Chemical Mechanical Planarization (CMP) Slurry Manufacturing

Introduction

The Chemical Mechanical Planarization (CMP) process plays a key role in the manufacture of data storage, video display panels, and semiconductor chips. The process combines the chemical (acidic or basic) effect of the slurry, which contains micro-abrasives with the mechanical effect provided by polishing to reduce the topography on the wafer or rigid disk substrate. The process for manufacturing integrated circuits (IC) uses a “tool” with a rotating wafer carrier (single or multi-head) and a polishing pad placed on a rotating platen. Wafers are held in the carrier and pressed against the polishing pad onto which chemical abrasive polishing slurry is dispensed (Figure 1).

Integrated circuits contain multiple levels, or layers of circuitry separated by insulating dielectric layers (ILD). Multilevel layers reproduce up areas and down areas from one level to the next. As each new metal layer gets added it magnifies the layer just below it. As more and more layers are built on the wafer surface the problem with non-planarity (Figure 2) becomes more severe resulting in poor step coverage of deposited films and impaired depth of focus of high resolution photolithography. CMP flattens out these uneven areas and allows subsequent photolithography to take place with greater accuracy, which allows film layers to be built up with minimal height variations. Similarly, the CMP process used for rigid disk substrates results in a high quality, mirror like finish that enables high density data storage onto the disk drive.

This Application Brief reviews the proper filtration methods that can be employed during the manufacturing of slurries to reduce large particles. Benefits to the slurry manufacturer include a highly consistent quality of slurry, increased throughput, revenues, and overall equipment effectiveness.

The Process

The process of manufacturing slurries starts with two or more raw materials that go through a dilution and blending process. Dilution occurs with DI water that has been filtered to reduce any particulate from the DI water that could contaminate the raw materials and ultimately the slurry. After dilution and blending, the slurry is fed to a storage tank and then to the filling station where drums and totes are filled for customer use. Prior to filling, the slurry is filtered through multiple (2 – 4) stages of progressively tighter filters. Raw material and post blending filtration systems typically utilize lower efficiency filter bags and cartridge filters; whereas, the final filtration stages utilize high efficiency filter cartridges and capsules. Figure 3 illustrates a typical slurry manufacturing process.
The Problem
Meeting the strict particle specification for each specific slurry type is a somewhat difficult task for the material supplier. Issues of concern include large particle counts, percent solids, and slurry consistency from lot to lot. In addition, slurry manufacturers desire that the entire batch be processed without having to replace the filter cartridges. This is critical as interruption of the filling process can significantly decrease production while increasing operating costs. Being able to filter an entire batch of slurry without having to shutdown for filter change-out can provide significant cost savings over the course of time.

Large Particle Counts (LPCs)
CMP slurries typically contain particles composed of alumina, silica, and ceria which are suspended in an acidic or basic solution. During the manufacturing process the suspended particles can settle and form aggregates, agglomerates, and gels. These “oversized” particles (Figure 4) are commonly referred to as large particle counts (LPCs). Of all large particle counts aggregates and agglomerates are the most detrimental because they can scratch inter-level dielectrics and metal layers potentially increasing overall wafer level defectivity (Figure 5). The source of large particle counts in the slurry can be categorized as either introduced to the process or created by the process.

Introduced to the Process - Oversized particles can be introduced through the raw materials and the DI water used for dilution. These process external sources are easily identified and controlled.

Created by the Process - Oversize particles created during production are often the result of the following:

1. Excessive shear during the blending process
2. As the fluid level drops in the storage tank slurry can dry and flake off
3. pH shock
4. Temperature changes
5. Settling
6. Interaction with other components in the process like fittings, valves, and pumps
7. Improper Filtration
Particle Size Distribution (PSD)

The filtration of CMP slurries is a unique and challenging process as compared to the filtration of chemicals used in electronics manufacturing. High purity chemical filtration is typically performed using 0.2 micron or tighter membrane filters that have a sharp particle reduction cut-off at the rated pore size. Since there are multiple types and manufacturers of slurry the “desired” mean particle size can range from 0.03 – 0.2 micron. Consequently, the filter that was specifically designed for particle clarification of high purity chemicals would strip out the desired particles and adversely affect the polishing characteristics of the slurry. Oversized particles in the slurry typically greater than 0.56 micron represent the “tail” of the PSD (Figure 6). If the PSD tail is not removed it can affect repeatability of the planarization process and potentially scratch inter-level dielectrics resulting in an increase to overall wafer level defectivity. While reduction of the oversized particles is important, it must be accomplished without altering the percent solids of the slurry.

Particle Reduction Characteristics

Depending on the location in the slurry manufacturing process both a filter with a sharp (classifying) cut-off and a filter with a broad (clarification) cut-off will be required to reduce large particles. For example, during the final filtration stage a classifying filter with a sharp cut-off that is slightly larger than the desired particle size distribution will be required. Figure 7 shows that a 0.5 micron classifying filter reduces nearly all particles 0.5 micron and larger while allowing nearly all particles 0.5 micron and smaller to pass through the filter. The proper classifying filter at the final filtration stage will not reduce the desired working size particles or alter percent solids thus preserving the polishing characteristics of the slurry. In the post blending stage, the application requires a clarifying filter with a broad reduction range like that depicted in Figure 8. The post blending filter should also contain a more porous media than at final filtration as it needs to reduce agglomerates and gels that were recently formed during the dilution and blending process. A clarifying filter will reduce the undesired PSD tail over a wider (1.0 - 10.0) micron range without sacrificing flow or filter lifetime.

