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Experimental Investigation of Material Removal Characteristics in Silicon Chemical Mechanical Polishing

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1. Introduction

Chemical mechanical polishing (CMP) is an essential process in manufacturing a silicon wafer substrate with a smooth surface and nanoscale roughness.¹) The CMP process is a typical tribological process because the contact pressure and the relative motion among the loaded silicon wafer due to Si–Si bond breaking during polishing. The abrasive particles in the slurry easily remove the reacted silicon surface, and the removal rate and wafer non-uniformity for abrasive concentrations of 1.5–3 wt% are better than those for other concentrations because of the low and steady coefficient of friction (COF) owing to the evenness of abrasive particles between the wafer and pad. Also, it was found that a high slurry flow rate of 700–1000 cm/min improves wafer non-uniformity owing to the reduced temporal variation of temperature, because the slurry acts as a good cooling source during polishing. However, the removal rate remains almost constant upon varying the slurry flow rate because of the effective dissolution characteristic of the slurry with abundant amine as an accelerator, regardless of the reduction of average temperature with increasing slurry flow rate. In the break-in process used to stabilize the material removal, the viscoelastic behaviors of the pad and the ground wafer surface with native oxide and wheel marks cause a temporal change of the friction force during polishing, which is related to the removal rate and wafer non-uniformity. As a result, the stabilization of removal rate and wafer non-uniformity is achieved through a steady-state process with elevated temperature and reduced COF after a total polishing time of 60 min, based on the removal process of the wafer surface and the permanent deformation in the viscoelastic behavior of the pad. © 2009 The Japan Society of Applied Physics

2. Experiments

2.1 Tests for analysis of wafer surface

A 4-in. silicon wafer was prepared to investigate the effect of the chemical composition of a silicon surface on material removal with a silicon-polishing slurry. All the experiments utilized the slurry, which was based on an alkaline solution with colloidal silica abrasive particles and an amine (NH₃) additive. On the basis of the Si and SiO₂ peaks obtained using an X-ray photoelectron spectroscopy (XPS; Scientics ESCALB 250), a dipping test was performed as follows. An additional wafer with a 1 μm thermal oxide film and a buffered oxide etching (BOE) solution, which can uniformly dissolve the SiO₂ material, were prepared, and the weight of the oxide film was measured using a precision balance (0.1 mg resolution, Ohaus). First, the weight of the 4-in. silicon wafer was measured after it was dipped in the BOE solution for 1 h, and was compared with that of a silicon wafer dipped in the alkali-based slurry without the treatment of BOE solution. Next the weight of the silicon wafer was measured after dipping it in slurry of temperatures 23 and 38 °C with the treatment of BOE solution.

The material removal characteristics of a silicon wafer were experimentally investigated with respect to the chemical dissolution and mechanical abrasion of the wafer during silicon chemical mechanical polishing (CMP) using an alkali-based slurry. The silicon surface without native oxide is rapidly dissolved by the slurry containing an amine agent, which effectively leads to the reduced hardness of the ground surface on test conditions.

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a nanoindentation triboscope (Hysitron) to investigate the mechanical property of the silicon wafer in the slurry. The samples measured were an as-received sample, a sample dipped in slurry, and a sample loaded against a pad with slurry. The sample surface was cleaned using a poly(vinyl alcohol) (PVA) brush and deionized water in order to prevent silica particles on the silicon surface from affecting the precision of the nanohardness measurement. In addition, the surface roughness of the silicon wafer was measured by a noncontact surface profiler (Nanosystem Nanoview).

2.2 Polishing tests
To investigate the effect of the abrasive concentration on material removal, an 8-in. ground wafer was polished using a felt-type polyurethane pad with different abrasive concentrations of the slurry. The slurry with 28 wt% abrasive concentration was diluted from 7 to 0.5 wt% abrasive concentration by adding deionized water and 5 wt% NH$_4$OH solution. Figure 1 shows a schematic of the CMP polisher (G&P Technology POLI-500). The friction force was also monitored during polishing to analyze the material removal characteristics. The pressure and rotational velocity were 250 g/cm$^2$ and 70 rpm, respectively. The slurry flow rate was kept constant at 700 cm$^3$/min. The removal rate was calculated from thickness measurements (ADE 9500 Ultragage).

In addition, the material removal characteristics were investigated using slurry of 2 wt% abrasive concentration at slurry flow rates of 100, 300, 500, 700, and 1000 cm$^3$/min. The process temperature on the pad was monitored by an IR camera (Thermo Tracer TH 9100WL).

