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Performance Evaluation of Cleaning Solutions Enhanced with Tetraalkylammonium Hydroxide Substituents for Post-CMP Cleaning on Poly-Si Film

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The cleaning solutions augmented with tetraalkylammonium hydroxides (TAAHs) with various chain-lengths of hydrocarbon substituents were developed for post-poly-Si chemical mechanical polishing (CMP) cleaning. The cleaning performance with respect to particle, organic, and metal removal as well as surface roughness was evaluated for a series of 3% NH_4OH solutions dosed with 0.26 M of a TAAH and 100 ppm of ethylenediaminetetraacetic acid (EDTA). The experimental results demonstrated that the cleaning solutions enhanced with these surfactants (TAAH) and a chelating agent (EDTA) achieved significantly better removal efficiencies of particle and metal impurities than the control solution containing 3% NH_4OH only. A conceptual model involving surface adsorption and double-layer formation was used to postulate the aqueous-phase surface interactions between the tetraalkylammonium cations and the poly-Si surface, and to explain the removal mechanisms of particle and metal impurities from the surface. The improved electrical properties (current density-electric field and charge-to-breakdown characteristics) of the post-CMP capacitor after cleaning further demonstrated the reliability and feasibility of the proposed cleaning recipes.
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The increasing complexity and miniaturization of modern integrated circuits demand a high device yield, meaning that lower defect density in the active region of silicon devices is a necessity.[1,2] For instance, the reliability of nonvolatile memory devices such as EEPROM, EPROM (electrically erasable and erasable programmable read-only memory, respectively), and flash memory is strongly dependent on the polyoxide quality characterized by low leakage current and high breakdown electrical field to prolong their data retention capabilities. Inadequate control over the thickness uniformity and surface roughness of the poly-Si layer directly leads to defect formation on the poly-Si/polyoxide interface, thus severely degrading the electrical properties. In recent years, the chemical mechanical polishing (CMP) process has been widely accepted as the mainstream planarization technique in the fabrication of deep submicrometer integrated circuits[3] in light of its capability to reduce surface roughness. However, as the device's dimension continues to scale down, the requirement for post-CMP cleaning becomes increasingly more stringent to ensure high device yield.

The brush-scrubbing technique in combination with dilute ammonium hydroxide solution has been employed for many years and was considered among the most effective methods for removing particles after the CMP process.[4] Numerous studies focusing on improving oxide layer CMP as well as the post-CMP cleaning efficiency have been reported.[5-9] Jolley[7] reported that the solution containing tetramethylammonium hydroxide (TMAH) showed enhancement of metal removal, and had little effect on the surface roughness for post tungsten-CMP cleaning as compared to ammonium hydroxide solution. Cady and Varadarajan[8] also proposed that the alkaline solution containing TMAH could replace the traditional multisteps cleaning method. The important merits of their proposed cleaning methods are low water consumption, high throughput, and effective particle removal. Our previous study[9] also indicated that the NH_4OH solution spiked with TMAH and a chelating agent was more effective for metal removal. Nevertheless, despite the favorable results exhibited by the TMAH-containing solution for post-CMP cleaning, the possible surface interactions and cleaning mechanisms remain to be elucidated.

In comparison to post-oxide-CMP cleaning, information on poly-Si CMP and the subsequent cleaning methods is scarcely available. This is a particular concern because the CMP process is a proven technology to improve polyoxide quality by reducing the surface roughness of the deposited poly-Si film,[10] yet the post-CMP cleaning efficiency continues to be a problem. Pietsch *et al.*[11] offered an explanation by postulating that the hydrophobic Si-H bonding would remain on the poly-Si surface after polishing with alkaline slurry. As a consequence, both the abrasives and the metallic contamination on the hydrophobic poly-Si surface cannot be effectively removed by the alkaline cleaning solutions.

To overcome the difficulty of post-polishing particle removal from the hydrophobic poly-Si surface, it is preferable to operate the cleaning process under a slight etching condition of wafer surface.[12] In the present study, several tetraalkylammonium hydroxides (TAAHs) with various chain lengths of hydrocarbon substituents were added to the NH_4OH-based cleaning solution. These surfactants included TMAH, tetraethylammonium hydroxide (TEAH), tetrapropylammonium hydroxide (TPAH), and tetrabutylammonium hydroxide (TBAH). The aim of this study, therefore, is to investigate the influences of the added surfactants on the hydrophilicity and the etching rate of the poly-Si surface, as well as the possible interaction mechanism between TAAH cleaning solution and the poly-Si surface. The overall cleaning performance of the solutions can be evaluated by analyzing the particle, organic, and metallic impurities remaining on the surface after cleaning. Furthermore, measurement of the electrical properties of the capacitors can also provide valuable information to the specific roles of the various tetraalkylammonium-containing solutions during post-CMP cleaning of poly-Si surfaces.

