AFM-Based Study of the Interaction Forces between Ceria, Silicon Dioxide and Polyurethane Pad during Non-Prestonian Polishing of Silicon Dioxide Films

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A colloidial AFM-based method was used to understand the role of diallyldimethyl ammonium chloride (DADMAC) molecules in non-Prestonian silicon dioxide removal obtained using ceria-based slurries. A series of force-distance measurements between silicon dioxide films, polyurethane IC1000 polishing pad and ceria abrasives in liquid media with and without DADMAC molecules were made. It was shown that at 15 mM concentration, DADMAC molecules yield non-Prestonian behavior by interfering between ceria particles and the oxide film. The effects of the DADMAC additive on the removal rates of the oxide films and their corresponding coefficients of friction (COFs) values were also investigated. Finally, the correlation between AFM force measurements, removal rates and COF values measured during polishing are discussed. Interestingly, below a threshold COF value of ∼0.23 to 0.24, corresponding to a threshold down pressure of ∼2 psi for both 15 mM and 25 mM DADMAC concentration, no oxide film removal was observed.

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Manuscript submitted May 27, 2015; revised manuscript received June 19, 2015. Published July 3, 2015. This paper is part of the JSS Focus Issue on Chemical Mechanical Planarization: Advanced Material and Consumable Challenges.

Chemical Mechanical Planarization (CMP) is a critical step during the fabrication of ultra large scale integrated circuits (ULSICs) in the semiconductor industry. Since the feature dimensions of current ICs are continually scaling down (latest technology node of 14 nm was recently announced by Intel corp.), the demands on surface planarity of various films have become more stringent. An improved understanding of polishing mechanisms can help to achieve the desired polishing requirements. These requirements include minimizing defects such as surface roughness, micro-scratches, etc.1,2

One of the main applications of CMP in the front-end-of-the-line is shallow trench isolation (STI) fabrication. The STI structure isolates two adjacent semiconductor device components (transistors, diodes, etc.) preventing electrical current leakage between them. Application of CMP for fabricating these STI structures has been widely studied. However, precise end-point detection during the fabrication of these structures remains a challenge. As a result, due to the non-uniform polishing of the oxide film across the wafer,3 over-polishing time is almost always required to remove any residual oxide from the nitride areas. This can result in considerable dishing in the oxide-filled trenches in the STI structure. Dishing takes place if the oxide material inside the trenches is polished, especially at the low pressures created by contact with the polishing pad. One efficient method that can minimize dishing is to use a non-Prestonian polishing slurry,4,5 which causes negligible removal below a threshold applied downward pressure. Recently, it was shown that a ceria-based slurry containing 15 mM diallyldimethyl ammonium chloride (DADMAC) as an additive resulted in non-Prestonian oxide removal6 with negligible removal below ∼2 psi applied pressure. Based on the measurements of the adsorption strength of DADMAC molecules on ceria and silica surfaces, using various techniques including thermo-gravimetric analysis (TGA) and UV-visible spectroscopy, the observed non-Prestonian behavior was attributed to the strong adsorption of DADMAC molecules on the oxide film.7 However, the interaction forces involved in the polishing process were not well characterized. Since the CMP removal mechanism is based on a synergetic combination of chemical and mechanical interactions, it is necessary to understand both aspects of CMP to obtain an optimized polishing result.8–11

The abrasive-wafer and pad-wafer interactions both play critical roles in polishing. The downward pressure applied during polishing is transformed to the wafer surface by wafer-pad interaction along with the contact between particles trapped between polishing pad asperities and the wafer surface. On the other hand, the material removal during polishing is mainly due to the shear force applied by the particles and the pad asperities to the wafer surface.12,13 Hence, characterizing the transferred normal and friction (shear) forces from particles and polishing pad asperities to the wafer surface can provide a better understanding of the polishing results.14–16

