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Applied Surface Science 92 (1996) 176–179

applied surface science

Post cleaning of chemical mechanical polishing process

Chi-Wen Liu [a], Bau-Tong Dai [b,*], Ching-Fa Yeh [a]

[a] *Department of Electronics Engineering & Institute of Electronics, National Chiao Tung University, 1001 Ta Hsueh Road,*
Hsinchu 30050, Taiwan, ROC
[b] *National Nano Device Laboratory, 1001-1 Ta Hsueh Road,*
Hsinchu 30050, Taiwan, ROC

Received 12 December 1994; accepted for publication 29 May 1995

Abstract

We describe a study on the effect of the electrostatic nature in silica particles on the post CMP cleaning behavior. A fall-off for the zeta potential of silica particles is observed as the pH of dip solutions is increased. In this study, we also observed that particle counts on the SiO_2 and the Si_3N_4 dielectric films had a similar dependence on the pH. Furthermore, we confirmed that surface hardness of the wafer is an important factor for particles physically embedded in different dielectric materials during and after the CMP process. The nanoscale surface hardness of dielectric films was measured by the nanoindentation technique. Experimental results showed that particles had difficulty attaching to a harder surface of the dielectric film.

1. Introduction

Since integrated circuit devices continue to be scaled down, global planarization is demanded as chip designs include multi-level interconnections with decreasing photolithographic depth of field. Traditional planarization methods such as SOG and etch back, planarize over a distance in a few microns range. Currently, chemical–mechanical polishing (CMP) is the only method available that planarizes effectively at local and global range. Therefore, CMP has become an important technology for inter-level dielectric and shallow trench isolation planarization [1,2].

The polishing process involves intimate contact between the wafer surface and the pad material in the presence of slurry. The colloidal debris from slurry will be left on the wafer surface after polishing. These residual silica will contaminate the subsequent processing steps and cause functional defects and lower yield in the finished integrated circuit. Therefore, an effective wafer surface cleaning is important for transferring CMP process from R&D labs to manufacture sites directly.

The particles attached on the wafer surface come from both chemical and physical procedures. In the CMP process, silica particles with sufficient mass and energy can penetrate into the surface of the polished film, resulting in abrasion. As the carrier and wafer subsystem lift up and leave the pad, those silica particles which have penetrated into the polished film but not removed in-situ during CMP

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[*] Corresponding author. Tel.: +886-35-726100 Ext. 7719; fax: +886-35-713403.

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SSDI 0169-4332(95)00226-X

process will be embedded in the polished film. Since the hardness is determined by the depth of penetration of an indenter forced (in our case, silica particles) into a surface [3], the amounts of the silica particles embedded in polished films would vary with surface hardness. On the other hand, since this slurry is a suspension of fused silica dispersed in aqueous potassium hydroxide, the electrostatic forces, i.e. particle zeta potential, will control the particle deposition [4]. Kern [5] reported that colloidal deposition on a hydrophilic wafer surface exhibits a dependence on the pH similar to the zeta potential. Thus, silica particles adhered to the polished film would depend on the pH of the dip solution. However, there was still a lack of experimental data to verify this point for the slurry used in the CMP process. In this article we reported on a comprehensive characterization of particle removal via in-situ dipping process which was performed with solutions of various pH values. We also demonstrated that hardness of the polished film was an essential factor in the post CMP cleaning behavior.

2. Experimental procedure

2.1. Sample preparation

All test samples in this study were prepared on p-type (100), 150 mm silicon wafers. Thermally grown silicon dioxide films were processed by wet oxidation (ASM/LB45 furnace system), in which the silicon was exposed to the ambient H_2, O_2 at 980°C. Si_3N_4 film was deposited by the interactions of NH_3, N_2, N_2O and SiH_2Cl_2 at 780°C (Vertex, DJ-802V, Kokusai Electric).