Experimental

Materials and cleaning solutions.—p-Type silicon wafers (⟨100⟩; resistivity 15-25 Ω cm) of 15 cm diam were used for device fabrication and cleaning experiments in this study. The compositions of the water-based cleaning solutions are specified in Table I. With the exception of the control solution, all other solutions were dosed with a predetermined concentration of TMAH (Mw 91), TEAH (Mw 147), TPAH (Mw 203), and TBAH (Mw 259), and were spiked with ethylenediaminetetraacetic acid (EDTA, Mw 292) having four pK_a values (*i.e.*, 1.99, 2.67, 6.16, and 10.26).[13] All reagents used were of electronic or higher grades from Merck (Darmstadt, Germany).

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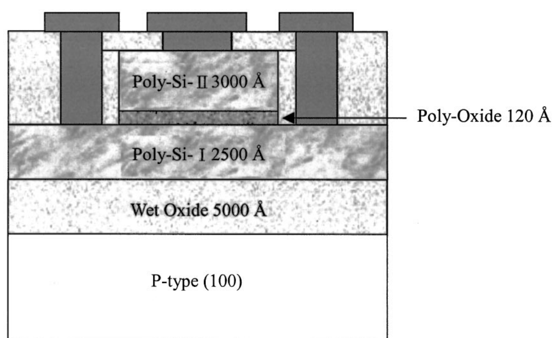
Table I. Various cleaning solutions used for post-CMP cleaning.

Solution	NH_4OH	Tetraalkylammonium (0.26 M)	EDTA	pH
A	3%	0	0	12.58
B	3%	2.38% TMAH	100 ppm	12.75
C	3%	3.84% TEAH	100 ppm	12.85
D	3%	5.31% TPAH	100 ppm	12.80
E	3%	6.77% TBAH	100 ppm	12.40

Capacitor fabrication and cleaning procedures.—Figure 1 describes the fabrication process, including the cleaning procedure, for a capacitor. A 5000 Å buffer oxide was thermally grown on the silicon substrate, and a poly-Si film of 3000 Å thickness (poly-Si-I) was subsequently deposited on the oxide in a low-pressure chemical vapor deposition (LPCVD) system. The poly-Si film was doped with $POCl_3$ at 875°C for 17 min, providing a resistivity of 40-80 Ω cm. The poly-Si film was then polished on a Westech polisher (model 372M) with diluted Cabot SC-1 slurry to remove 500 Å poly-Si. After the CMP process, the wafer was treated by spraying with 3% NH_4OH solution with 0.95 MHz megasonic, followed by dispensing the various cleaning solutions (Table I) with a PVA [poly (vinyl alcohol)] brush. The RCA cleaning was then performed to complete the cleaning operation. Afterward, an inter-polyoxide layer of 120 Å was deposited on the poly-Si-I by growing tetraethylorthosilicate (TEOS) in LPCVD, and the samples were annealed in a rapid thermal reactor (950°C, 30 s, in N_2 ambient). Subsequently, a poly-Si-II layer with 3000 Å was deposited and $POCl_3$-doped to 40-80 Ω cm resistivity. After the poly-Si-II layer was defined, an oxide was again thermally grown to 1000 Å by wet oxidation. Contact holes were defined and opened, and Al film was deposited and patterned. Finally, the wafers were sintered at 350°C for 30 min in N_2 ambient to complete the capacitor fabrication process.



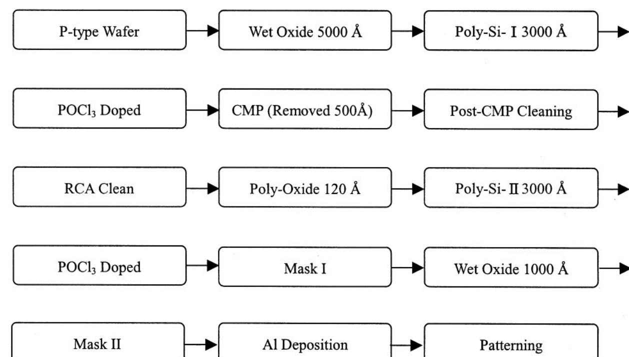


Figure 1. Capacitor structure and the fabrication steps for polyoxide capacitors using the CMP process.

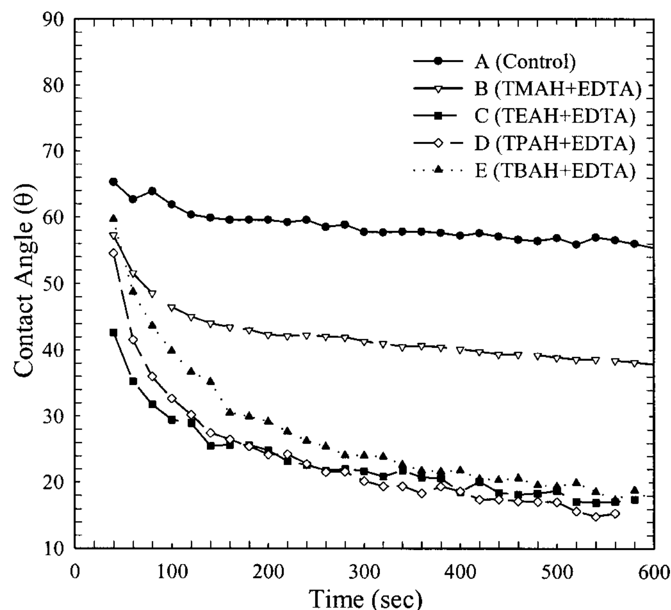


Figure 2. Contact angles for the cleaning solutions as a function of time.

