

## Selective Chemical Mechanical Polishing Using Surfactants

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Device fabrication using high density, small pattern size shallow trench isolation (STI) processes requires material removal selectivity during chemical mechanical polishing (CMP) steps for optimum product processing and quality control. To improve the selectivity of STI CMP processes, surfactants were applied to selectively polish silica as opposed to silicon nitrides surfaces. A ten-fold increase in selectivity over conventional colloidal silica slurry was achieved by the addition of sodium dodecyl sulfate (SDS) and pH adjustment. Adsorption characteristics of SDS on silica and silicon nitride were measured as a function of slurry pH and concentration of SDS. As indicated by streaming potential measurements and solution depletion adsorption experiments under acidic pH conditions, SDS adsorption on silicon nitride was significantly higher than silica primarily due to the electrostatic interactions. It was concluded that the preferential adsorption of SDS on silicon nitride results in the formation of a material-selective self-assembled passivation (lubrication) layer leading to selective polishing. Effects of different alkyl chain length of surfactants were tested. Various mixed surfactant systems were tested and it is believed that the addition of second surfactants promotes the adsorption on silica diminishing selectivity. The material-targeted boundary layer lubrication concept may be used to develop selective CMP polishing slurries.

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The fabrication of next-generation devices will require advances in shallow trench isolation (STI) technology.<sup>1-3</sup> STI chemical mechanical polishing (CMP) is particularly challenging due to the highly integrated and variable pattern density as well as smaller pattern sizes. Less than optimal CMP system can cause several defects such as dishing, nitride erosion, and failure to clear oxide that are detrimental to global planarization. To minimize such defects, current STI CMP processes comprised multistep or raw structure modification such as reverse mask, dummy active area, and additional active area.<sup>1</sup> For better productivity and process simplicity, a minimum number of processing steps is highly desirable. Accordingly, "high selectivity single-step" slurry designs are being widely investigated.<sup>4,5</sup> The term "selectivity" in this paper is defined as the ratio of material removal rate (MRR) of silica to that of silicon nitride as described below. It is the slurry property as to how much more it can polish silica than silicon nitride

# $Selectivity = \frac{Material Removal Rate of Silica}{Material Removal Rate of Silicon Nitride}$ [1]

In general, conventional silica-abrasive-based STI CMP slurries exhibit selectivity in the range of 3–4.<sup>5</sup> Besides its influence on planarization, high selectivity slurries are known to provide more reliable endpoint detection capability. Generally, if the oxide to nitride selectivity is greater than 15, monitoring wafer carrier motor current can be utilized for efficient endpoint detection.<sup>6</sup>

In recent years, ceria-based abrasives have shown potential for high selectivity and have attracted attention in developing advanced selectively polishing systems. In ceria CMP, maximum polishing rate and selectivity are achieved at around pH 8, the isoelectric point (IEP) of ceria. This, however, leads to agglomeration of ceria abrasive particles yielding poor surface morphologies with scratches and higher roughness.<sup>6</sup> Alternatively, polymer additives have been used to improve selectivity, however, the overall material removal rate (MRR) decreased significantly upon polymer addition.<sup>7</sup> In this study, a different approach to increase STI CMP selectivity by introducing a selective passivation (lubrication) layer using surfactants is investigated.

Previous research at the University of Florida<sup>8-10</sup> involving surfactant mediated lubrication effects in CMP is well documented. Basim and co-workers showed that the addition of long-chain cationic surfactant produced better defect-free surface morphology but the polishing rate was extremely small due to the lubrication effect of the added surfactant.<sup>8</sup> In other words, long-chain surfactants were determined to act as antipolishing agents. Subsequently, Vakarelski and co-workers showed that formation of an intervening film of surfactants on solid surfaces caused a significant reduction in friction between the wafer and the abrasive particles.<sup>10</sup> In the proposed strategy, it was envisioned that a selective coating of surfactant on silicon nitride can act as an antipolishing barrier without significantly affecting the MRR of silica. Considerations that the selected surfactant needs to be easily removed at the end of the CMP process preclude surfactants that chemisorb onto any of the CMP substrates.

