FILTRATION CHARACTERISTICS OF CMP SLURRIES

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Abstract

Filters for current and next-generation CMP slurries must now exhibit high retention of particles above 0.5 µm while passing particles 0.03–0.2 µm in size at high flow rates and without significant pressure drop. We have developed a range of filters using various depth media that meet the requirements for the retention of large particles (>0.5 µm) in slurries with sub-µm working particles. We demonstrate how 1.5 and 5.0 µm rated filters show suitable filter characteristics for use as point-of-use and distribution loop filters for silica slurry. Also, the importance of empirical evaluation in filter selection is demonstrated, with large variations in filter performance measured for 0.5 and 1.0 µm filters processing polystyrene latex (PSL) bead challenge solution, and silica, ceria and alumina slurries.

Introduction

The specifications for chemical mechanical planarization (CMP) processes are becoming more complex and demanding as new devices manufactured with new materials on larger wafers are introduced. The call for improved planarity and lower defect levels has resulted in the size of the working particles in CMP slurries being reduced. Slurry filtration to remove unwanted large particles while maintaining the required working particle size distribution (PSD) is critical to CMP process quality. Most CMP slurries now use 30–200 nm particles at typical concentrations of 0.3–12 wt % solids. Such slurry characteristics have resulted in increasing demand for high retention, low-cost, 0.5, 0.3 and 0.2 µm filters with stringent large particle counts (LPC) and PSD specifications. At the same time, such filters must have reasonably high flow rates and long lifetimes.

If a filter removes particles in the working PSD range, it will become clogged and soon fail. The challenge to filtration technologies is especially acute if the difference in size between the particles to be retained and those to be passed is less than one order of magnitude. For filtering sub-µm CMP particles, depth media made from melt-blown fibrous materials are used (Figure 1a). Performance is strongly dependent upon the distribution of fiber sizes and the arrangement of the fibers within the media.[1, 2] Fibers with small diameters provide better retention and pressure drop characteristics than larger fibers, but a proper balance of fiber sizes is needed for optimal filter performance for any particular slurry.

Filter media show a gradual transition from retention to passage over a range of particle sizes. This gives an s-shaped percent-retention versus particle size curve (Figure 1b). Given the move to more complex chip designs using thinner layers, there is a need to remove all particles greater than 500 nm from typical CMP slurries with working particle sizes of 30–200 nm. However, this is difficult to achieve when the transition curve of most media covers several microns. Filter media allowing passage at 200 nm may not reach retention above 80% until particle sizes are over 2000 nm.

However, there are several ways of sharpening the retention curve. By using multiple layers of the same media the required smaller particles will pass through while the larger particles will experience an increasing likelihood of capture at each successive layer. If the depth of media required to stop particles of a certain size happens to be small, then increasing the surface area of the media (for example, by pleating) further improves the large particle retention. Depth media can also be graded, placing more open layers at the filter inlet and tighter layers toward the outlet (Figure 1b).

Filter design complexity is increased by the presence of gels in some slurries. To remove gels effectively without dramatic reduction in filter lifetime requires a large 3-D structure that allows gel capture through the depth of the most open media layers. Gel capture just on the top surface of the thicker media will quickly lead to flow-reduction and blocking. Formation of this layer often leads to media compression and significant increase in retention as well as pressure drop. Silica-based slurry in particular requires consideration of gels while other slurries (e.g., alumina- or ceria-based) typically do not.

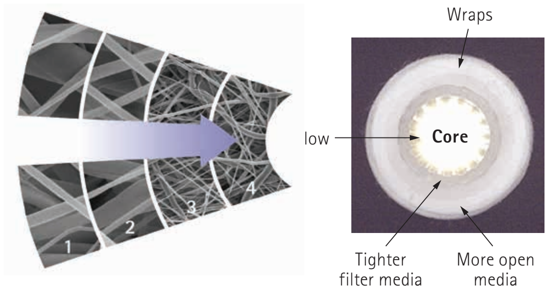

Figure 1a. A typical graded density depth media for CMP slurry filtration.