Instrumental analysis and electrical characterization.—The cleaning performance of the wet cleaning recipes specified in Table I was evaluated by various surface analytical methods, including measurements of the contact angle and the trace impurities. The contact angles were measured by injecting one drop (0.01 mL) of cleaning solution onto the poly-Si surface, which had been previously treated with dilute HF solution to remove native oxide. Then the image of the liquid drop on the wafer surface was recorded and analyzed. The residual particle (>0.2 μm) after cleaning was counted by the Tencor Surfscan model 4500 system. The residual organics was determined with the Hitachi thermal desorption system (TDS) model UG-21 in conjunction with the atmospheric pressure ionization mass spectrometer (APIMS) model UG-400P. The desorption temperature of the TDS-APIMS was ramped from room temperature to 600°C at 10°C/min. The surface outgassing was analyzed at m/z 30, 44, and 58. The metallic impurities (Ca, Fe, Cu, Zn, and K) were determined using the Rigaku total reflection X-ray fluorescence spectrometer (TXRF, model 3700) with an incident angle of 0.07°. The detection limits are 5×10^{10}, 5×10^{9}, 5×10^{9}, 3×10^{9}, and 10^{11} for Ca, Fe, Cu, Zn, and K, respectively. The polyoxide thickness of the capacitor was obtained using a Keithley C-V (capacitance-voltage) system. The electrical properties of the polyoxide, including the current density-electric field (J-E) and the time-dependent dielectric breakdown (TDDB) characteristics, were also measured by using a Hewlett-Packard HP-4145B semiconductor parameter analyzer.

Results and Discussion

Effects of cleaning solutions on poly-Si surface characteristics.—The physical and chemical interactions between the poly-Si surface and the TAAH-containing solutions strongly dictate the post-CMP cleaning performance of the solutions. One of the most useful parameters characterizing the surface modification is the contact angle (θ) measurement. As shown in Fig. 2, the decrease in contact angle as a function of time is due to the (slow) reoxidation of the poly-Si surface by O_2 present in the different cleaning solutions. In addition, the control solution (solution A, containing 3% NH_4OH only) exhibited the largest contact angle. This result reflects the hydrophilic nature of solution A because the surface structure of the poly-Si layer is predominantly hydrophobic in Si-H bonding. The extent of hydrophilicity for solution B (with TMAH) laid between those of solution A and solutions C (with TEAH), D (with TPAH),

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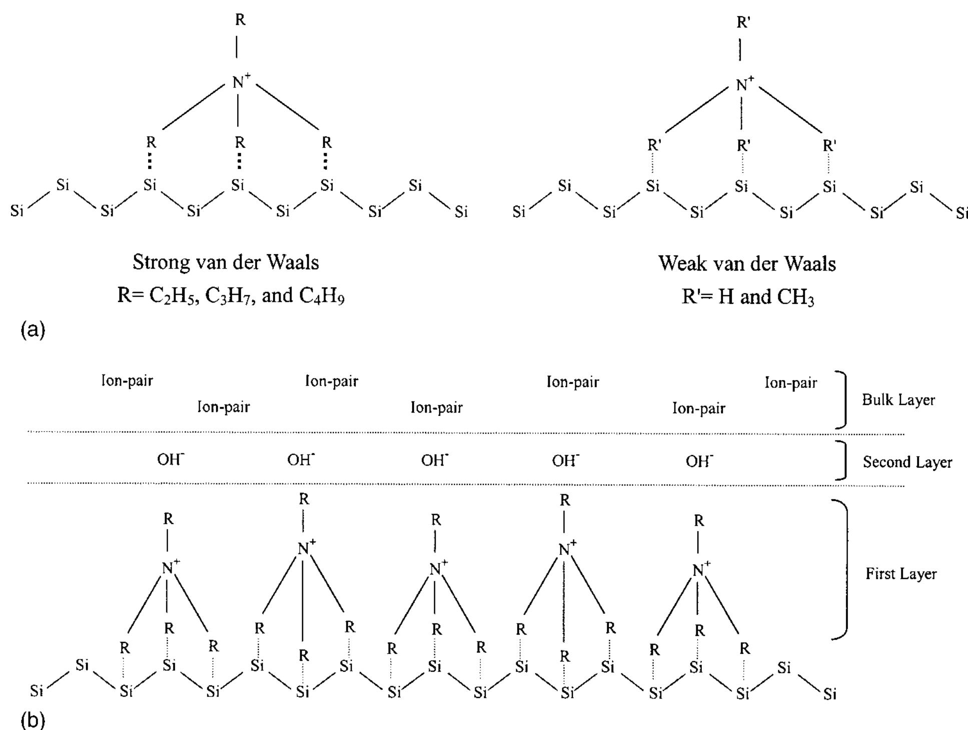