Finally, the break-in characteristic was investigated using nine wafers while they were polished using a new felt-type pad. The pressure, rotational velocity, and slurry flow rate were 250 g/cm$^2$, 70 rpm, and 700 cm$^3$/min, respectively. The friction force and temperature on the pad were also measured in this experiment. The viscoelastic behavior of the pad was measured using a viscoelastic measurement system (VMS; G&P Technology), which can monitor the change of pad thickness through a dial gauge (1 μm resolution), to determine its relationship with loading and unloading time, and then the strain of the pad was analyzed. A pressure of 250 g/cm$^2$ was applied to the pad sample ($40 \times 40$ mm$^2$) for 30 s (loading time) and then released for 30 s (unloading time).

3. Results and Discussion
3.1 Silicon wafer surface in alkali-based slurry
Figure 2 shows the XPS narrow-scan spectrum of the silicon wafer. The binding energies of 98.9, 102.9, and 103.4 eV correspond to Si 2p, SiO$_2$, and SiO$_2$–nH$_2$O, respectively. The peak at the binding energy of 98.9 eV was observed because the wafer material was silicon. A small shoulder corresponding to SiO$_2$ appeared at the binding energy of 102.9 eV, and the peak at the binding energy of 103.4 eV was assigned to SiO$_2$–nH$_2$O. Thus, the surface of the Si wafer may have a native oxide (SiO$_2$) layer during the manufacturing of the silicon wafer.

Figure 3 shows the weight loss of the silicon wafer in the silicon CMP slurry. The results were used to investigate the effect of the native oxide on material removal in Si polishing. A native oxide layer was assumed to exist on the
Si surface; thus, BOE solution was used for stripping the oxide layer. “A” shows that the thermal oxide film on the 4-in. Si wafer was dissolved by dipping the wafer in BOE solution. The weight of the 1-μm-thick oxide film was about 34.6 mg. “B” shows that there was very little weight loss of the 4-in. polished wafer (about 0.5 mg) during 1 h of dipping the wafer in BOE solution. This low weight loss was because the native oxide layer on the Si wafer was dissolved by the BOE solution. “C” shows that there was no weight loss of the Si wafer after dipping the wafer in the alkali-based slurry with amine, because the native oxide on the Si surface prevented the silicon from dissolving in the slurry.17) “D” and “E” show that the Si wafer dissolved in slurry after the native oxide was removed by the BOE solution. “C” shows that there was no weight loss of the Si wafer after dipping the wafer in the alkali-based slurry with amine, because the native oxide on the Si surface prevented the silicon from dissolving in the slurry.17) “D” and “E” show that the Si wafer dissolved in slurry after the native oxide was removed by the BOE solution. The weight losses were 14.1 and 35.5 mg for the Si samples dipped in slurry of 23 and 38 °C, respectively. Therefore, the silicon was dissolved by the alkali-based slurry after the native oxide was removed during polishing. The dissolution rate of the silicon wafer during polishing also depends on the slurry temperature.18)

The silicon wafer is polished by the applied load and the sliding motion among the wafer, slurry, and pad. Thus, the action of the loaded wafer against the pad surface can have an effect on the chemical reaction distribution and the mechanical property of the silicon surface as well as the dissolution characteristic of the silicon wafer in an alkali-based slurry.7,12,13) Figure 4 shows the hardness of an as-received sample, a sample dipped in slurry for 1 h, and a sample loaded against the pad surface with slurry for 1 h. The average hardnesses of the as-received sample, dipped sample, and loaded sample were 13.12, 12.53, and 11.38 GPa, respectively. The slurry altered the chemical molecular structure of the silicon surface, and consequently led to the slight reduction of hardness measured by nanoindentation. This reduction is due to the weakening of the Si–Si backbond by the formation of the silanol group.7,12,13) In particular, the hardness of the loaded sample was lowest because a uniform chemical reaction occurred on the silicon surface in the contact area between the wafer and pad owing to the pressure applied at the surface of the loaded sample. In addition, Fig. 5 shows the roughness and surface images of the as-received sample, dipped sample, and loaded sample. As shown by the results of roughness [Fig. 5(a)], the initial roughness of 3 nm increased to 82 nm in the sample dipped in slurry, and also its deviation increased owing to the static etching of the silicon surface by the slurry. However, the roughness of the loaded sample increased only slightly to 27 nm and its deviation was lower because of the uniform

![Fig. 4. Hardness of as-received sample, dipped sample, and loaded sample.](image1)