“Matching” the slurry to the proper post blending and final filters will provide the peak level of performance needed to dramatically reduce defect causing large particles.
The Solution

Major reductions in large particle counts can directly be associated to proper filtration, resulting in a consistent quality of slurry that enables repeatability of the planarization process. The proper filtration system requires both a filter design that will “classify” and “clarify” the slurry at the post blending and final filtration stages. Graded porosity depth filters are the most efficient and cost effective means of filtering slurries. The varying porosity of the filter media, when properly selected, will retain the oversized particles and allow the desired “working” particles to pass through while maintaining the polishing characteristics of the slurry. Betapure™ CMP filters meet these exacting requirements.

The slurry manufacturing process contains four important filtration points (see Figure 3). They include filtration of the DI Water, Raw Materials, Post Blending, and Final Filtration prior to drum and tote filling of the slurry. 3M Purification Inc. offers customized solutions for each stage of the slurry manufacturing process.

Location 1 – DI Water

The filtration of the DI water is most effectively accomplished by a two-stage filtration system that consists of large surface area, high efficiency filter cartridges that will retain unwanted particles before they can potentially contaminate the CMP slurry during the blending stage. Water quality varies greatly based on the incoming feed, for this reason a pleated membrane filter with an absolute rating of 0.2 or 0.1 micron will provide the best combination of efficiency, contaminant holding capacity, and service life for the pre-filtration stage. In addition to those attributes, the final filter should be rated at 0.04 micron and contain a charge modification to provide enhanced particle reduction characteristics of submicron contaminants like colloidal silica and bacteria fragments.

3M Purification’s 0.2 and 0.1 micron, absolute rated LifeASSURE™ IMC filter cartridges with Advanced Pleat Technology manufacturing (APT, depicted in Figure 9) and LifeASSURE™ EF series filters were specifically designed for electronics grade DI water applications, providing the best combination of enhanced particle reduction efficiency, long filter life, and superior flow characteristics. These absolute rated filters ensure that unwanted particles in the DI water system are not allowed to contaminate the CMP slurry.

Location 2 – Raw Materials

Filtration of the raw materials is best accomplished by an economical, rigid depth structure that has a controlled pore size like Betapure™ filter. Raw material filtration with Betapure filter cartridges rated from 2 to 100 micron is recommended prior to the dilution and blending stage. A non-rigid filter’s pore structure changes as the system differential pressure increases resulting in changes to contaminant reduction efficiency and filter lifetime. The rigid filter structure of Betapure retains a consistent pore size even under severe process conditions like pump fluctuations, stopping and restarting the system, or high differential pressure.

Location 3 – Post Blending

The post blending filter is designed to extend the lifetime of the final filters by reducing large particles that were previously formed during the dilution and blending stages. During dilution, when the raw materials and DI water are combined, it is common to see the formation of agglomerates resulting from pH shock and temperature changes to the slurry. Large particles can also form as a result of excessive shear during the blending stage. Depending on the percent solids and type of slurry either a 5 or 10 micron (CMP560
or CMP570) clarifying depth filter with a graded porosity structure like Betapure™. CMP filter is recommended prior to delivery to the final filters. Effective reduction of agglomerates and other large particles by Betapure CMP filters not only reduces the particle loading on the final filtration system but also reduces the number of available sites for particle agglomeration during the final filtration stages. As a result the particle loading on the final filtration system is greatly reduced and the likelihood that the final filtration process is accomplished without having to replace the filter cartridges is greatly improved. In the long run, effective post blending with Betapure CMP filters, improve production throughput.

**Location 4 – Final Filtration**
The final filters are the most critical, capturing unwanted oversized particles prior to drum and tote filling. Graded porosity depth filters with a sharp retention profile are the most efficient and cost effective means of final filtration for CMP Slurries. The varying pore sizes of the filter media, when properly selected, will retain the undesired or oversized particles while allowing the desired particles to pass through thus preserving the polishing characteristics of the slurry.

Betapure CMP filters are offered in a wide range of pore sizes making them ideal for all types and stages of slurry filtration. The high efficiency, low shear, graded porosity construction of Betapure CMP filters provide effective classification of the slurry, reducing the oversized, undesired particles while maintaining the desired particle size distribution. The novel design and superior flow characteristics of the Betapure CMP filter provides enhanced contaminant holding capacity which allows for a properly sized system to filter the entire batch of slurry without requiring multiple filter cartridge change-outs. Figure 10 shows the graded porosity design of the Betapure CMP filter media.