Figure 3. Surface adsorption model for (a) cleaning solutions and (b) double-layer formation.

and E (with TBAH), whose θ values significantly decreased and clustered closer to each other as time progressed. This phenomenon can be explained by the surface adsorption model schemed in Fig. 3a. This figure illustrates that the longer chain lengths of alkyl groups in solutions C, D, and E (corresponding to carbon numbers of 2, 3, and 4, respectively) tend to form stronger van der Waals attraction force with the poly-Si surface, whereas solutions A and B (carbon numbers 0 and 1, respectively) exhibit weaker interaction with the surface. By further adapting the Stern-Gouy-Chapman model[14] for surface adsorption and double-layer formation, we postulated that the tetraalkylammonium cations of TEAH, TPAH, and TBAH were first adsorbed onto the poly-Si surface. This positively charged layer is referred to as the "primary layer," as shown in Fig. 3b. The second layer, in opposition, was negatively charged with the anions (*e.g.*, hydroxide). Consequently, the ion pairs from TEAH, TPAH, and TBAH were formed in the bulk layer. This model is also useful to vindicate the origin of surface roughness for each cleaning solution, as is discussed later in the paper.

The physical and chemical properties of poly-Si after CMP and cleaning.—The particle number and surface roughness were characterized for the poly-Si surface after the post-CMP cleaning process. Figure 4 presents the results of residual particle numbers after cleaning with each type of solution. Evidently, the TAAH-containing solutions manifested much improved particle removal capability as compared to the solution without TAAH (*i.e.*, solution A). In an attempt to justify these results, we hypothesized the possible surface interaction with two types of particles having either hydrophilic or hydrophobic nature, as illustrated in Fig. 5. It has been previously documented[15] that the zeta potentials of particle and poly-Si surfaces are both negative at pH $\geqslant$12, a condition normally attained by solutions containing TMAH. As a result, the hydrophobic particle shows stronger interaction with the hydrophobicity of the poly-Si surface than the hydrophilic particle, and is more difficult to remove from the surface. From this perspective, the TAAH components in solutions B, C, D, and E become critical to the removal of hydrophobic particles by penetrating into the particle-surface interfacial region. As mentioned earlier in Fig. 3, the tetraalkylammonium ions are effectively adsorbed onto the poly-Si surface as well as the particle surface. This surface adsorption phenomenon facilitates particle removal from the poly-Si surface due to the reduction of interfacial attraction. On the contrary, the NH_4OH in solution A hardly penetrates into the particle-surface interfacial region because of its hydrophilic characteristics, and hence could not remove particles (particularly of hydrophobic nature) as effectively.

In order to satisfy the total cleaning criteria of the TAAH-containing solutions, the residual organic and metallic impurities were also analyzed with TDS-APIMS and TXRF techniques, respectively. The results indicated that the peak intensities of residual C_2H_6, C_3H_8, and C_4H_{10} after cleaning with solutions B, C, D, and E were in the range of one- to twofold higher than those cleaned with solution A. However, the differences of these peak intensities became insignificant when RCA cleaning was performed following

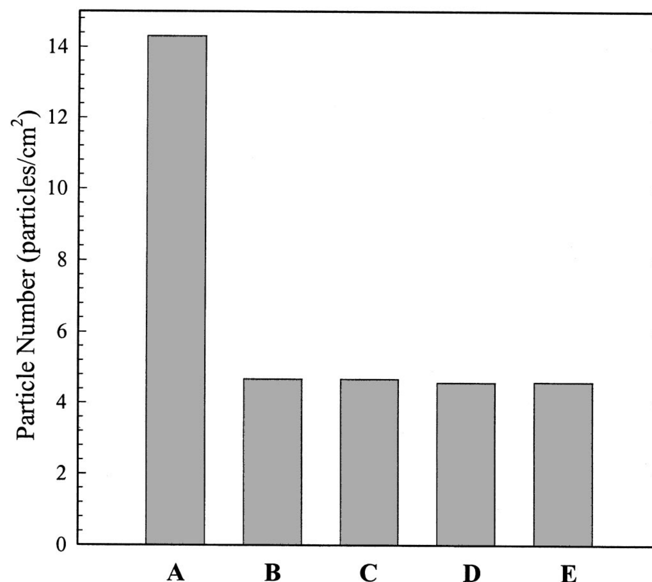

Figure 4. Number of particles remaining on the polished poly-Si surface after cleaning with various solutions.