#### Experimental

CMP experiments were conducted using Klebosol 1501-50 colloidal silica slurry obtained from Rodel Co. after diluting the original slurry at 30 to 12 wt %. The slurry pH was measured to be around pH 10.4 after dilution. The original slurry pH was around pH 10.8. Both silica and silicon nitride thin films were purchased from Silicon Quest International. Silica thin films were fabricated by the plasma-enhanced chemical vapor deposition (PECVD) method using tetra ortho silicate (TEOS) as the precursor. Silicon nitride films were fabricated with low-pressure chemical vapor deposition (LPCVD) by using dichlorosilane (SiCl<sub>2</sub>) and ammonia (NH<sub>4</sub>). Hardness values were measured by the Nanoindentation method using Hysitron Triboindenter purchased from Hysitron Co. All the CMP experiments were performed under a 7 psi load using 1 imes 1 in. silica and silicon nitride blanket wafers. Relative errors in MRR from CMP experiments were around 5%, and several points are randomly repeated for reproducibility check. IC 1000/Suba IV stacked pads supplied by Rodel Inc. were utilized as CMP pads. The TegraPol-35 with TegraForce-5 from Struers Co. tabletop polisher was used for polishing experiments. The rotation speed was controlled at 150 rpm both for the pad and the wafer. MRR was measured by the decrease in thickness after CMP by ellipsometry (Woollam EC110 Ellipsometer). For measurement of surface roughness after CMP, a Digital Instruments Nanoscope III atomic force microscope was used. Zeta potential of silica and silicon nitride wafers was measured by the streaming potential technique (Paar Physica Electro Kinetic Analyzer). Cleaning was done with 99% acetone, ethyl alcohol, and nano purity water produced by a Millipore filtration system, which have internal specific resistance of more than 18.2 M $\Omega$ . No further chemicals were used on the wafers. For adsorption studies, silica from Geltech Co. and silicon nitride from Ube Co. (SN-E10) were used to simulate silica and silicon nitride substrates. The particle size of the silica was measured to be around  $0.53 \ \mu m$  by Coulter and that of silicon nitride, which was measured by centrifugal sedimentation, was reported to be around 0.5 µm by the manufacturer. Their specific surface areas were measured to be

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**Figure 1.** (Color online) (a) Variation of material removal rate (MRR) as a function of slurry pH with and without 16 mM sodium dodecyl sulfate (SDS). (b) Accompanying selectivity of the slurry.

8.1 and 10.4 m<sup>2</sup>/g, respectively, by Quantachrome Nova 1200, Brunauer-Emmett-Teller (BET) surface area measurement technique. The particle size of the colloidal silica contained in the slurry was measured to be around 90 nm by Coulter LS13320, and its specific surface area was measured to be 34 m<sup>2</sup>/g by Quantachrome Autosorb 1C-MS. Phoenix 8000 UV-Persulfate TOC Analyzer was used to measure adsorption density of SDS on silica and silicon nitride particles. 99% Sodium dodecyl sulfate (SDS) surfactants from Acros Organics Co. and Fisher Scientific Co. were used as received. 98% dodecyl alcohol from Eastman Kodak Co., 95% sodium tetradecyl sulfate from Acros Organics Co. and Tween 80 from Fischer Scientific were also used as received.

