Abstract

Chemical mechanical planarization (CMP) slurries contain a small amount of large particles that will contribute to micro-scratches on a wafer’s surface. Capturing large particles from slurry with high solids concentration without changing the working particle distribution is one of the main challenges for a slurry filter. In general, filter performance evaluation utilizes polystyrene latex (PSL) beads to determine particle-size retention. The PSL retention test provides good resolution under low particle content conditions. However, it is not relevant when simulating high solid content solution, such as CMP slurry. Therefore, developing a new method to fill this technology gap is critical. This study focuses on characterizing CMP filter performance by using ceria ($\text{CeO}_2$) and silica ($\text{SiO}_2$) particles and comparing PSL bead retention. Based on our study we have developed a new method to evaluate slurry filters. Using this new method further identifies the filtration retention efficiency and emphasizes the discrepancy between commercial slurry and PSL beads. It also helps to advance new sub-100 nm media development for CMP filtration.

Introduction

In the advanced node CMP planarization application, scratch defect become a key factor in the process yield performance. Slurry manufacturers use a variety of nano abrasive particles (10 to 100 nm), such as silicon oxide (silica), cerium oxide (ceria) and alumina, in order to achieve the planarization demands while improving the efficiency and yield of the process. The CMP slurry abrasive particles will agglomerate by pH shift, shear stress and temperature effects. It will cause the particles to agglomerate into large particle or gel, which is mainly caused by the scratching factors. Nano abrasive particles are a challenge of an advanced CMP filter. We can find the results of the retention tests distinguish between product performance with a variety of abrasive particles and particle size distribution. The CMP filter performance is difficult to test one by one for the variety of commercial CMP slurry types. Filter performance testing mainly focuses on variety of abrasive particles and particle size distribution. This study is a comparison of commercial slurry filtration efficiency on abrasive particles.

Currently, the most commonly used filters for CMP slurry applications are “graded density depth filters.” These filters, constructed of wrapped nonwoven polypropylene media, have a retention gradient along their depth (Figure 1). This retention gradient provides gradual removal of particles of different sizes throughout the depth. An ideal depth filter should have a retention gradient that closely matches the size distribution of the “particles” to be removed in the intended application. As a result, the mass loading of the removed “particles” will be “uniform” throughout the depth of the filter media to achieve the maximum particle and/or gel holding capacity.

Figure 1. Graded-density depth filter
Depth filters do not have a sharp retention cut-off. Unlike Membrane filters that have a fairly sharp cut-off on the rated micron size (Figure 2). Furthermore, there is no accepted standard for the retention rating of a depth filter. Most filter suppliers use nominal or absolute pore size ratings to specify a depth filter’s performance.

The filter’s retention efficiency is one of the key factors that determine filter lifetime. A tight filter (higher retention for smaller particles) is preferred (at point-of-use) for removing the majority of defect-causing particles. However, if used alone, the filter may have a short lifetime since it will be quickly loaded with particles of a wide size range. More open filters will provide longer lifetime with less benefit on defect reduction. Therefore, serial filtration should be used, which can provide better lifetime with desirable defect-reduction results. Furthermore, serial filtration can be implemented at various locations in a slurry delivery system.

Retention Test Materials

Retention with PSL Beads

PSL beads are a general method used to define particle removal efficiency of the filters. Retention with PSL beads for CMP filters has been used generally to confirm filtration efficiency.

Retention with Slurries

Retention with slurries can be an index of particle removal efficiency of CMP filter for slurry.

There are various slurries used in applications, so this test can be applied for a specific slurry.

Retention with Abrasives

Retention with abrasives (Table 1) can be an alternative for retention with slurries once the performance between abrasive and slurry is confirmed. Abrasive type and concentration can be adjusted based for this test type.

<table>
<thead>
<tr>
<th>Slurry Base</th>
<th>Abrasive Type</th>
<th>Chemical Formula</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica</td>
<td>Colloidal silica</td>
<td>SiO₂</td>
<td>STI, ILD, metal</td>
</tr>
<tr>
<td>Ceria</td>
<td>Ceria</td>
<td>CeO₂</td>
<td>STI, ILD</td>
</tr>
</tbody>
</table>

Experiments

In this study, commercial abrasives, focused on colloidal silica (SiO₂) and ceria (CeO₂) slurries were used to simulate slurry filtration behaviors. The colloidal silica shape is spherical type is 20% concentration, particle size 30~60 nm at pH 7.3; ceria shape is irregular 30% concentration, particle size 50~150 nm at pH 6.6.