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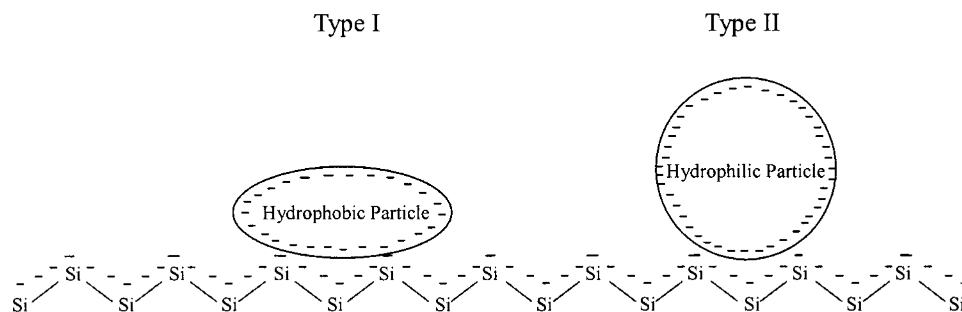


Figure 5. Schematic representation of surface adsorption phenomenon of hydrophobic and hydrophilic particles on poly-Si surface.

the post-CMP cleaning with the various TAAH solutions. Furthermore, no deterioration in the electrical properties was observed for the capacitors at the level of residual organics. With regard to the control of metallic contamination, the 1999 International Technology Roadmap for Semiconductors (ITRS) specifically recommended that the control limit for surface metal should be at least four orders of magnitude more critical than that for organic contamination. In this study, the metallic impurity on the poly-Si surface was below the TXRF detection limit for all cleaning solutions except solution A. The metal levels detected on poly-Si using cleaning solution A are approximately 10^{13} atoms/cm^2. It was found that the metal cation and the adsorbed tetraalkylammonium cation were of the same charge type; hence, the electrostatic repulsive force helped the removal of metal ions. However, the electrostatic interaction was not sufficient to explain the removal of metal oxides (*e.g.*, chromium oxide, iron oxide). The more plausible removal mechanism can be ascribed to the presence of chelating agent in the cleaning solution, namely the EDTA (100 ppm). Under alkaline conditions (pH > 12), the hydrogen ion was fully dissociated from EDTA due to its pK_a value ($pK_4 = 10.26$). As a result, the EDTA in the solutions formed a hexadentate coordination with the surface metal, thereby efficiently removing them from the poly-Si surface. This result was consistent with our previous examination[9] that spiking with low dosage of EDTA in cleaning solutions was beneficial for metal removal.

Yet another important factor influencing the device yield is the surface roughness. Tardif and co-workers[16] have reported that the charge-to-breakdown of their capacitor increased initially with the dipping time in hydrofluoric acid (HF) solution due to improved surface suitability; however, it gradually degraded because of the increase in surface roughness caused by excessively long dipping time. Therefore, it is imperative to evaluate the potential effect of the TAAH solutions on surface roughness. Figure 6 presents the results of the poly-Si surface roughness measurement as well as the

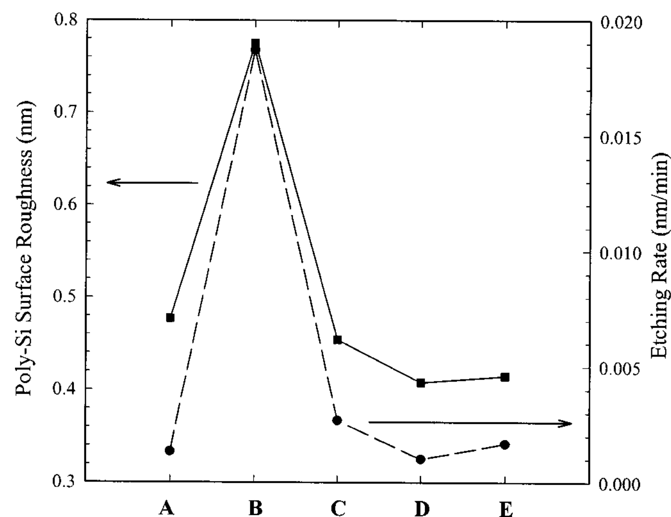
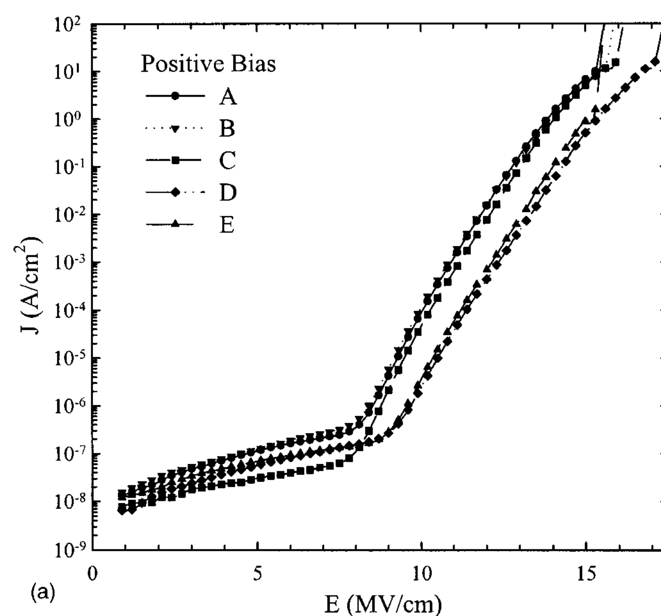


Figure 6. The poly-Si surface roughness and etching rate after post-CMP cleaning with various solutions.