Recently, several atomic force microscopy (AFM)-based techniques have been used to measure the interaction forces between various surfaces in CMP for better understanding of the removal mechanisms.17–21 For example, Ong et al.17 functionalized AFM probes with nano-size particles and measured the force between these particles and various surfaces. Colloidal AFM is commonly used to measure interactions between colloidal particles and also between particles and planar surfaces.22 Applying this method, Armini et al.18 measured the interaction forces between a large silica particle representing an oxide surface and two types of silica-coated and ceria-coated poly (methyl methacrylate) based terpolymer abrasives attached to a flat surface that is relevant for CMP applications. Volkov et al.20 modified this method to measure the adhesion force of silica and ceria particles of irregular shape with silica surfaces and correlated it with the polishing rates obtained using slurries containing those abrasives. Similar to Armini et al.,22 they used large silica spheres to mimic a flat silica film surface, but embedded the nanoparticles in a thick layer of epoxy on a wafer surface. They claimed that the silica and ceria nanoparticles, which are only partially embedded in the epoxy, form a sufficiently flat surface.

In this paper, employing a colloidal AFM method similar to the one reported in Ref. 20, the interaction forces between silicon dioxide, polyurethane pad and non-spherical ceria abrasives were measured in the presence and absence of DADMAC. These non-spherical ceria particles are the same as those that were used in Ref. 7 and several
of our earlier publications cited there which include images of these ceria particles. Also, we do not repeat here many of the useful and necessary details that are well described in Ref. 20 including the nature of the particle-coated surface topography and its impact on AFM scanning. The measured force-distance curves in DI water at pH 4 with and without DADMAC additive are correlated with the non-Prestonian oxide removal rate behavior reported in Ref. 7 along with the coefficient of friction (COF) values obtained during CMP.

Materials and Experimental Procedures

Substrate materials.— A piece of flat polyurethane IC1000 pad containing no grooves (supplied by Dow chemical company, Michigan, USA) and a cleaned silicon coupon coated with ceria particles with a nominal average diameter of 140 nm (supplied by Ferro corp. Ohio, USA) were used in the force measurement experiments. The following method was employed to coat 1.3 × 1.3 cm² polished silicon coupons with ceria particles. A silicon coupon was cleaned with dichloromethane, ethanol and DI water sequentially for 5–15 min in a sonication bath to remove any surface contamination. The coupon was then dipped in H2O2:NH4OH:water (1:1:1) solution for 1 h in a water bath at a constant temperature of 60 °C to form a thin oxide layer on the substrate. Afterwards, the surface of the silicon coupon was functionalized by dipping it in a 1 wt% solution of diethylphosphatoethyltriethoxysilane (DPTS) in dry toluene for 6 hours. Finally, ceria nanoparticles were deposited from a dispersion by spin-coating on the DPTS-functionalized silicon dioxide surface. All samples were calibrated for quantitative measurements. The image of the imaged cap shown in Figure 1c as \( R = \frac{(x/2)^2 + h^2}{2h} \) is 2.12 \( \mu \)m. This image provides only the dimensions of the particle but also further reveals the spike radius with the colloid sphere introduces negligibly small errors of the imaged cap shown in Figure 1c as \( R = \frac{(x/2)^2 + h^2}{2h} \). The image of the cap not visualizations of the microsphere confirming that the particle is rather smooth and spherical (Figure 1b). The radius of the sphere was calculated from the width (\( x = 2.63 \mu \)m) and the height (\( h = 0.53 \mu \)m) of the imaged cap shown in Figure 1c as \( R = \frac{(x/2)^2 + h^2}{2h} = 1.9 \mu \)m using the formula shown in Figure 1d. The image of the cap not only provides the dimensions of the particle but also further reveals the nature of the particle-coated surface topography and its impact on AFM scanning. The measured force-distance curves in DI water at pH 4 with and without DADMAC additive are correlated with the non-Prestonian oxide removal rate behavior reported in Ref. 7 along with the coefficient of friction (COF) values obtained during CMP.