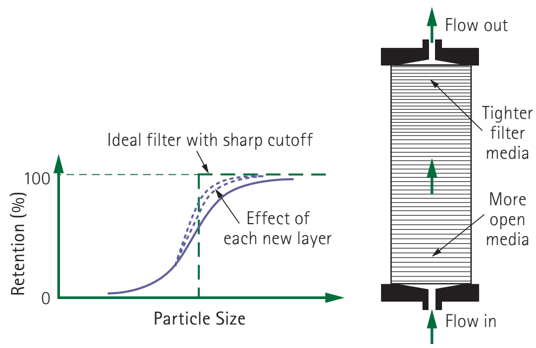

Figure 1b. The effect of multiple layer depth media on filter retention.

We have developed filters using the above depth media designs that meet the most demanding CMP slurry filtration applications. This paper demonstrates the suitability of the filters for processing slurry at the point-of-use (POU) and in the distribution loop. We also highlight the importance of empirical verification of a filter's performance with particular slurries as part of filter selection. Results from filtration studies of slurries for oxide (silica slurry), shallow trench isolation (STI) (ceria slurry) and copper (alumina slurry) CMP as well as PSL bead challenge solution are presented which show that retention, flow rate and pressure drop (Δp) have very different behaviors for the same filters processing different slurries. This confirms that CMP slurry filter optimization remains empirical in nature. We also show that graded density depth filters are effective in managing large particles in new CMP slurries.

Methodology

A silica slurry (silica-A with ~12 wt % solids) was evaluated to generate filter recommendations for distribution loop and POU filtration. In the first test, the slurry was re-circulated through an Entegris Planargard® CMP5 5.0 µm nominal rating graded density depth filter (Figure 1a) for five hours at 4.3 L/min. In the second test, the slurry was filtered using an Entegris Solaris®-01 1.5 µm nominal rating multiple layer POU depth filter (Figure 1b) at 400 mL/min, while being recirculated through the 5.0 µm loop filter. The distribution loop and POU filters' performance was evaluated by monitoring the LPC and Δp in the filter. Slurry feed and filtrate LPC were measured using a Particle Sizing Systems AccuSizer® 780 APS analyzer in the top chamber addition mode. The five-hour recirculated slurry from the above test was also filtered in single-pass tests using two graded density depth filters: 0.50 µm Planargard CS05 and 1.0 µm Planargard CMP1.

A second set of experiments was conducted to obtain filter retention, flow rate and pressure drop data for above CS05 and CMP1 filters in different abrasive slurries. Each filter was used to process a ~25 wt % solids silica-based slurry for oxide CMP (silica-1), a <1 wt % solids ceria-based slurry for STI CMP (ceria-1) and a <1 wt % solids alumina-based slurry for copper CMP (alumina-1). Further tests were conducted with the tighter CS05 media to filter a different ~25 wt % solids silica-based dispersion (silica-2) and another <1 wt % solids alumina-based slurry for copper CMP (alumina-2). A MasterFlex® peristaltic pump with Tygon® long flex life tubing was used to feed the slurries through the filter media. With no filters in the loop, the pump passed DI water at ~500 and 535 mL/min for the CS05 and CMP1 test filter housings, respectively.

A third set of experiments was conducted to obtain filter retention, flow rate and Δp data for a DI water-based PSL (polystyrene latex) bead challenge solution prepared using particles ranging from bead diameters of 0.772 to 20 micron. It is a common practice to use PSL bead challenge solutions to obtain relative retention data for various filters. These solutions are expected to retain stable particle size distribution and provide more consistent information compared to real CMP slurries which may change particle characteristics over time. Similar to CMP slurry samples, LPC for PSL bead samples were also measured using AccuSizer 780 APS in the top chamber addition mode.

Results

LPC results for the POU and distribution loop filters for silica-A slurry are presented in Figure 2. Figure 2a shows that the distribution loop LPC was stable in the five-hour run during which slurry undergoes ~170 turnovers or passes through the 5 µm CMP5 filter. In a real life fab operation, the slurry typically goes through one hundred turnovers before it is consumed.[3] Figure 2b shows the feed and filtrate LPC for 1.5 µm SLR01 POU filter in removing large particles from the slurry in a single pass.