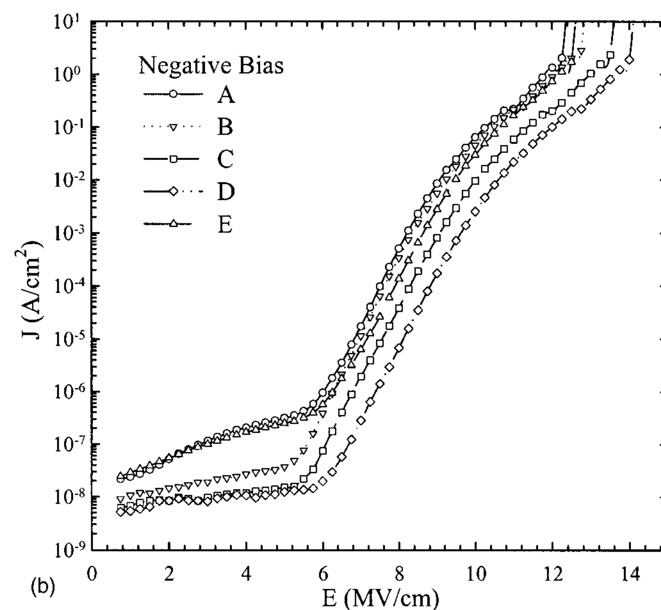


Figure 7. The J-E characteristics for the top gate applied with (a) positive and (b) negative bias using different solutions for post-CMP cleaning.

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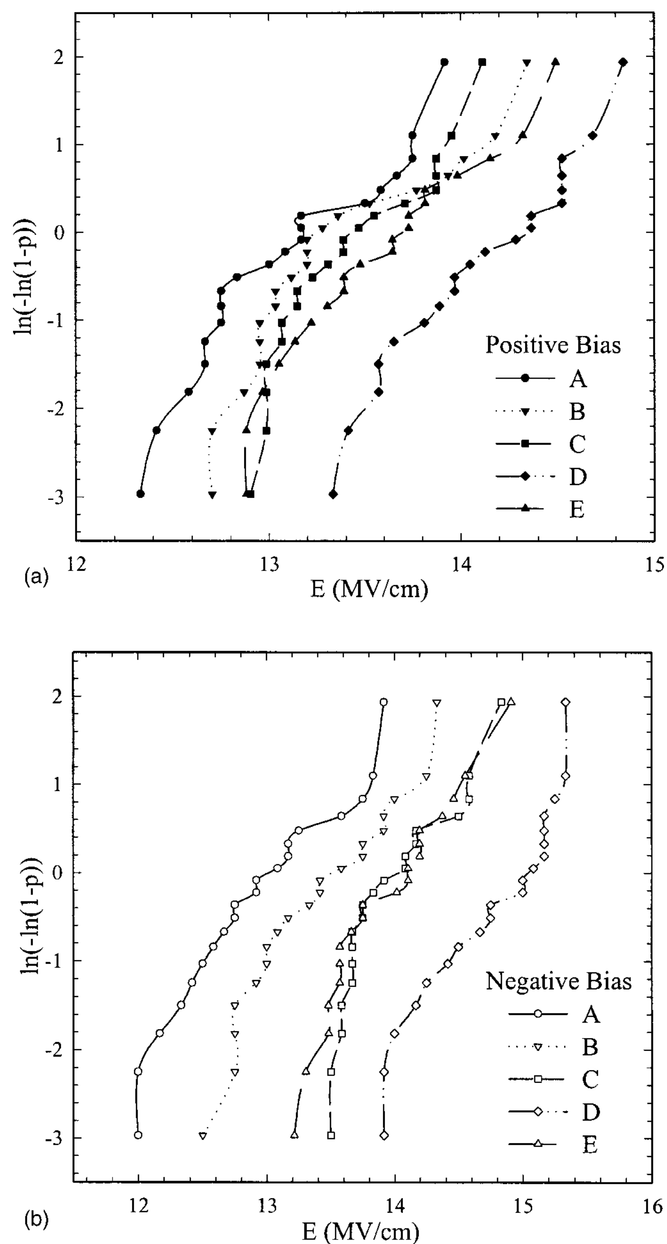

Figure 8. The breakdown field distribution for the top gate applied with (a) positive and (b) negative bias using different solutions for post-CMP cleaning.