#### **Results and Discussion**

Selective adsorption of a surfactant can be achieved if there is adequate difference in the surface charge characteristics of the substrates. This concept has been successfully used to achieve selective coating of surfactants in the mineral flotation.<sup>11,12</sup> Interactions between solid surface and charged polar head of the surfactant molecules determine the adsorption strength and the resultant adsorption density. In the present study, an anionic surfactant, SDS, was selected to achieve a selective passivating layer on silicon nitride. It is well known that the IEP of silicon nitride is higher than that of silica resulting in less negative potential for silicon nitride above the IEP.<sup>13</sup> The concentration of SDS was adjusted to 16 mM, twice the critical micelle concentration (CMC), which has been shown previously to yield dispersion of silica abrasives in a CMP slurry.<sup>8</sup>

Figure 1 shows the variation of MRR of silica and silicon nitride



Figure 2. Surface finish of silica and silicon nitride wafers processed with standard and high selectivity slurry.

and accompanying selectivity of the slurry, with and without SDS addition, as a function of CMP slurry pH. It has been well documented that colloidal silica suspensions show stable dispersion at two pH regions from pH 2 to 4 and more than pH 8. At pH higher than 8, electrostatic repulsion between almost completely ionized silanol groups  $(SiO^{-})$  on silica surface leads to dispersion stability.<sup>14,15</sup> In the intermediate pH range (4–8), due to the reduced electrostatic repulsion, silica particles can come closer and gelation occurs by the formation of a silicic acid chain [-Si(OH)<sub>2</sub>-O-Si(OH)<sub>2</sub>-OH], or formation of siloxane bonds (Si-O-Si) by reaction between acidic ionized silanol (SiO<sup>-</sup>) and surface silanol groups.<sup>14,15</sup> Stability at lower pH (2–4) has been attributed to hydration forces due to modified water structure at the silica surface or repulsion between silanol (SiOH) surface groups.<sup>14-16</sup> Silanol groups are extremely hydrophilic, resulting in higher interaction through hydrogen bonding with water than particle-particle interaction. Formed water layer prevents silica surfaces from contacting each other and prevents agglomeration by van der Waals interaction. Alternative explanation involves the formation of a surface gel layer or short polymer-like hairs17 protruding from silica surface, giving rise to steric repulsion. It is well known that gelation depends on the solids loading, particle size, temperature, etc.<sup>18</sup> In the present study, no gelation was observed in the entire pH range probably due to low solids loading (12 wt %) and magnetic stirring of slurry prior to CMP. However, the slurry at pH 2 showed gelation after 1 week in the absence of agitation.

Slurry selectivity without SDS was measured to be around 3, which means that silica showed around three times higher MRR than silicon nitride in the entire pH range tested. This MRR difference is closely related to the hardness of the two materials. Hardness values measured by nanoindentation method showed  $8.5 \pm 0.3$  and  $23.5 \pm 1.0$  GPa for silica and silicon nitride wafers, respectively.

With the addition of SDS to the slurry, MRR of silica and silicon nitride was determined to be lower in the entire pH range investigated in the present study. The selectivity increased slightly at pH 10.4, because the MRR decrease is slightly higher for silicon nitride than for silica. At pH 3, MRR of silicon nitride decreased more substantially than that of silica leading to an increased selectivity of 92%, which is similar to the selectivity of state-of-the-art ceria abrasives of 5. Finally, at pH 2, the MRR of silicon nitride was minimal and selectivity increased to 25, or higher than in the absence of surfactant addition. The surface of silicon nitride appeared to be fully passivated with the surfactant layer at pH below its IEP of pH 4.5, with minimal effect on silica CMP. Surface qualities of wafers after CMP by slurry are shown in Fig. 2. Surfactant addition did not



Figure 3. (Color online) Variation of zeta potential of silica and silicon nitride wafers and adsorption density of 16 mM SDS on silica and silicon nitride powder measured by total organic carbon (TOC) method.

cause any defects on either surfaces in terms of root-mean-square (rms) roughness and maximum deformation  $(R_{\text{max}})$ .