![Fig. 5. (Color online) Roughness and surface images of as-received sample, dipped sample, and loaded sample: (a) roughness and (b) surface image.](image2)
dissolution in the contact area between the wafer and pad. The surface images show the different features of the silicon surface between the dipped sample and loaded sample [Fig. 5(b)]. In the case of the dipped sample, the silicon was non-uniformly dissolved across the wafer surface. A difference in the microtopography of the surface of the loaded sample was also observed because of the difference of the silicon dissolution rate between the contact area and non-contact area of the pad. Therefore, it is predicted that the loaded wafer can be easily removed by the wear behavior of abrasive particles and pad asperities at the interface between the wafer and pad during polishing because of the uniform reduction of hardness with silicon dissolution.

3.2 Dependence of material removal rate on slurry supply

Figure 6(a) shows the material removal rate with increasing abrasive concentration. The material removal rate increased nonlinearly as the abrasive concentration increased from 0.5 to 5 wt% concentration. At 7 wt% concentration, the removal rate became saturated at 0.57 μm/min. Many abrasive particles may not be needed to remove the ground surface in the Si polishing process, because an excessive number of abrasive particles reduces the mechanical removal efficiency of each abrasive particle. Thus, the presence of an active abrasive in the slurry increases the wear rate of the silicon wafer. However, the number of inactive abrasive particles between the wafer and pad increases as the abrasive concentration in the slurry increases, which does not lead to an increase in removal rate because of the sliding of the silicon wafer against pad asperities without causing indenting and plowing of the Si surface by the highly inactive abrasive particles on the wafer surface.

As shown in Fig. 6(b), the wafer thickness after CMP was the most non-uniform (0.33%) at the abrasive concentration of 0.5 wt%, and the non-uniformity decreased to 0.23% upon increasing the abrasive concentration to 3 wt%. The non-uniformity also increased when the abrasive concentration was increased to 5 and 7 wt%. Thus, a reasonably uniform removal rate can be obtained with a suitable abrasive concentration in the slurry, although the removal rate increases with abrasive concentration in silicon polishing.

Figure 6(c) shows the coefficient of friction (COF) with increasing abrasive concentration. The plots of COF and wafer non-uniformity were very similar according to abrasive concentration. The COF decreased to 0.23 as the abrasive concentration increased to 3 wt% then increased to 0.25 at 7 wt% abrasive concentration. Therefore, the relationship between wafer non-uniformity and the COF can be discussed as follows. The non-uniformity of wafer thickness for abrasive concentrations of 1.5 to 3 wt% was better than that for other abrasive concentrations, because the friction force induced at a single abrasive particle decreased and became steady as the abrasive concentration increased to 3 wt%, leading to a uniformly distributed sliding friction on the silicon wafer due to the evenness of the abrasive particles between the wafer and pad.

When the abrasive concentration is very low (i.e., below 1.5 wt%), the silicon surface is in uneven contact with both the abrasive particles and the pad asperities. This uneven contact occurs because the force acting on a single abrasive particle increases during silicon polishing using a slurry of low abrasive concentration, and then the active abrasives on pad asperities enter into the pad surface. Thus, the silicon surface is polished by the two-body abrasion (i.e., sliding motion) of abrasives, which is induced by the strong elastic contact force between the abrasive particles and pad asperities. Therefore, CMP with low abrasive concentrations can result in a high COF because of the low number of abrasive particles.
In contrast, the abrasive particles are evenly distributed on the pad asperities when the abrasive concentration is relatively high (i.e., 1.5 to 3 wt %) in the CMP process. The pad deformation caused by active abrasive particles on the pad asperities decreases with increasing abrasive concentration, and then the silicon surface is removed by the low elastic contact force. Moreover, three-body abrasion (i.e., rolling motion) by some of the abrasive particles may occur owing to the decrease of the force acting on a single abrasive particle, and the abrasives can be either active or inactive in material removal. Thus, CMP with a high abrasive concentration can yield a decrease in COF because of the large number of abrasive particles.

In particular, it can be expected that the reduced COF between the wafer and pad led to stable wear across the whole surface of the wafer with the increase in abrasive concentration, which resulted in the improvement of the within wafer non-uniformity of wafer thickness. Furthermore, the COF is correlated with the non-uniformity of the removal rate because of the tilting of the carrier by the gimbals during CMP. When the wafer slides against the pad, a higher pressure located less than 10 mm from the leading edge on the wafer than the pressure at the wafer center is generated by the gimbals and the pad rebound phenomenon. Thus, the distribution of the friction force on the wafer is similar to the pressure distribution; thus, the removal rate is generally higher at wafer edges than at its center. However, the decrease of COF yields a parallel of the carrier against the pad on the platen, leading to a more uniform pressure distribution on the wafer. Subsequently, the reduced COF results in uniform sliding friction between the pad and wafer, which leads to an improvement of within wafer non-uniformity of wafer thickness after silicon polishing.