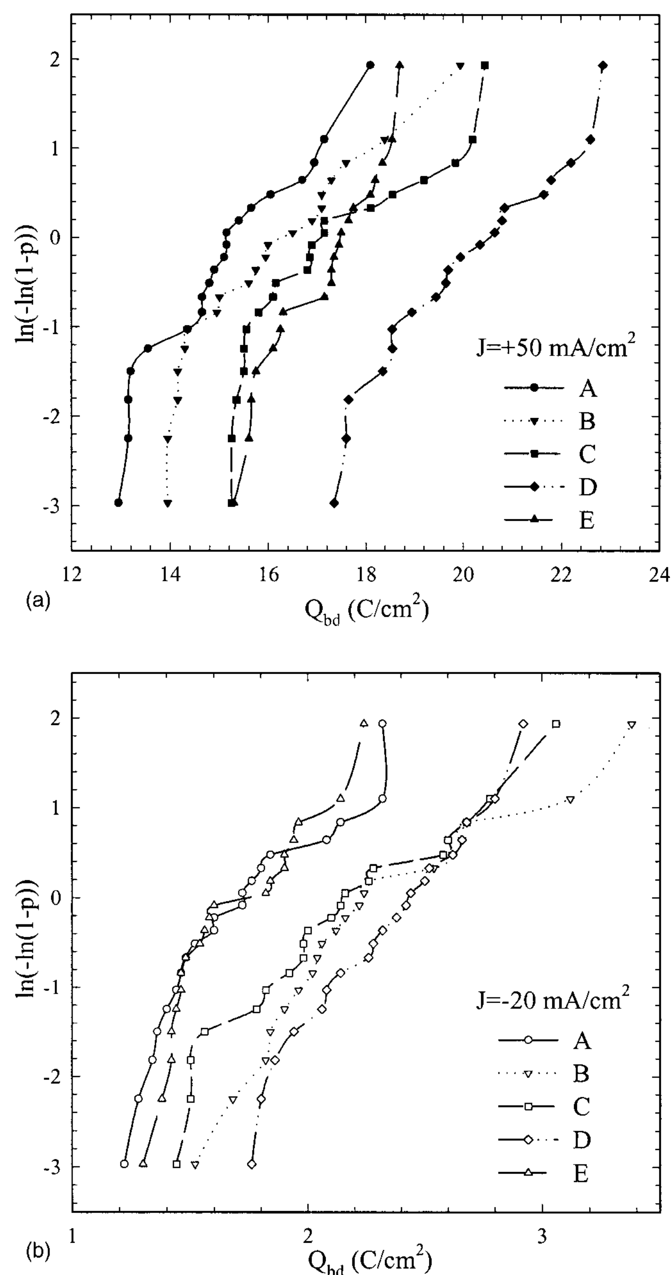

Figure 9. The charge-to-breakdown (Q_{bd}) characteristics under (a) positive and (b) negative constant current density stress.

wet etching rate for the cleaning solutions. It was found that the surface roughness exhibited strong correlation with the etch rate, suggesting that the degree of roughness formation was primarily dictated by etching rate. This figure also indicates that the TMAH-containing solution (solution B) had appreciably higher etching rate than the others. This result can be attributed to the strong adsorption of tetraalkylammonium cations of the long-chain TAAHs (*e.g.*, TEAH, TPAH, and TBAH) in the "primary" layer, as illustrated in Fig. 3, preventing direct etching of the surface by the hydroxides existing in the second and bulk layers. In contrast, the cations in the TMAH-containing solution only weakly interacted with the poly-Si surface, so the wet etching by the hydroxide occurred much more readily. Furthermore, the lower etching rate of solution A was due to the highly hydrophilic nature of NH_4OH relative to the hydrophobic poly-Si surface. Consequently, the NH_4OH (or hydroxide) could not effectively approach the surface, resulting in a lower etching rate than solution B.

Electrical properties of capacitors after cleaning.—Figure 7a and b shows the typical J-E characteristics under positive and negative biases, respectively, for the capacitors cleaned with the various TAAH solutions. The electric field (E) was obtained by $E = V/T$, where V is the applied voltage and T represents the effective oxide thickness determined by C-V measurement. The results show that the capacitors cleaned with solutions C and D generally demonstrated higher electric breakdown field and lower leakage current than those cleaned by other solutions for either positive or negative bias. Further investigation of the breakdown field distribution (Fig. 8) indicates that the polished poly-Si-I film cleaned with solution D showed the highest positive and negative electric breakdown fields than the others. This result was an apparent consequence of the favorable performance by solution D with respect to low degree of poly-Si surface roughness and effective particle removal.

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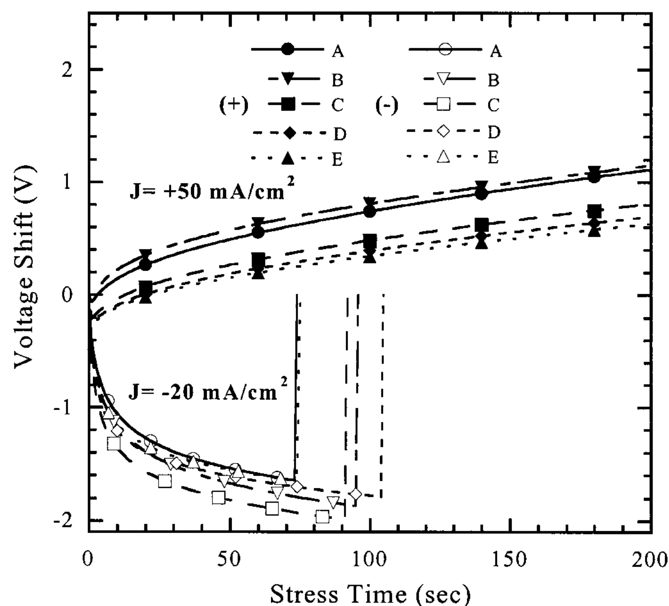

Figure 10. The gate voltage shifts *vs.* time for post-CMP capacitors cleaned with different solutions.