2.2. CMP process

A Westech Model 372M CMP System, consisting of a IC 1000/Suba IV (made of polyurethane impregnated polyester) pad affixed to a circular polishing platen and a carrier to hold wafers against the pad, was used for polishing. During the polishing operation, both platen and carrier were rotated at 20 and 42 rpm, respectively. We pressed the wafer in the carrier against the platen with a force of 7 psi, and the back pressure against the wafer was 2 psi. After the abrasive process, the wafer and carrier were submerged in a tub of deionized water immediately, and then the wafer was transported by the carrier to unloaded cassette.

Cab-O-Sperse SC-1 slurry (a common type, available from Rippey Corporation) was used for polishing. SC-1 is a suspension of fused silica dispersed in aqueous potassium hydroxide. The number of particles (≥ 0.2 μm) on a wafer were counted with a Surface Scan (TENCOR-4500).

2.3. Zeta potential measurement

In a colloidal dispersion of particles there will generally be a net charge at the particle–liquid interface. The existence of this surface charge gives rise to the formation of the zeta potential [5,6]. The values of zeta potentials for silica particles were calculated by the Smoluchowski equation [6] using the mobility measurements (Zeta-meter system 3.0, Zeta-Meter, Inc.).

2.4. Nanoindentation measurement

An indentation system (Nanotest 500, Micro Materials, Inc.) with a Berkovich indenter was used to measure the nanoscale mechanical properties of dielectric films. All tests were performed at a nominal constant loading rate of 1.18 mN/s of the maximum set displacement (300 nm). For each sample, 10 separate indents, spaced 15 μm from each other, were made on the surface. At each load, the load versus displacement curve was recorded, from which hardness can be calculated using the standard formula [7].

3. Results and discussions

As it is well known, the zeta potential can be altered by adjusting the pH. In our experiment, the pH of the slurry was adjusted with HNO_3 to various values, which were measured (with a precision of ± 0.01) by a microprocessor pH meter (SP 2200, Suntex). Fig. 1 shows the change of the zeta potential with the pH in SC-1 slurry. Also included is the zeta potential on SiO_2 and Si_3N_4 film surface by streaming potential measurements proposed by

Bousse and coworkers [8,9]. The result shows that raising the pH will decrease the value of the zeta potential of the silica particles. The same tendency exhibits on the surface of the dielectric films [8,9]. The fact that both silica particles and film surfaces in liquids become electrically charged implies that electrostatic forces can affect the deposition process.

In the post CMP cleaning test, adjustment of the dip solution pH was carried out by adding NH_4OH and HCl to deionized water. Particle counts on the SiO_2 and the Si_3N_4 films immersed in various pH solutions for 30 min are shown in Fig. 2. A decrease in particle counts is observed as the pH value is increased except for the case of pH less than 7. The pH dependence of particle counts is very similar to the situation in the zeta potential of silica particles. Because adsorbed monomeric silica or colloid silica on the wafer may gel if it is exposed to a solution of pH 7 or less [10], the silica particles of solution pH 6 in our study would be aggregated, hence, particle counts were less than that of the dip solution with pH 7. The solubility of silica as a function of pH is given in Ref. [11] in which it was found that the solubility of silica increases when pH is greater than 8. These silica particles exist in the basic solution in the monomeric form, primarily as $Si(OH)_4$. Consequently, with increasing pH, more particles on the film surface would be desorbed into the solution resulting in lower particle counts.

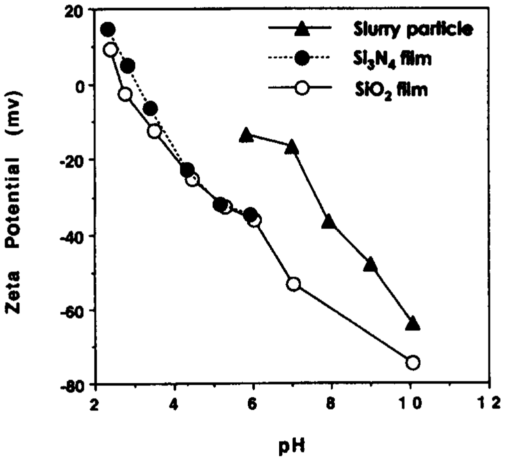

Fig. 1. Change of zeta potential with pH in SC-1 slurry, SiO_2 [8] and Si_3N_4 [9] film.