To understand the reasons for observed selectivity, zeta potential and adsorption density measurements were conducted as a function of slurry pH (Fig. 3). IEP of silicon nitride and silica wafers were measured to be about pH 4.5 and 2.2, respectively. The difference in IEP results from the different surface groups on each material. It is well known that acidic silanol (SiOH) are the major surface groups on silica, while silicon nitride surface consists of basic amine (Si<sub>2</sub>NH) and acidic silanol (SiOH) groups.<sup>13</sup> These surface groups can acquire a charge in aqueous solution according to following reactions

$$SiOH = SiO^{-} + H^{+}$$
[2]

$$Si_2NH + H^+ = Si_2NH_2^+$$
 [3]

Consequently, the zeta potential of silicon nitride is higher due to the positively charged amine groups on its surface.

The adsorption density of SDS was measured to be higher on silicon nitride than on silica below their IEPs. This is attributed to the electrostatic interaction between substrate and surfactant molecules. At pH 2, the zeta potential of silicon nitride was measured to be +40 mV, whereas, that of silica was around +3 mV. Accordingly, the adsorption density on silicon nitride was measured to be more than six times higher than on silica, resulting in complete passivation of the former. Above the IEPs for both materials, there was still measurable adsorption, and the adsorption density on silicon nitride was higher than for silica probably due to more positive sites on silicon nitride from surface amine groups. The exact mechanism of anionic surfactant adsorption on negatively charged surfaces at high pH is not completely known at this stage. However, there have been several reports on SDS adsorption on the negatively charged silica surface.<sup>19,20</sup> Hydrogen bonding and sodium-ion-mediated surfactant bonding are proposed as plausible mechnisms.

To measure the effect of surfactant concentration on selectivity, the MRR and resultant selectivity were determined as a function of added surfactant concentration (Fig. 4). Upon adding SDS, the MRR for both silica and silicon nitride started to decrease and reached a minimum above 16 mM. The maximal decrease in MRR for silica was around 20% from the original value, and that for silicon nitride was more than 90%, thus resulting in ten times higher selectivity than without surfactant addition. No further change in MRR or selectivity was observed once the added surfactant concentration exceeded 16 mM. It has been reported that once the equilibrium concentration reaches CMC, no more adsorption changes are observed due to electrostatic repulsion between surfactant aggregates.<sup>21</sup> Re-



Figure 4. (Color online) Variation of MRR and accompanying selectivity of Klebosol slurry (12 wt %) as a function of added SDS concentration at pH 2.

search by lateral force microscopy by Vakarelski et al. has shown that beyond the CMC of the surfactant (dodecyltrimethylammonium bromide,  $C_{12}TAB$ ), there was no further decrease in the lateral force on silica wafer.<sup>10</sup> Consequently, it is hypothesized that maximum decrease in MRR will occur when the bulk concentration reaches the CMC of SDS (around 8 mM).<sup>22</sup> However, in this research, two times higher concentration of surfactant than its CMC was required to achieve maximum selectivity. Measurement of SDS adsorption on the CMP slurry as a function of pH showed that about 91% of added 16 mM SDS adsorbed on the abrasive particles at pH 2 as shown in Fig. 5. Area per molecule calculated by Gibbs adsorption equation<sup>2</sup> by using adsorption density measured at pH 2 was around 70 Å<sup>2</sup>/molecule. Higher area per molecule than the literature value of 53 Å<sup>2</sup>/molecule<sup>22</sup> from monolayers in water-air interface implies that at a given condition, SDS formed less dense monolayer on colloidal silica particles. The driving force for SDS adsorption is electrostatic attraction between surfactant and abrasive particles and hydrophobic interaction between adsorbed alkyl chains of surfactants. The possible reason for the higher dosage of surfactants than expected is that real CMP process does not generate the equilibrium conditions needed for the surfactants to adsorb on the wafers. Due to process requirements, the wafer and polishing pad should be rotated (150 rpm in our research). Centrifugal force induced by this rotation limits residence time for the silica abrasive particles and the surfac-

2.38 2.36 Adsorption density ( $\mu$ mol/m<sup>2</sup>) 2.34 2.32 2.30 2.28 2.26 2.24 2.22 2.20 2 4 6 8 10 12 pН