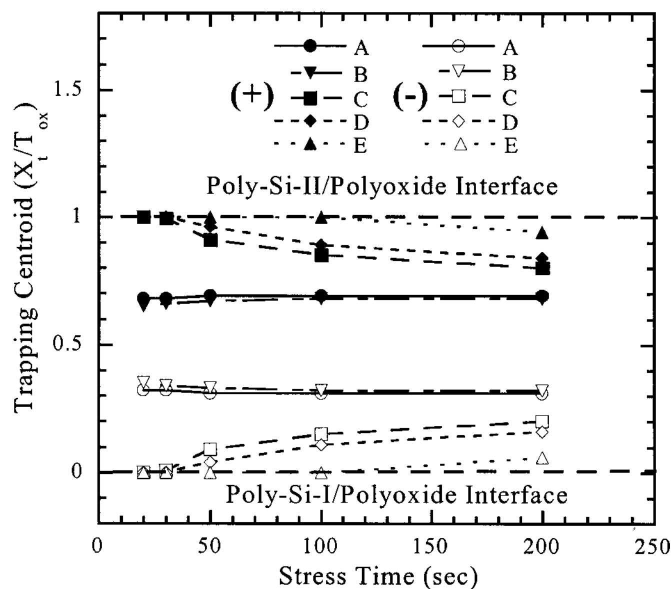

Figure 11. The centroids of trapped charges (X_t) of different solutions for post-CMP cleaning at various $+V_g$ and $-V_g$ injection times.

To investigate the polyoxide reliability, charge-to-breakdown (Q_{bd}) measurements were performed on the capacitors. In Fig. 9a and b the Weibull distributions are shown under positive and negative constant current density at +50 and −20 mA/cm^2 stress, respectively. The distribution for the post-CMP capacitor cleaned with solution D showed a slightly higher Q_{bd} due to higher particle removal efficiency and a smoother poly-Si surface in the polyoxide. It is clear that the capacitor with polished poly-Si-I had a higher Q_{bd} than unpolished poly-Si-II. This observation indicates that polished poly-Si-I was a smoother poly-Si-I/polyoxide interface and that the unpolished poly-Si-II was a rougher polyoxide/poly-Si-II interface. Figure 10 depicts the charge-trapping characteristics of the polished poly-Si films cleaned with various solutions. Obviously, the gate voltage shift of these solutions increased with time at $+V_g$ and $-V_g$ constant current stress. These figures also reveal that capacitors with solution D cleaned after the CMP process exhibited a smaller voltage shift than those cleaned with the other solutions. This improvement implies that the capacitor traps fewer electrons and has a lower electron-trapping rate than others, and that unpolished poly-Si-II tends to have a higher electron trapping rate than polished poly-Si-I. Furthermore, the rougher polyoxide/poly-Si-II interface leads to a smaller conduction area and a higher local current density, subsequently leading to a higher electron trapping rate.

A previous work has mentioned the centroids of trapped charge (X_t) in the polyoxides.[17] By the bidirectional I-V measurement and by the shifts of Fowler-Nordheim (F-N) I-V characteristics before and after stress for both polarities, the centroids of the trapped charges are calculated from the following relationship

$$X_t = T_{ox}[\Delta V_{g^+}/(\Delta V_{g^+} + \Delta V_{g^-})] \quad [1]$$

where X_t is measured from the polyoxide/poly-Si-II interface, ΔV_{g^+} denotes the voltage shift when poly-Si-II is positively biased, ΔV_{g^-} represents the voltage shift when poly-Si-II is negatively biased, and T_{ox} is the polyoxide thickness.

Figure 11 presents the centroid of trapped charges (X_t) at various $+V_g$ and $-V_g$ injection times for these solutions. The X_t of capacitors using cleaning solutions C, D, and E for polished poly-Si film appeared closer to polyoxide/poly-Si-II interface than those using cleaning solutions A and B. This phenomenon is due to the fact that the surface morphology of using cleaning solution B for poly-Si film after the CMP process was much rougher than that of other solutions; therefore, centroids moved away from the polyoxide/poly-Si-II interface.

Conclusion

The cleaning performance of the various TAAH-containing solutions and the effects of hydrocarbon chainlength on the tetraalkylammonium cations were investigated in this study. It was found that overall cleaning efficiency with respect to particle, organic, metal, and surface roughness could be significantly improved over the use of conventional NH_4OH solution. These improvements were mainly due to surface modification by adsorption of tetraalkylammonium ions that facilitated removal of microcontaminants. The chelating agent (EDTA) in the solution was also instrumental in the removal of surface metals. Electrical characterization of the post-CMP capacitors cleaned with the TAAH-containing solutions further confirmed their improved cleaning performance. The results indicated that the solution containing TPAH and EDTA was the foremost choice among the TAAH solutions based on the overall cleaning efficiency as well as the electrical properties of the capacitor.

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