Effects of slurry components on the surface characteristics when chemical mechanical polishing NiP/Al substrate

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Received 22 October 2003; accepted in revised form 21 December 2004
Available online 26 January 2005

Abstract

A series of experiments were conducted to study the effects of slurry components on surface characteristics as well as material removal rate when polishing NiP/Al substrate disk. It was shown that with a finer and softer abrasive, a better surface roughness and waviness could be achieved but the material removal rate is relatively slow. Comparing with \( \text{H}_2\text{O}_2 \) oxidizer \( \text{HNO}_3 \) results in higher material removal rate, better surface roughness but slightly degraded surface waviness. The increase in oxidizer concentration will increase material removal rate, slightly improve surface roughness but slightly degrade the surface waviness.

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PACS: 83.70.H; 81.65; 46.30.P

Keywords: Planarization; Surface roughness; Aluminium oxide; Silicon oxide

1. Introduction

Chemical Mechanical Polishing (CMP) has been a widely applied process for the planarization for integrated circuit applications [1–6] as well as for hard drive substrate polishing [7,8]. The major compositions of slurry are the abrasive and the oxidizer. Precise stable particle size distribution is a key-contributor to maintaining low defect levels [9]. While the particle size, concentration, and fill factor affect polishing rate, large particles are more likely to contribute to workpiece scratching [2,10,11].

Several researchers [12,13] study the effects of slurry combinations for copper CMP. Wang et al. [14] found that removal rate decreased with the increasing ratio of deionized water to slurry. The additional deionized water led to significant reduction of abrasive concentration and oxidizer concentration in the slurry. The pH value of the diluted slurry was also slightly changed. Romagna and Fayolle [15] believed that decreasing the concentration of slurry leads to the drop of the removal rate.

Wang et al. [14] found that the removal rate increased markedly and the removal uniformity slightly degenerated when the slurry flow rate was reduced in tungsten CMP. The removal rate increase with less slurry flow rate may be attributed to the increase in temperature of slurry and tungsten film in the polishing process. And Wang et al. [16] believed that higher polishing removal rate has been proven to result in higher pad temperature.

Ishimoto [17] found that the removal rate decreased when the slurry flow rate was reduced before the midpoint of polishing. But removal rate was not affected as slurry flow rate was reduced after the midpoint of polishing. He suggested that the slurry is needed only at the start of polishing in interlayer dielectric CMP and that the wafer can be planarized without the slurry after the midpoint of polishing.

Jiang et al. [18] concluded that the water-based slurry was essential for CMP of \( \text{Si}_3\text{N}_4 \) workpiece. Grover et al. [19] found that the viscosity of the slurry has a strong effect on the removal rate of material on the wafer when polishing tungsten. It was also found that increasing the pH value of slurry at the time of polish increases the removal rate.
Table 1

<table>
<thead>
<tr>
<th>Factors</th>
<th>Levels</th>
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<tbody>
<tr>
<td>Abrasive type</td>
<td>SiO₂ (25-50 nm)</td>
</tr>
<tr>
<td>Abrasive concentration (wt%)</td>
<td>2</td>
</tr>
<tr>
<td>Oxidizer type</td>
<td>HNO₃</td>
</tr>
<tr>
<td>Oxidizer concentration (vol%)</td>
<td>2</td>
</tr>
<tr>
<td>Flow rate of slurry (mL/min)</td>
<td>250</td>
</tr>
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In this study, it is of interest to study the effects of slurry components on surface characteristics and material removal rate when polishing NiP/Al substrate. The slurry components studied includes the type of abrasive, the concentration of abrasive, the type of oxidizer, and the concentration of oxidizer. A series of experiments were conducted. The experimental design and experimental setup is shown in the following section. The experimental results are then analyzed and discussed. Finally, conclusions are made based on the results observed.

2. Experimental design and experimental set-up

In order to study the effects of slurry components on surface characteristics and the removal rate of NiP/Al substrate, a series of experiments were conducted with three levels of abrasive types, two levels of abrasive concentration, two levels of oxidizer types, two levels of oxidizer concentration, and two levels of flow rates. All the experimental parameter levels are listed in Table 1.