However, the COF also increased when the abrasive concentration was very high (i.e., 5 and 7 wt %) in the silicon-polishing slurry. This is because above 3 wt % concentration, the large number of abrasive particles increased the non-uniformity of the mechanical abrasion of the silicon surface because of the unevenness of the abrasive particles between the wafer and pad. The slurry with excessive abrasive concentration also has a higher removal rate owing to the increase of the abrasive’s erosion against the leading edge of the wafer, and thus the COF increases due to the tilting of the carrier by the gimbals although the force acting on a single abrasive particle decreases with increasing the number of abrasive particles. Accordingly, the non-uniformity of wafer thickness deteriorates during polishing. From the results of removal rate, non-uniformity, and COF, a suitable abrasive concentration of slurry should be determined because it is important to ensure the good performance of the silicon CMP process.

Figure 7(a) shows the material removal rate with increasing slurry flow rate. The removal rate was almost constant at 0.55 μm/min with the increase of the slurry flow rate from 100 to 1000 cm³/min, although there was a slight change of the removal rate. However, the non-uniformity of wafer thickness improved after polishing with a higher slurry flow rate, as shown in Fig. 7(b). Figure 7(c) shows that the temperature on the pad rapidly increased to over 29°C when the polishing began, and then it continued to increase to 31°C during 10 min of polishing at low slurry flow rates of 100, 300, and 500 cm³/min. At high slurry flow rates of 700 and 1000 cm³/min, the temperature variations during polishing were much less than those at low slurry flow rates. Therefore, the slurry flow rate had an effect on the temperature variation during the silicon CMP process. The temporal change of temperature decreased with increasing slurry flow rate, improving the non-uniformity of the removal rate. This temperature reduction results from the
fact that a high slurry flow rate is a good cooling source in the polishing process.\(^{28}\) In addition, the almost constant removal rate with the increase of slurry flow rate is due to the effective dissolution characteristic of the slurry with abundant amine as the accelerator agent, regardless of the decrease of the average temperature with the increase of slurry flow rate.

### 3.3 Stabilization of material removal process during silicon CMP

Figure 8 shows the friction force variation during silicon CMP using the alkali-based slurry. After the friction force rapidly decreased from 28 to 19 kgf in about 10 s, it gradually increased up to 60 s, as shown in Fig. 8(a). The friction force was constant at about 21 kgf between 60 and 600 s, as shown in Fig. 8(b). The native oxide on the silicon wafer appears to cause the initial rapid decrease of friction force.\(^{25,29}\)

Also, the surface roughness of the ground wafer may be related to the variation of the friction force during the polishing time. The surface roughness of the silicon wafer was high with \(R_a = 50\) nm before polishing, as shown in Fig. 9, because of the wheel marks on the silicon surface. The surface roughness rapidly decreased to 6 nm during 1 min of polishing then further improved to about 2 nm after 10 min polishing. Thus, the mirrorlike surface of the silicon wafer (\(\sim 24\) nm) improved the stability of the friction force after 1 min polishing.

In addition, the initial variation of friction force during polishing was caused by the deformation of the polishing pad. Figure 10 shows SEM images of the felt-type polishing pad. The pad consists of a fiber coated with polyurethane and a pore, as shown in the top and cross-sectional views. Thus, the pad is compressed by the loaded wafer and recovers during the revolution of the platen owing to the rotary CMP polisher, as shown in Fig. 1. This causes the well-known viscoelastic behavior of the polishing pad,\(^{30,31}\) which can be classified into elastic, viscoelastic, and permanent deformations based on the four-element model shown in Fig. 11(a). When the stress (\(\sigma\)) (i.e., pressure on pad) is constant, the elastic strain (\(\varepsilon_1\)) and viscoelastic strain (\(\varepsilon_2\)) are defined as the ratio of the stress to each modulus (i.e., \(E_1\) and \(E_2\)), respectively. As shown in Fig. 11(b), elastic deformation immediately occurs when the wafer presses and relaxes the pad surface during polishing, and viscoelastic deformation depends on the time delay due to the damper (\(\eta_2\)). Also, permanent strain (\(\varepsilon_3\)), which is not recovered by the damper (\(\eta_1\)) owing to the polymer characteristics and pad structure, occurs when the pad is unloaded during each revolution of the platen. However, it is difficult to measure the strains corresponding to the three deformations during polishing because the period of viscoelastic behavior is very short and is based on the rotational velocity of the pad and wafer. Therefore, the characterization of the viscoelastic behavior of the polishing pad was investigated by VMS, as shown in Fig. 11(c). As a result, the loaded pad was gradually deformed for 30 s, then the pad thickness recovered for 30 s when the weight was removed from the pad surface because of the temporal volume variation characteristic of the polymer.\(^{30}\) Therefore, the pad is deformed by the loaded
wafer during polishing, changing the friction force between the pad and wafer. Also, this phenomenon may affect the polishing results with increasing the polishing number, i.e., when a new pad is attached on the polishing platen.