The filters for these characterization tests were selected based on the silica-A slurry properties such as mean particle size and LPC, abrasive type, wt % solids, and application requirements including target retention level, flow rate, allowable Δp and expected filter lifetime.[4] This study shows that the tested 1.5 µm rating POU and 5.0 µm distribution loop filters are suitable for filtration of this silica slurry formulation. However, if the slurry is expected to see a very large number of turnovers before usage, a more open 7.0 µm nominal rating Planargard CMP7 may be employed for the distribution loop filtration.

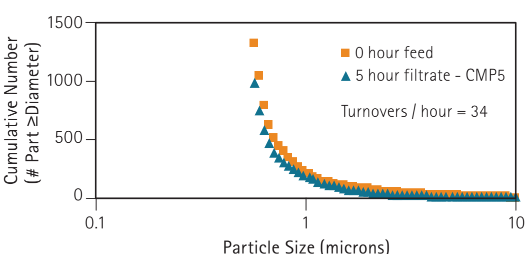

Figure 2a.

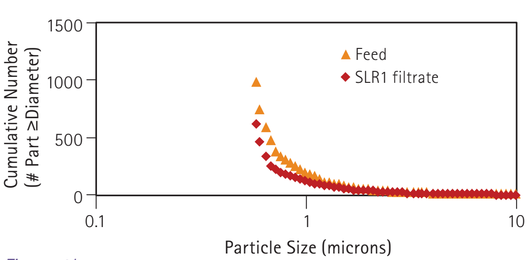

Figure 2b.

Figure 2. LPC for 5.0 µm CMP5 distribution loop depth filter and 1.5 µm multiple layer POU SLR01 filter in silica-A slurry. (a) Initial feed to distribution loop filter and filtrate from loop filter after five-hour recirculation, and (b) five-hour recirculated slurry feed to the POU filter and filtrate from POU filter.

The results from the filtration tests of five-hour recirculated slurry using 0.50 µm and 1.0 µm depth filters are presented in Figure 3. The cumulative % retention of the particles ≥0.56 µm in single-pass tests using CS05, CMP1 and SLR01 was close to 55, 38 and 37 %, respectively. Typical single-pass target retention % may range from 30 to 90% in CMP slurry filtration. In some slurries, more than one pass through the filter may be essential to achieve the target retention level. However, it is important to note that extremely high retention level usually results in limited filter lifetime and very high Δp. In most real-life slurry filtration applications, the objective is to have a reasonably tighter retention of large particles with acceptable filter lifetime. The selection of filters for a specific slurry requires empirical study. Although prior experience with similar slurries may indicate the filter requirements, the results below demonstrate that the performance of a filter can vary greatly across slurries due to abrasive particle morphology, LPC, PSD, wt % solids, particle settling characteristics, and chemical content and nature of additives and oxidizers.

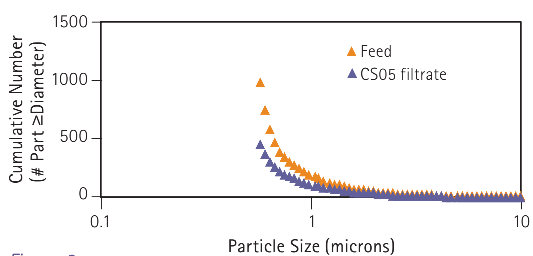

Figure 3a.

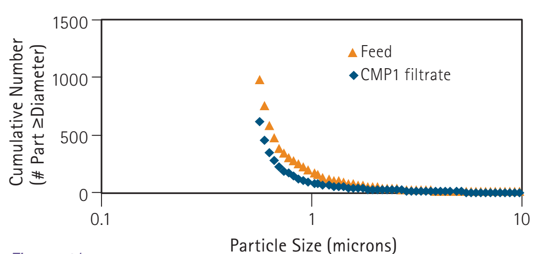

Figure 3b.