The two abrasives were diluted with DI water to 1% concentration and fully mixed for 40 minutes to prepare for the filtration test. After the mixture is complete, the pH value is measured (Table 2).

<table>
<thead>
<tr>
<th>Abrasive Type</th>
<th>Concentration (%)</th>
<th>Shape</th>
<th>pH</th>
<th>Dilution to 1% pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colloidal silica (CS)</td>
<td>20</td>
<td>Spherical</td>
<td>7.3</td>
<td>6.8</td>
</tr>
<tr>
<td>Ceria (CE)</td>
<td>30</td>
<td>Irregular</td>
<td>6.6</td>
<td>6.4</td>
</tr>
</tbody>
</table>
The filtration evaluation has used an advanced CMP filter. The Planargard® NMB CMP slurry filter (NMB 01 and NMB 03) is constructed of a new polypropylene membrane technology that produces nanofibers and multi-layer continuous-melt-blown (CMB) media for improved flow path with high particle retention. Before installing the filter into the CMP test stand (Figure 3), pressure activation was ensured on each filter.

Nanofiber manufacturing processes will trap micro air bubbles in tiny spaces in the melt blown media structure. Sometimes the initial pressure would be higher than normal levels. To address this before usage, a liquid flush process will drive air away from media and activation filter to allow the filter to show better performance (Figure 4). Pressure activation, use DIW (deionized water) to flow through the filter at 20 psi and pulse several times. As a result, we can see the initial pressure decrease significantly.

**Experimental Procedure:**
1. Dilute the abrasive to 1% concentration
2. Install the filter into CMP test stand
3. Initiate the pressure activation operation
4. Using a abrasive at 1% concentration, flush the filter and the entire system for 5 minutes
5. Collect a downstream sample for LPC measurement
6. Collect an upstream sample for LPC measurement
7. Continue recording pressure increases by time
Result/Discussion

LPC Result

From this experiment, the results show colloidal silica (CS) and ceria (CE) slurries show a different LPC curve model. In Figure 5, colloidal silica particles decrease significantly after filtration. The LPC curve from small to large shifts to a low level through this test shows capture of the large particles from the slurry. By comparing different retention ratings, it could help distinguish which is more suitable for the particular product application. This test method is also providing new CMP filter media development evaluation references. In Figure 6 ceria abrasive particles decrease significantly after filtration with particles larger than 2 µm being completely removed. Comparing differential filter pore size ratings, the LPC curve is similar but still can distinguish the improved performance of NMB01 over NMB03.

![LPC Curve Colloidal Silica—Compare Pore Rating](image1)

Figure 5. LPC curve colloidal silica — compare pore rating

![LPC Curve Ceria—Compare Pore Rating](image2)

Figure 6. LPC curve ceria — compare pore rating
Retention Result

Based on LPC results, we can calculate retention performance to compare filtration efficiency. PSL, slurry and abrasive are suitable for filter evaluation, but which one best emulates real-life conditions. Compare this with the three methods and we can see the retention results are different but show a similar trend. PSL retention is a more suitable representation of micron retention rating filter performance. Slurry/abrasive retention is a suitable representation of micron to nano scale retention ratings and it is a more fitting real-life condition. PSL results show very high retention of nearly 100% at the >0.8 µm particle size. This result has a great gap with the real situation, but PSL still is a reproducible method suitable for a reliable study (Figure 7).

Colloidal silica-based slurry shows a different retention bar chart than ceria-based slurry. Colloidal silica slurry shows very good retention at >0.8 µm, colloidal silica abrasive has similar trend with commercial slurry. We can use colloidal abrasive particle for experiments to study colloidal silica-based slurry filtration behavior (Figure 8).

Ceria slurry shows very good retention large particle scale, ceria abrasive also has similar trend with commercial slurry. We can use this method to distinguish which one is more suitable for the particular product application (Figure 9).

Summary

PSL, slurry and abrasive are all suitable for filter evaluations. This study seeks to determine which type is most similar to real-world-conditions in the fab. Comparing these three methods, we can see the retention results are different, but have a similar trend. PSL retention is a more suitable representation of micron-scale pore rating (retention) filter performance. Slurry/abrasive retention is a suitable representation of micron to nano scale pore rating (retention) filter performance.

Selection of a pure abrasive particle is effective for filter evaluations. Abrasive not only distinguishes different pore size rating performance, it also provides the end user and slurry matching reference.
References


