inhibiting the copper corrosion in alkaline media. Hydrazine and citric acid do not reduce the corrosion rate of this alkaline slurry and in fact increase the corrosion rate. The proposed alkaline slurry provides excellent Cu/SiO$_2$ polish rate selectivity.
Acknowledgements

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References


Figure and Table Captions

Fig. 1. Molecular formula of (a) L-arginine and (b) uric acid

Fig. 2. Polish rates of copper at different vol% of H₂O₂ containing 1 wt% of arginine and 1 (wt/vol)% silica, with and without 0.05M of uric acid.

Fig. 3. Potentiodynamic polarization curves showing the effect of uric acid on Cu in solutions containing 5 vol% H₂O₂ and 1 wt% arginine.

Fig. 4. Etch rate and corrosion current as a function of uric acid concentration.

Fig. 5. Potentiodynamic polarization curves and reverse scan profiles obtained from positively polarizing copper in a solution containing 5 vol% H₂O₂, 1 wt% arginine and 0.05 M uric acid. Scan direction was reversed at potentials of 0.5, 0.7 and 1.0 V.

Fig. 6. Polish rates of Cu at various concentrations of uric acid in a slurry containing 5 vol% H₂O₂, 1 wt% arginine and 1 (wt/vol)% silica.

Table 1. Static etch rates of copper in a solution containing 5 vol% H₂O₂ and 1 wt% arginine, various of additives
Figure 1.a

\[
\begin{align*}
\text{H}_2\text{N} & \quad \quad \text{H} \\
\text{N} & \quad \quad \text{N} \\
\text{NH} & \quad \quad \text{NH}_2 \\
\text{C} & \quad \quad \text{C} \\
\text{OH} & \quad \quad \text{OH}
\end{align*}
\]

Figure 1.b

\[
\begin{align*}
\text{O} & \quad \quad \text{O} \\
\text{N} & \quad \quad \text{N} \\
\text{H} & \quad \quad \text{H} \\
\text{C} & \quad \quad \text{C} \\
\text{O} & \quad \quad \text{O}
\end{align*}
\]
Figure 2

Concentration of H$_2$O$_2$ (vol %)

Cu polish rate (nm/min)

- 0 M Uric acid
- 0.05 M Uric acid
Figure 4

- $I_{corr}$
- Etch Rate

[Graph showing the relationship between uric acid concentration (M) and $I_{corr}$ (mA) and Cu etch rate (nm/min).]
Figure 5

Scans reversed at
- 0.5 V
- 0.7 V
- 1.0 V
Figure 6

The graph shows the relationship between Cu polish rate (nm/min) and Uric acid Concentration (M). The data points indicate a decrease in Cu polish rate as the Uric acid Concentration increases.
<table>
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<th>Additive</th>
<th>Concentration (M)</th>
<th>Static etch rate (nm/min)</th>
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</thead>
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<tr>
<td>None</td>
<td>0</td>
<td>14</td>
</tr>
<tr>
<td>Hydrazine</td>
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</tr>
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