For comparison, experimental tests without either abrasive or oxidizer and tests using commercial slurry with two levels of abrasive concentration, two levels of oxidizer concentration, and two levels of flow rate, were also conducted. All these experimental parameter levels are listed in Table 2. The commercial slurry used is FUJIMI H3523 which contains an Al₂O₃ abrasive with a particle size around 0.65 μm. And the pH value is around 3.3. The polishing time for each test was 4.5 min.

Furthermore, in order to study the effects of the oxidizer on workpiece hardness, a series of corrosion experiments were conducted. Oxidizer, erosion time and levels used in corrosion experiments are listed in Table 3.

Table 2

<table>
<thead>
<tr>
<th>Factors</th>
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<tbody>
<tr>
<td>Abrasive concentration (wt%)</td>
<td>x</td>
</tr>
<tr>
<td>Oxidizer concentration (vol%)</td>
<td>y</td>
</tr>
<tr>
<td>Flow rate of slurry (mL/min)</td>
<td>250</td>
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Table 3

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<tr>
<td>Oxidizer type</td>
<td>HNO₃</td>
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<tr>
<td>Oxidizer concentration (vol%)</td>
<td>2</td>
</tr>
<tr>
<td>Corrosion time (s)</td>
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<tr>
<td></td>
<td>10</td>
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<td></td>
<td>30</td>
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<td></td>
<td>60</td>
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<td></td>
<td>300</td>
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studied. All the experiments were conducted on a DSM 11.8B-10P-AL double sides polishing machine. The polishing pad used is FUJIBO H9900. Fig. 1 shows the schematic diagram of the polishing machine used. Other fixed polishing parameters are listed in Table 4.

The surface roughness of specimens was measured using the Tencor P-12 probe profilometer. The scanning length was 200 μm, and the scanning directions were all along the radial direction. The surface waviness of specimens was measured using the OptiFlat interferometer. A sampling data of 107,926 that covered the entire workpiece surface were collected, and they were used to calculate the surface waviness by two-dimensional fast Fourier transformation (FFT) with different wavelength filter. For each test, disks at three different locations are used as specimens. Therefore, there are three measures for each test used to estimate the pure error of the test.

![Schematic diagram of DSM 11.8B-10P-AL polishing machine.](image-url)
A MTS nano indenter XP was used to measure the change of hardness from the surface of the workpiece to the deeper area of the workpiece.

3. Experimental results and discussion

The effects of slurry components and flow rate on surface characteristics of NiP/Al substrate and removal rate were analyzed and discussed.

3.1. Removal rate

In this study, the thickness removed per minute is used as a removal rate index. The analysis of variance (ANOVA) [20] is used to study effects of slurry components and flow rate on removal rate. The calculated sample standard deviation is 0.0078 µm/min.

It is found that the main effects of oxidizer, oxidizer concentration, abrasive type, and abrasive concentration on the removal rate are significant while the effects of slurry flow rate are shown insignificant. As for the two-level interaction effect, oxidizer and abrasive type, oxidizer and abrasive concentration, oxidizer concentration and abrasive type, as well as abrasive type and abrasive concentration, all were shown significant.

Fig. 2 shows the interaction effects of abrasive type, oxidizer, and abrasive concentration on removal rate. Fig. 3 shows the interaction effects of abrasive type, oxidizer, and oxidizer concentration on removal rate. Fig. 4 shows the interaction effects of abrasive type, oxidizer, and slurry flow rate on removal rate.

As shown in the figures, the removal rate was 0.105 µm/min on average when a finer and softer SiO₂ abrasive was used, while the removal rate was increased to 0.314 µm/min and 0.426 µm/min when Al₂O₃-γ abrasive and a larger Al₂O₃-α abrasive were used, respectively. This would be attributed to the fact that the particle size of Al₂O₃-α abrasive is bigger than Al₂O₃-γ abrasive, and the particle hardness of Al₂O₃-γ abrasive is harder than SiO₂ abrasive. The particle size and hardness of the abrasive have strong effects on the removal rate of the material [10]. Komanduri et al. [21] and Cho et al. [22] found similar results when using alumina abrasive in Cu and Al CMP.