Colloidal AFM tip materials.— To prepare colloidal probes, standard tipless silicon cantilevers with a nominal spring constant and nominal resonance frequency of 37 N/m and 300 kHz, respectively, coated on the reverse side with a reflective aluminum layer (Applied NanoStructures, Inc., California, USA) were used. A silica sphere was glued onto the cantilever using the manual x-y and motorized z micromanipulator of a Bruker Multimode 8 AFM microscope (Bruker, California, USA). Silica particles and a small droplet of two component epoxy glue (Araldite) were placed aside on a freshly cleaved mica substrate. Using the micromanipulators, the cantilever was brought into contact with the glue and the excessive glue was removed by wiping the probe against a clean mica surface. Then the cantilever was manually brought into contact with a silica particle visible in the instrument’s microscope by raising the mica substrate using the z-stage manipulator and allowing the particle to attach itself to the cantilever by capillary forces. The mica sample was then lowered and the particle-cantilever attachment was confirmed by SEM (Scanning Electron Microscopy) microscopy and AFM imaging of the calibration sample (see Figures 1a and 1b). Due to the short pre-cure time of the epoxy glue (approximately 5–10 minutes), having several pre-selected silica spheres on the mica surface and a proper alignment of the cantilever relative to the sample before the gluing process can increase the chances of a successful attachment considerably. After an overnight wait for complete hardening of the epoxy glue, probes were calibrated for quantitative measurements.

Colloidal probe characterization.— To inspect the quality of silica particle attachment, an SEM image of the prepared probe was obtained and shown in Figure 1a. A silicon calibration grating substrate TGT1 (prepared by NT-MDT, Arizona, USA) which creates an image that consists of an array of spherical caps (Figure 1b) was also used to image the particle probe. The grating substrate is made of a 2 × 2 mm² array of sharp spikes with a tip curvature radius that is less than 10 nm and a tip angle less than 30°. Convolution of the spike radius with the colloid sphere introduces negligibly small errors for the particle size used in this work. It must be noted that the spikes are approximately 0.6–0.8 \( \mu \)m tall, the pitch between closest spikes is 2.12 \( \mu \)m and the diagonal pitch is 3 \( \mu \)m. This image provides visualizations of the microsphere confirming that the particle is rather smooth and spherical (Figure 1b). The radius of the sphere was calculated from the width (\( x = 2.63 \mu \)m) and the height (\( h = 0.53 \mu \)m) of the imaged cap shown in Figure 1c as \( R = \frac{(x/2)^2 + h^2}{2h} = 1.9 \mu \)m using the formula shown in Figure 1d. The image of the cap not only provides the dimensions of the particle but also further reveals...
the presence of any debris that may be present on its surface or deformations in its shape. This enables a faulty colloid probe to be identified prior to force measurements and avoid its use. The deflection sensitivity was calibrated on a sapphire surface in water, and the spring constant of the cantilever was calibrated using thermal tune method at the same conditions. The calibration procedures were performed before and after each experiment to ensure accurate measurements.

However, the addition of DADMAC in this medium leads to the adsorption of DADMAC on the silica particle due to electrostatic attractions. The resulting neutralization of the silica negative surface charges results in the elimination of the attractive regime between silica and ceria as shown in Figure 3. This attractive to repulsive transition appears to be responsible for the observed non-Prestonian oxide removal regime reported in Ref. 7 when DADMAC was used as an additive for ceria-based slurries. This is discussed further below.

Results

AFM force measurements.— A schematic representation of force-distance curve measurement method using a silica sphere probe in a liquid medium involving an IC1000 polyurethane pad coupon and an oxide surface coated with ceria nanoparticles is shown in Figure 2.

Force-distance curves were measured in liquid media using a Bruker Multimode 8 SPM with NanoScope (version 8.15) and NanoScope V Controller (Bruker, California, USA), operating in PeakForce Quantitative Nanomechanical mode (PF-QNM). This technique was elaborated in several previously reported publications,23–26 The standard tapping model liquid cell, MTFML (Bruker, California, USA), was used for these measurements which were performed in DI water with pH adjusted to 4 using nitric acid, both in the presence and absence of DADMAC (Sigma-Aldrich, Missouri, USA). In each experiment, the surface was moved in a sinusoidal manner with an amplitude of approximately 100 nm. The maximum deflections of the cantilever (the peak force) were kept constant with a feedback system, while the cantilever deflection and piezo position were recorded continuously during the scanning of the surface in point and shoot modes. Force measurements were performed with a scan rate of 1.0 Hz on a sample area of $1 \times 1 \mu m^2$ and a lateral resolution of $10 \times 10$ pixels. All AFM data analysis and data processing were made with the NanoScope Analysis software version 1.40.