Figure 5. Adsorption density of SDS on 12 wt % Klebosol slurry with 16 mM SDS as a function of pH.

tants to be captured in the regions between wafer and polishing pad. Surfactants which are already adsorbed on particles should diffuse to a newly introduced wafer surface. This phenomenon is more closely related with dynamic aspect of surfactant adsorption. Slow relaxation time  $(\tau_2)$  is attributed to the micelle formation and dissolution process in surfactant solution. The reported  $\tau_2$  for SDS is around 2.32  $\times$  10<sup>-3</sup> s.<sup>23</sup> However, according to the report by Patist and co-workers, when 15 mM SDS was used for foaming experiments, dynamic surface tension decreased as a function of bubble lifetime until it reached saturation after about 2 s.<sup>24</sup> Recently, Philipossian et al. reported the mean residence time (MRT) of colloidal silica slurry in the regions between pad and wafer through the frictional coefficient measurement.<sup>25</sup> Assuming that other conditions are similar, MRT in our study is expected to differ by several seconds from their data. Those two numbers are comparable, which means that migration of surfactants to newly formed wafer surfaces can be limited due to the high speed rotation. Standard free energy of adsorption can be calculated by a modified Stern-Graham equation<sup>2</sup>

$$\Gamma = 2rC_{\rm o} \exp\left(-\frac{\Delta G_{\rm ads}^{\rm o}}{kT}\right)$$
[4]

where  $\Gamma$  is the adsorption density, r is the effective radius of the adsorbed ion, k is the Boltzmann constant,  $C_0$  is the bulk concentration, T is 298 K, and  $\Delta G_{ads}^o$  is the adsorption free energy. Adsorption free energy is the driving force for surfactant adsorption and is the sum of various molecular interactions.<sup>21</sup> In our current study, its components can be categorized to two interactions. One of them is the interaction between the polar head of SDS and the surface which consists of electrostatic and hydrogen bonding interactions. The other one is hydrophobic interactions between alkyl chains of SDS. By using the measured adsorption density in Fig. 5 with the radius of SDS micelle (20 Å),<sup>22</sup> adsorption free energy of SDS on silica abrasives was calculated to be -6.05 kT at pH 2. With all other conditions the same, driving force for surfactant migration from silica abrasive to silicon nitride wafer must be caused by the zeta potential difference between them. Electrostatic component of adsorption free energy can be given by  $ze\psi_{\delta}$ ,<sup>21</sup> where z is the valence of the adsorbate species, *e* is the charge of the electron, and the  $\psi_{\delta}$  is the potential at the  $\delta$  plane, which is assumed to be the zeta potential. It was calculated to be -1.28 kT by using the zeta potential of silica and silicon nitride at pH 2, which is much lower than the adsorption free energy of SDS on abrasive particles. This difference can act as an additional energy barrier for this process and is attributed to the hydrogen bonding and hydrophobic interactions.

From the above discussion, it is clear that the adsorption density of surfactant on wafer is an important factor in determining the slurry selectivity. To reduce the required dosage of the surfactant, a longer alkyl chain length surfactant was examined, as it was expected to exhibit better lubrication effects at a smaller added concentration. This is attributed to the formation of more compact surfactant layers.<sup>8</sup> MRR and polishing selectivity as a function of alkyl chain length of the sodium alkyl surfactant are plotted in Fig. 6. The surfactant concentration was selected to be twice the CMC value to compensate for the loss of surfactant on the silica abrasive particles. As was expected, SDS with longer alkyl chain length  $(C_{12})$  resulted in higher MRR decrease for silicon nitride with a negligible effect on MRR for silica, resulting in higher selectivity than sodium decyl sulfate  $(C_{10})$ . However, when sodium cetyl sulfate  $(C_{14})$  was examined, there was a smaller decrease of MRR for silicon nitride, resulting in a lower selectivity. Considering the Krafft point of C14 sodim sulfate (30°C) <sup>23</sup> to be higher than room temperature and higher than that of SDS (16°C), <sup>23</sup> the slurry was heated to higher temperature than the Krafft point. It is suspected that a lowering of surfactant adsorption or lower rigidity of adsorbed surfactant layer is responsible for the measured MRR at higher temperatures.