The removal rate also increased as abrasive concentration increased. When 2% abrasive was used, the removal rate on average was 0.250 µm/min, and the removal rate of 4% abrasive was 0.313 µm/min. Within the range studied, it is shown that the abrasive wear becomes more significant when there are more abrasives in the slurry. A similar phenomenon was observed when Fujimi slurry was used, while the removal rate with Fujimi slurry was a little higher than other solutions used. It is also shown that the removal rate was very low when there was no abrasive in the slurry.
Cho also found the similar results when using Al₂O₃ abrasive and K₂Cr₂O₇-based slurry in Al CMP [22].

As shown in the figures, the removal rate was 0.443 μm/min when HNO₃ oxidizer was used while that was only 0.149 μm/min when H₂O₂ oxidizer was used. The removal rate of 2% oxidizer was 0.258 μm/min, and the removal rate of 4% oxidizer was 0.334 μm/min. This is because the HNO₃ had a higher corrosion rate than H₂O₂ when polishing NiP/Al substrate. The corrosion rate increased with increasing oxidizer concentration. When there was no oxidizer in the slurry, there was no chemical action but slow mechanical removal only. Wang et al. [12] found that similar results for increasing the volume percentage of HNO₃ from 1% to 3% added resulted in a slight increase in polishing rate for copper CMP.

Fig. 5 shows the change of hardness from the surface of the workpiece to the deeper area of the workpiece when no solution was used. It was found that the material was harder closer to the surface. The hardness is ranging from 8 to 15 GPa. Fig. 6 shows a typical result of effects the change of hardness from the workpiece to the deeper area of the workpiece when oxidizer solution was used. The range of hardness is now ranging from 1 to 5 GPa. It is interesting to find that the hardness close to the surface is reduced significantly. A five times reduction in hardness around 50 nm from the surface is observed. It shows that 5 s is long enough for a chemical reaction to affect the surface hardness effectively in the area, although a longer corrosion time will further reduce the hardness. It is of interest to look at the area with shorter corrosion time; however, the measurement facility is not able to reveal this aspect.

Fig. 7 summarizes the effect of the oxidizer on average surface hardness around 100 nm from the surface. Both HNO₃ and H₂O₂ can effectively reduce the hardness. The hardness is lower when HNO₃ is used. It confirmed the expectation that the HNO₃ had a higher corrosion rate than H₂O₂ when polishing NiP/Al substrate. The hardness is lower when higher oxidizer concentration is used. This fact can explain why the removal rate increased as the oxidizer concentration increased.

### 3.2. Surface roughness

It was shown from the ANOVA that the oxidizer, oxidizer concentration, abrasive type, and abrasive concentration affect the surface roughness significantly. The effect of flow rate of slurry is insignificant. As for the two-level interaction effects, such as oxidizer and oxidizer concentration, as well as abrasive type and abrasive concentration, these are shown significant. The calculated sample standard deviation is 0.10 nm.

Fig. 8 shows the interaction effects of abrasive type, oxidizer, and abrasive concentration on surface roughness. Fig. 9 shows the interaction effects of abrasive type,
oxidizer, and oxidizer concentration on surface roughness. Fig. 10 shows the interaction effects of abrasive type, oxidizer, and slurry flow rate on surface roughness. When SiO₂ abrasive was used, the surface roughness of workpiece was 1.17 nm on average, while the surface roughness was 1.16 nm and 1.68 nm when Al₂O₃-γ and Al₂O₃-α abrasive were used, respectively. This is because the particle size of Al₂O₃-α abrasive is bigger than Al₂O₃-γ and SiO₂ abrasive, and produced more micro scratching. The finer and softer SiO₂ abrasive is generally superior to Al₂O₃ abrasive in achieving better surface roughness unless no oxidizer is used. In this case the removal rate is too low to smooth the surface completely.

In most cases the surface roughness decreases as the abrasive concentration decreases. Cho also found the similar results when using Al₂O₃ abrasive and K2Cr2O7-based slurry in Al CMP [22]. The reason for this would be the reduction of micro scratching produced by the abrasives. However, in cases of very low removal rate (for example, no oxidizer was used, or no abrasive was used), the decrease of the abrasive concentration will degrade the ability to smooth the surface completely.

The surface roughness was improved by using HNO₃ oxidizer rather than H₂O₂. When HNO₃ was used as the oxidizer, the surface roughness on average was 1.11 nm, while when H₂O₂ was used, surface roughness was 1.41 nm. And, the surface roughness was improved by the higher oxidizer concentration (4% in this case).