Figure 3. LPC for 0.5 µm (CS05) and 1.0 µm (CMP1) nominal rating depth media filters in single-pass filtration test.

Figures 4 and 5 show the filtration test results for CS05 and CMP1 filter media with silica-1, ceria-1 and alumina-1 slurries. The feed PSD data show large differences as may be expected from different slurries. The filters' effectiveness in reducing LPC shows considerable variation across the three slurries. For example, the CS05 filter seems to be very effective in removing large particles from the alumina-1 slurry, but less so for the ceria-1 slurry, despite both slurries having comparable low wt % solids. The silica-1, ceria-1 and alumina-1 slurries source samples were measured to have ~8×10^5, 2400×10^5 and 130×10^5 particles/mL (for size ≥0.56 µm), respectively, suggesting that these ceria and alumina slurries have close to 300 and 17 times, respectively, higher number of particles as compared to the silica slurry. Also, the silica-1, ceria-1 and alumina-1 slurries source samples were found to have ~1.4×10^5, 73×10^5 and 8.2×10^5 particles/mL (for size ≥1.10 µm), respectively.

The resulting LPC reduction, Δp and flow rate for each filter media are summarized in Table 1. The Δp and flow rates were obtained for the filters at 10 minutes after start of filtration tests. The experimental uncertainty in the measurement of LPC, flow rate and pressure drop measurements is estimated to be ±5%, ±10 mL/min and ±0.5 psi, respectively. The LPC reduction is presented in terms of % cumulative reduction of ≥0.56 µm particles for CS05 (0.5 µm rating) and ≥1.01 µm particles for CMP1 (1.0 µm rating) filters.

There are considerable differences in filter performance across the different slurries for CS05 as well as CMP1 filters. As seen in Table 1, alumina-1 and alumina-2 slurries with similar low % wt solids of abrasives showed similar retention and flow rate for CS05 media. A similar result of alumina-1 and alumina-2 slurries was seen for CMP1 filter. For CS05 media, silica-2 with similar wt % solids as silica-1, showed lower retention, much higher Δp and lower flow rate. Also for CS05, alumina-1 with similar wt % solids as ceria-1 resulted in much higher retention as well as higher Δp and a slightly lower flow rate. The LPC retention seen in ceria-1 is much lower than alumina-1 for CMP1 filter.

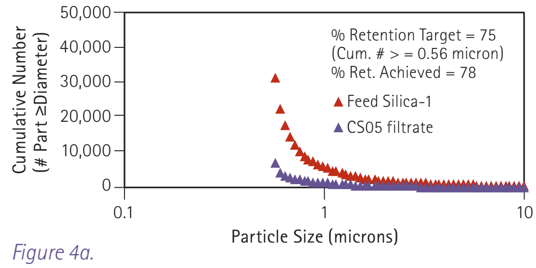

Figure 4a.

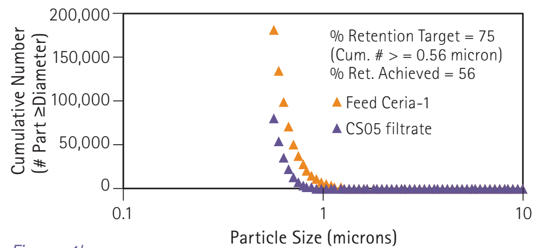

Figure 4b.

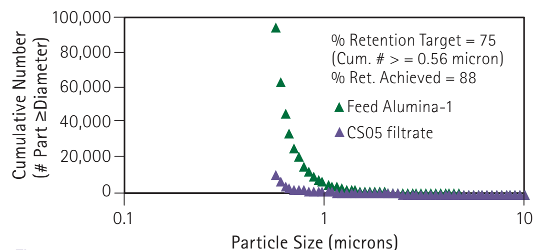

Figure 4c.

Figure 4. LPC for 0.5 µm (CS05) nominal rating depth media filters in single-pass filtration experiments. (a) Silica-1, (b) Ceria-1 and (c) Alumina-1 slurry.