Alternatively, to decrease the dosage of the surfactant required to achieve desired selectivity, a mixed surfactant system including Tween 80/SDS, and dodecyl alcohol/SDS were evaluated at pH 2.



**Figure 6.** Effect of alkyl chain length of sodium alkyl sulfate on MRR and selectivity at pH 2. The concentration was adjusted to two times the CMC to compensate for the loss during the CMP process.

MRR and selectivity are plotted in Fig. 7. It is well known that a small quantity of nonionic surfactant such as alcohol can significantly decrease surface tension by facilitating packing of surfactants in the interface.<sup>26</sup> In the case of dodecanol and SDS, selectivity was lower for mixed surfactant systems than for 16 mM SDS alone. It is possible that the addition of small amounts of dodecanol promotes adsorption of SDS on both silica and silicon nitride. While there was no appreciable change in MRR on silicon nitride, the adsorption of SDS was highly increased by dodecanol addition, enabling higher MRR decrease on silica.

It has been reported by Pala and co-workers that surfactant mixture of SDS and various nonionic surfactants can produce synergistic effects for dispersion of slurry under high ionic strength.<sup>27,28</sup> When 8 mM Tween 80 was added to 16 mM SDS, MRR of silica was highly suppressed, whereas that of silicon nitride remained almost unchanged, resulting in poor selectivity. It is well known that nonionic surfactants such as Tween 80, which has ethylene oxide group (OC<sub>2</sub>H<sub>4</sub>), can adsorb on silanol group (SiOH) on silica through hydrogen bonding.<sup>29</sup> It is possible that for silicon nitride, the driving force for surfactant mixture adsorption was smaller due to the charge shielding effect of nonionic surfactant (Tween 80) on



Figure 7. MRR and selectivity obtained by slurries with various surfactant and surfactant mixtures at pH 2. Slurry A (16 mM SDS) was included for comparison.

anionic surfactant (SDS). However for silica, the adsorption of surfactant mixture was highly facilitated with additional driving force of adsorption through hydrogen bonding. These observations strongly suggest that surfactant or surfactant systems that exhibit strong preference for silicon nitride is crucial for developing surfactant-based high selectivity slurries.

#### Conclusions

Colloidal silica, which shows high dispersion stability in the range of pH 2-11, was utilized to develop high selectivity slurry. Addition of SDS at pH 2 resulted in more than ten times higher selectivity than conventional slurry. Additionally, AFM roughness measurement showed that surfactant addition does not have any detrimental effect on surface finish. Adsorption density measurements revealed that there is a preferential higher adsorption of SDS on silicon nitride possibly due to high electrostatic attraction as compared to silica. SDS adsorption behavior is believed to result in differential lubrication and hence lower polishing efficiency of silicon nitride as compared to silica. The CMP characteristics examined as a function of added SDS concentration showed that decrease in MRR and increase in selectivity leveled off at about twice the surfactant CMC and remained unchanged thereafter. Surfactant requirement appears to be driven by their adsorption primarily on silica abrasive particles. To reduce surfactant dosage overall, it appears that selective surfactant coatings on substrate can yield higher selectivity without any adverse impact on surface finish. Longer alkyl chain length surfactants were tested, which yielded higher selectivity at lower dosage. The addition of long-chain-length alcohol to substitute for surfactant resulted in lower selectivity due to a higher decrease in MRR for silica. Mixed ionic and nonionic surfactant systems, on the other hand, resulted in poor selectivity due to larger decrease in MRR on silica and smaller decrease in MRR on silicon nitride. These findings can be used as a platform for developing selective polishing CMP slurry.

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