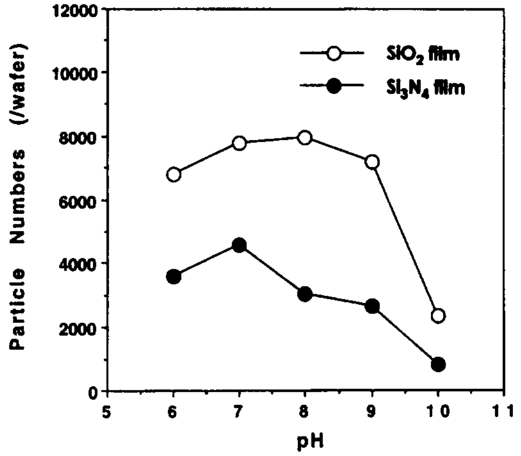

Fig. 2. Particle numbers on SiO_2 and Si_3N_4 films immersed in various pH solutions for 30 min after CMP process.

The preceding results can also be perceived as follows [12]: (i) The fresh surface is obtained just after the CMP process is finished. Thus, there is an attraction between particles with negative zeta potential close to the surface and positive ions present in the oxide or bulk of the silicon substrate. This results in a quick particle deposition. (ii) As the wafer is transported to a tank with an alkali solution, the fresh surface gradually disappears and a negative charge (negative zeta potential) is built up on the wafer surface by OH^- radicals adsorbed on the surface. So the repulsive forces dominate the interaction between the particle and the surface, leading to weaker contact between the two, and chemisorbed silica particles were converted to water soluble residue, which is aqueously removed. This phenomenon will become more pronounced when the numbers of OH^- radicals or pH value of the dip solution increase.

The zeta potential data in Fig. 1 revealed that Si_3N_4 and SiO_2 films have similar global chemical nature, conforming to results from other studies [8,9]. Nevertheless, the deposited silica particle counts of Si_3N_4 are much less than that of SiO_2 as can be seen in Fig. 2. This implies that contributors other than the electrostatic forces are also responsible for particle deposition or removal in the post-cleaning process.

In the CMP process, silica particles are fed into a gap between pad surface and polished film. Kragelsky et al. [3] proposed the probability of adherence of particles to pad surface (hardness $H1$) and polished film (hardness $H2$) are

$$\Pr(\text{pad surface}) = \frac{H2}{H1 + H2},$$

$$\Pr(\text{polished film}) = \frac{H1}{H1 + H2}.$$

Since $H1 \ll H2$

$$\Pr(\text{polished film}) \approx \frac{H1}{H2}.$$

This suggests that the number of silica particles physically embedded in the wafer surface is inversely proportional to the hardness of the polished film, i.e. particles are more difficult to adhere to, or embedded in, the harder surface. Since the hardness of the SiO_2(36 GPa) film is less than that of the Si_3N_4 film (50 GPa), there are more silica particles counted in the SiO_2 film than in the Si_3N_4 film. Most of these debris can be removed via scrubbing process.

4. Conclusion

This study was undertaken to investigate the mechanism of the particle removal in a dip solution after CMP process. The zeta potential of silica particles in solutions with various pH values was determined. Experimental results showed that both the zeta potential of silica particles and silica particle counts decrease as the solution pH increases. This means that the zeta potential is one determining factor for particle deposition after CMP process.

From a statistical viewpoint, particle adherence on the wafer surface is inversely proportional to the surface hardness of the polished film, therefore, more silica particles were detected on the softer film (SiO_2) than on the harder film (Si_3N_4).

Acknowledgements

The authors express appreciation for helpful discussions with Dr. T.Y. Yen. This work is supported by National Science Council (NSC84-2622-E009-007). We thank the Precision Instrument Development Center for providing the Nanotest 500 used for these experiments.

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