Figure 3 shows the measured averaged force-distance curves (each averaged from 100 different curves) of the interactions between a silica spherical particle (representing a SiO$_2$ film) with the ceria surface and a silica spherical particle with the polyurethane pad in DI (deionized) water adjusted to pH 4 using nitric acid with and without 15 mM DADMAC as additive. As shown in Figure 3, a weak repulsion was observed between the SiO$_2$ sphere and the IC1000 pad in DI water at pH 4. Since both the IC1000 pad and the silicon dioxide surface are negatively charged at pH 4, as reported in Refs. 7 and 27, the observed repulsive force originates from the electrostatic repulsion between these surfaces. Adding 15 mM DADMAC to the medium at pH 4 did not change the repulsion between IC1000 pad and SiO$_2$ surfaces even though the DADMAC molecules with positively charged groups adsorb on both. In contrast, force measurements between the ceria-coated silicon substrate and the silica particle in DI water resulted in an attractive/adhesive force at pH 4. Abiade et al.28 also showed similar results between a silica probe and ceria thin films at pH 4.5. This attractive force arises from the interaction of a silica surface consisting of negatively charged species with positively charged ceria particles.

Material removal rate and COF measurements.— Blanket silicon dioxide films deposited on 8” diameter silicon wafers by plasma enhanced chemical vapor deposition (PECVD) using tetraethoxysilicate (TEOS) as the silicon source were polished for 1 min on a POLI-500 polisher (G & P technology, South Korea). Down pressures in the range of 1 to 4psi, 87/93 rpm carrier/platen speed, and a slurry flow rate of 200 ml/min were used as the experimental conditions during polishing. The slurries were prepared using ceria particles ($d_{mean} \sim 140$ nm, Ferro Corp, Ohio, USA) with and without DADMAC (Sigma-Aldrich, Missouri, USA). The oxide removal rates were determined by measuring the thickness of the films before and after polishing using an F-20 interferometer (Filmetrics, California, USA). The removal rates were calculated from the average of the change in the thickness measured at 20 points across a diameter of the wafer, and the standard deviation reported was based on the data from a total of 40 locations obtained from two different polished wafers. The polishing pad (IC1000, K-groove) was conditioned in-situ using a 4” diamond grid pad conditioner (SI-60, obtained from 3 M Inc. Minnesota, USA).

The real time coefficient of friction (COF) was measured using the multi-sensor monitoring system$^{29}$ available with the POLI-500 polisher that enables dynamic data collection. In this system, the frictional sensor (SlimLine Sensor—9134B21 from Kistler group, Switzerland) is used to measure the net friction between the wafer, particle and the
pad. Friction forces were measured for every 10 ms over a polishing time of 60 seconds for each polishing. The standard deviation of the frictional force was also determined using these measurements.

Friction characteristics between the polishing pad, the abrasives and the wafer surface play a key role in material removal rate in CMP. COF depends on the applied down pressure, particle-surface interactions, and material properties of the surfaces in contact. In Figures 4.1 and 4.2, the COF data and removal rates of SiO2 films, respectively, are shown as a function of the applied down pressure. The COF and removal rate data were obtained by polishing SiO2 films with 1 wt% ceria slurry without any additive and with 15 and 25 mM DADMAC all at pH 4.

When the applied down pressure was increased during polishing, the value of COF increased continuously in the absence of DADMAC (Figure 4.1a) along with the oxide removal rate (Figure 4.2d). However, the addition of 15 mM DADMAC to the polishing slurry completely suppressed the removal rates at 1 and 2 psi applied pressure, followed by a linear increase beyond the threshold pressure of 2 psi (Figure 4.2e). Similarly, the COF data for 15 mM DADMAC slurry showed a relatively low value for down pressures of 1 and 2 psi and a considerable increment beyond 2 psi (see Figure 4.2b). A further increment of DADMAC concentration to 25 mM suppressed the oxide removal rates completely all the way up to 4 psi down pressure (Figure 4.2f). The behavior of the COF data was also similar and in the range of ∼0.2-0.24, relatively low values in comparison to the ones obtained for no DADMAC and with 15 mM DADMAC (Figure 4.1c).