From these results for the variation of friction force, the break-in process, i.e., the stabilization of material removal, was investigated using a new pad during silicon CMP with the alkali-based slurry. Figure 12(a) shows the removal rate as a function of the number of polishes. The removal rate increased to 0.5 μm/min as the number of polishes increased to the sixth polish. After the seventh polish, the removal rate became constant at 0.51 μm/min. The non-uniformity of the thickness after polishing decreased from the fourth polish, as shown in Fig. 12(b). This decreased non-uniformity might be related to the steady COF from the fourth polish because of the uniform abrasion of the silicon surface during polishing, as shown in Fig. 12(c). Figure 12(d) shows the temperature variation on the pad surface during the break-in process. Similar to the stabilization of the removal rate from the seventh polish, the temperature remains constant from the seventh polish. Thus, the removal rate is related to the elevated temperature as the number of polishes increases due to the improvement of the chemical dissolution of silicon at the higher temperature. Also, the native oxide is more easily abraded by the silica abrasive particles in the slurry at a higher temperature. Thus, the stabilization of the material removal rate and the reduced wafer non-uniformity might be caused by the rapid removal of the native oxide and the ground surface on the silicon wafer, which results in the steady COF and temperature during silicon CMP. In particular, the permanent deformation of viscoelastic behavior is related to the break-in process. During the viscoelastic behavior measurement over 90 cycles (i.e., the total polishing time for nine wafers), the compressive deformation and recovery occurred repeatedly and the permanent strain was stabilized after 60 min, as shown in Fig. 13. The time required to achieve permanent deformation can become shorter with the increase of temperature during polishing. Therefore, the permanent deformation of the pad affected the COF during the break-in process, and then the uniform COF improved the wafer non-uniformity due to the stabilized pad during silicon CMP using an alkali-based slurry.

4. Conclusions

The material removal characteristics in the silicon CMP process were investigated by monitoring the friction force and temperature. The native oxide on the silicon wafer prevented the dissolution of silicon in an alkali-based slurry with an amine agent. The amount of dissolution of the silicon wafer without a native oxide film increased with the slurry temperature. Si–Si bond breaking by the slurry reduced the hardness of the silicon. In particular, the hardness of the silicon wafer loaded against the polishing pad with slurry was lower than that of the as-received wafer. Also, the surface roughness and microtopography of the loaded silicon wafer were better than those of the silicon wafer dipped in slurry.

The removal rate increased nonlinearly with the abrasive concentration of the slurry. It appeared that the mechanical removal efficiency of each abrasive particle decreased as the abrasive concentration increased to 7 wt %. Also, the wafer non-uniformity after CMP was correlated with the COF, and the non-uniformity and the COF after polishing with 1.5–3 wt % abrasive concentrations were lower than those for other concentrations. This was because uniform abrasion due to the evenness of the sliding friction between the wafer and pad occurred at a suitable abrasive concentration of the slurry. In addition, the removal rate was nearly constant as the slurry flow rate increased, although the temperature on
the pad gradually decreased. Moreover, the increased slurry flow rate improved the wafer non-uniformity because the slurry was a good cooling source at high rates of 700–1000 cm$^3$/min.

During the break-in process, the native oxide and surface roughness of the silicon wafer caused a change of the friction force during polishing. Furthermore, the removal rate and wafer non-uniformity were constant from the seventh polish and fourth polish, respectively. The stabilization of the material removal rate was related to that of the temperature, and the stabilization of the wafer non-uniformity was related to that of the COF. In particular, the change in pad thickness due to the permanent deformation of the polishing pad during the CMP process may affect the non-uniformity of wafer thickness, which is related to the COF.

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