The surface roughness was 1.37 nm on average when 2% oxidizer was used, while the surface roughness was reduced to 1.14 nm when 4% oxidizer was used. The surface roughness polished without oxidizer was 1.91 nm. This is because the solution with higher corrosion rate (such as HNO₃ or higher oxidizer concentration) can soften the work surface quicker, and the possibility of micro scratching is reduced because of the softer work surface.

Fig. 11 shows the correlation between surface roughness and removal rate. As shown in the figure, surface roughness was bad when the removal rate is below 0.1 μm/min. It is attributed to the fact that the polishing time is not long enough to smooth the surface completely. Otherwise, the solution with finer and softer abrasive would be superior to the Fujimi slurry in achieving better surface roughness.

3.3. Surface waviness

The range of spatial wavelength of surface waviness (Wₐ) to be taken into account was about 1/3 – 4 times the
length of the slider. In this study, the average surface waviness with a spatial wavelength range of 0.4-5.0 mm was estimated.

It was found that surface waviness was affected by oxidizer concentration, abrasive type, abrasive concentration, and slurry flow rate. As for the two-level interaction effects, there is no significant second-level interaction effect. The calculated sample standard deviation is 0.04 nm.

Fig. 12 shows the interaction effects of abrasive type, oxidizer, and abrasive concentration on surface waviness. Fig. 13 shows the interaction effects of abrasive type, oxidizer, and oxidizer concentration on surface waviness. Fig. 14 shows the interaction effects of abrasive type, oxidizer, and slurry flow rate on surface waviness.

As shown in the figures, the surface waviness decreases as finer abrasives are used. The surface waviness of the workpiece were 0.95 nm and 0.97 nm on average, respectively, when finer abrasive SiO₂ and Al₂O₃-γ were used, while it was 1.14 nm when Al₂O₃-α abrasive was used.

The surface waviness slightly improved with an increase in abrasive concentration. The surface waviness was 1.08 nm when 2% abrasive was used, while the surface waviness was slightly improved to 1.02 nm when 4% abrasive was used. The average surface waviness was 1.25 nm when no abrasive was used. The experiments of Fujimi slurry show similar results. Cho also found similar results when Al₂O₃ abrasive and K2Cr2O7-based slurry were used in Al CMP [22].

The surface waviness was slightly improved by H₂O₂ instead of HNO₃ oxidizer. The surface waviness was 1.05 nm with HNO₃, while that was 1.02 nm with H₂O₂. The surface waviness also slightly improved by reducing the oxidizer concentration. The surface waviness was 1.05 nm with 4% oxidizer, while it was slightly reduced to 1.01 nm with 2% oxidizer. Without the oxidizer, the surface waviness was 1.16 nm. In the Fujimi slurry experiments, the surface waviness slightly improved by increasing oxidizer concentration.

Fig. 15 shows the correlation between the surface roughness and the removal rate. As shown in the figure, the surface waviness was bad when removal rate was below 0.05 μm/min. It is attributed to the fact that the polishing time is not long enough to planarize the whole surface. Otherwise, the solution with finer and softer abrasive might be superior to the Fujimi slurry in achieving better surface waviness.

4. Conclusions

In this study, a series of experiments were conducted to study the effects of slurry components on surface characteristics as well as material removal rate when polishing NiP/Al substrate disk. It is shown that with finer abrasives, better surface roughness and waviness can be achieved while the material removal rate is lower. The increase in abrasive concentration will increase the material removal rate, slightly improve surface waviness but slightly degrade the surface roughness. Comparing with H₂O₂, the oxidizer HNO₃ results in higher material removal rate, better surface roughness but slightly degraded surface waviness. The increase in oxidizer concentration will increase material removal rate, slightly improve surface roughness but slightly degrade the surface waviness. Without the oxidizer or abrasive in solution, the material removal rate is low, and the improvement in surface roughness and waviness is not
identified. The slurry flow rates in the ranges studied show little effects on surface roughness, surface waviness or material removal rate.

Acknowledgements

The authors wish to thank the National Science Council, R.O.C. for their final support (NSC91-2212-E-007-049) and the Trace Storage Technology Corporation for providing experimental set-up, specimen and technical assistance.

References