In Table 1, a quantitative summary of the measured COF data shown in Figure 4 for oxide polishing using 1 wt% ceria slurry without additive and with 15 and 25 mM DADMAC at pH 4 is listed.

### Table 1. Quantitative summary of the measured COF and RR data shown in Figure 4.

| Pressure (psi) | No DADMAC | 15 mM DADMAC | 25 mM DADMAC |
|---------------|-----------|--------------|--------------|
|               | RR (nm/min) | COF          | RR (nm/min) | COF          | RR (nm/min) | COF          |
| 1             | 175.4      | 0.49         | 0           | 0.19        | 0           | 0.19        |
| 2             | 326.4      | 0.58         | 0           | 0.22        | 0           | 0.20        |
| 3             | 487.6      | 0.60         | 69.3        | 0.36        | 0           | 0.23        |
| 4             | 632.7      | 0.63         | 154         | 0.39        | 0           | 0.24        |

#### Figure 4. 1. COF and 4.2. removal rates of SiO2 films as a function of applied down pressure using 1 wt% ceria slurry with and without 15 and 25 mM DADMAC at pH 4.

### Discussion

As elaborated in Ref. 7, DADMAC creates an adsorbed layer on the oxide film which can presumably restrict the contact of ceria particles with the oxide film and, consequently, suppress the removal rates in low pressure zones. This is consistent with the AFM results shown in Figure 3 as the adhesion force between silica particles (representing an oxide film) and ceria-coated silicon substrate (representing ceria particles) vanished in the presence of DADMAC in the liquid medium. When an attractive bond is present between two surfaces, the friction force is relatively larger due to the resistance of the bond to rupture in comparison to the case in which the two surfaces do not attract or repulse each other. The adsorbed DADMAC layer can also reduce the COF values considerably because it partially restricts the direct contact of surfaces or acts as a lubrication layer and eliminates the strong adhesion force between oxide and ceria abrasives (see Figure 3).

The interaction force-based technique described here can also be employed in characterizing removal mechanisms of various film and slurry combinations and to facilitate optimizing other formulations by identifying additives with characteristics similar to DADMAC used here that can reduce dishing during STI and, indeed, other CMP processes. Also, as listed in Table 1, it appears that the COF has a threshold value of ∼0.23-0.24 below which no removal of the oxide film (bold numbers in Table 1) occurs in this system. We have not investigated the implications of this specific COF value in this process and it will be interesting if a similar threshold for COF exists with other slurry and film combinations.

### Conclusions

A colloidal AFM-based method was used to measure the interaction forces between silicon dioxide, polyurethane IC1000 polishing pad and ceria abrasives in liquid media with and without DADMAC molecules. The results obtained were used to explain the observed non-Prestonian oxide removal reported earlier in Ref. 7. The AFM force measurements showed that there is a repulsive interaction between the IC1000 pad and oxide film surface at pH 4 with and without DADMAC additive indicating that the interaction of these surfaces has minimum effect in oxide removal. On the other hand, a strong adhesion force was measured between the ceria-coated substrates and silica in a solution with no DADMAC. It was concluded that this strong adhesion interaction between ceria and oxide film is responsible for the reported high removal rates and COF values in water adjusted to pH 4 with no DADMAC additive. Presence of DADMAC molecules eliminated the adhesion between ceria and silica surfaces by restricting the close-range electrostatic interaction between these surfaces and, in the process, reduced the removal rates and COF values. This effect increased when the DADMAC concentration was increased from...
15 mM to 25 mM. It appears that the positively charged DADMAC molecules create an adsorbed film on the silica surface that acts as a lubrication layer to reduce the COF and also as a restricting layer blocking the direct contact between the abrasives and the oxide surface, reducing the removal rate. Also, a threshold value of ∼0.23 to 0.24 was observed for COF while polishing oxide films below which there was no film removal.

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