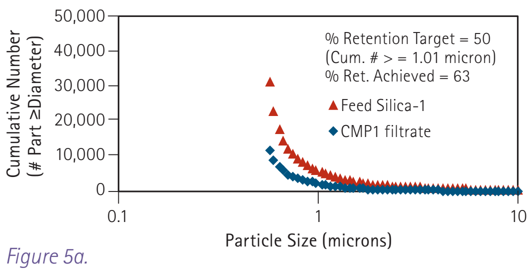

Figure 5a.

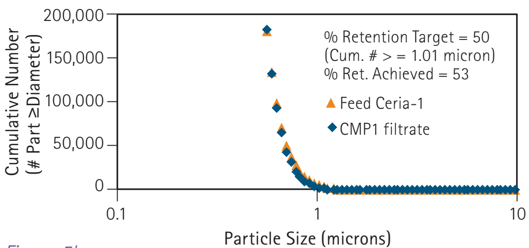

Figure 5b.

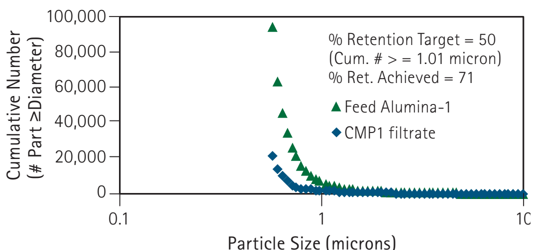

Figure 5c.

Figure 5. LPC for 1.0 µm (CMP1) nominal rating depth media filters in single-pass filtration experiments. (a) Silica-1, (b) Ceria-1 and (c) Alumina-1 slurry.

As expected, for CS05 as well as CMP1 filters the PSL bead challenge solution with negligible wt % solids as compared to slurries, showed lower retention, lower Δp and lower flow rates as compared to most of the slurries. The settling rate of various slurries[5] can be significantly different depending on the colloidal stability of the particles and their densities (e.g., silica, alumina and ceria abrasive densities are ~2, 4 and 8 g/cc, respectively). The mean particle size of the tested slurries ranged from 120 nm to 160 nm.

These results demonstrate that filter media large particle retention, pressure drop and flow rate are strongly influenced by the chemical additives and abrasive characteristics in the slurry, and that empirical filter characterization and optimization is essential for current and next generation CMP slurries.

TABLE 1. FILTERS SHOW LARGE SLURRY DEPENDENT VARIATIONS IN PERFORMANCE.

Slurry/ Challenge Solution	CS05 (Cumulative % LPC reduction for particles ≥0.56 µm)	Pressure Drop Δp (psi)	Flow Rate (mL/min)
Silica-1	78	40	127
Ceria-1	56	12.7	469
Alumina-1	88	19	450
Silica-2	90	28	275
Alumina-2	83	14	458
PSL Bead Solution	62	11.8	500

Slurry/ Challenge Solution	CS05 (Cumulative % LPC reduction for particles ≥1.01 µm)	Pressure Drop Δp (psi)	Flow Rate (mL/min)
Silica-1	63	7.8	423
Ceria-1	53	5.2	519
Alumina-1	71	3.5	535
Alumina-2	69	5.2	531
PSL Bead Solution	36	4.4	535

Conclusion

Current- and next-generation chemical mechanical planarization slurry filtration targets tighter retention of large particles at much smaller large-particle cutoff to achieve improved polishing performance. Graded density depth filters can be effectively used to manage large particle behavior in these slurries. Optimum filter design should consider slurry abrasive morphology and composition, chemical additives, large and mean particle distributions, wt % solids, viscosity, density, abrasive settling, pressure drop, flow rate, filter lifetime and cost of ownership. Large particle retention, flow rate and pressure drop across the filters in filtration tests using tighter graded density depth media show dramatically different behavior in the PSL bead challenge solution, and silica, alumina and ceria abrasive slurries, indicating that new CMP slurries filter optimization still remains empirical in nature.

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