### The Filter Recommendations

<table>
<thead>
<tr>
<th>Filtration Location (see Figure 3)</th>
<th>Recommended Filter</th>
<th>Micron Rating (µm)</th>
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<tbody>
<tr>
<td>(1) DI Water – Stage 1</td>
<td>LifeASSURE™ IMC</td>
<td>0.2 or 0.1</td>
</tr>
<tr>
<td>(1) DI Water – Stage 2</td>
<td>LifeASSURE™ EF series</td>
<td>0.04</td>
</tr>
<tr>
<td>(2) Raw Materials</td>
<td>Betapure™</td>
<td>2.0 - 100.0</td>
</tr>
<tr>
<td>(3) Post Blending</td>
<td>Betapure™ CMP</td>
<td>5.0 - 50.0</td>
</tr>
<tr>
<td>(4) Final Filtration</td>
<td>Betapure™ CMP</td>
<td>0.3 - 3.0</td>
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### The Conclusions
The slurry manufacturing process contains four important filtration points which include DI Water, Raw Materials, Post Blending, and Final Filtration. 3M Purification offers filtration solutions for the necessary steps required to ensure that the slurry meets particle count specifications thereby reducing the potential for micro-scratches.

1. Utilizing high efficiency filters noted in “The Solutions” section above will reduce the potential for significant defects to occur on the electronics devices. Scrapping of a 300-mm wafer due to micro-scratches, or ineffective polishing because the desired particle size distribution has been altered, can result in as much as $150,000 in lost revenue to the device manufacturer.

2. Major reductions in large particle counts can directly be associated to proper filtration, resulting in a consistent quality of slurry that enables repeatability of the planarization process. The proper filtration system requires both a filter design that will “classify” and “clarify” the slurry at the post blending and final filtration stages. “Matching” the slurry to the proper post blending and final filters will provide the peak level of performance needed to dramatically reduce defect causing large particles.

3. Betapure CMP filters used in the final filtration stages allow the specifications of the slurry to be met quickly and effectively in a single pass. Betapure CMP filters allow for a properly sized system to filter the entire batch of slurry without requiring multiple filter cartridge change-outs.
SASS

3M Purification Electronics performs in-house and on-site filtration studies worldwide through its Scientific Applications Support Services (SASS) group using the most advanced particle counting technologies (PSS-780A) to optimize the CMP process for the integrated circuit or data storage manufacturer. In addition, 3M Purification Electronics market team works closely with the slurry manufacturers and end users to characterize and customize filtration solutions that meet current and future requirements.

The Reference Material and Related Product Information

**LifeASSURE™ EF Series Filters** – LifeASSURE EF series filter cartridges deliver superior flow performance and advanced particle retention for ultra high purity water applications. Utilizing 3M Purification’s charge modification technology the filter combines both mechanical sieving (pore size) and electro-kinetic adsorption (positive charge) to enhance particle reduction capability of submicron contaminants like colloidal silica and bacteria fragments. LifeASSURE EF series filters are absolute rated, naturally hydrophilic, charge modified Nylon 6,6 membranes incorporated into an all polypropylene cartridge. Combined with 11 ft² of filter surface area per 10” cartridge, long life, high flow rates, and low pressure drop are realized with LifeASSURE EF series filters. A quantum leap in process economies is now within the reach of every LifeASSURE EF series filter user. For more information please ask for 3M Purification literature LITZREL02.

**LifeASSURE™ IMC Series Filters** – LifeASSURE IMC series filter cartridges are high efficiency naturally hydrophilic Nylon 6,6 filter elements designed to meet the exacting requirements of macro-electronic applications. Utilizing 3M Purification’s Advanced Pleat Technology (APT) and FlexN Multi-Zone Membrane, LifeASSURE IMC series filter cartridges provide much longer filter life with lower pressure drop when compared (see product data sheet) to other membrane cartridges. Designed with pleated Nylon 6,6 membrane in an all polypropylene construction, LifeASSURE IMC series filter cartridges are ideally suited for DI Water and Parts Cleaning applications. For more information please ask for 3M Purification literature LITZROPT1.

**Betapure™ Filter Cartridges and Capsules** – Betapure filters utilize state-of-the-art technology to produce a clean, rigid, filter structure with consistent and reproducible filtration characteristics for Pre-RO, CMP Slurry Raw Materials, and Ceramic Slurry applications. The filter matrix is constructed using long bi-component fibers, each fiber having an inner core and an outer sheath (See product data sheet). Betapure filters are available in two bi-component fiber structures, polypropylene/polyethylene or polyester/co-polyester, to provide the greatest range of chemical compatibility.

The bi-component fibers of the filter matrix are thermally bonded by utilizing the difference in melt temperatures of the two fiber components. Heating the matrix to the melt temperature of the
polyethylene sheath, but below that of the polypropylene core causes the fiber-to-fiber bond at every contact point. The high degree of fiber-to-fiber bonding provides a rigid structure that reduces the need for a core support and any possibility of media migration. Dynamic applications where the filtration system is pulsed, cycled, or exposed to high differential pressures can cause non-rigid filters to unload contaminants back into the process fluid. The rigid structure of Betapure™ filter resist deformation, particle unloading, and by-pass to deliver consistent particle reduction efficiency even under adverse operating conditions. For more information please ask for literature LITCBP001.

**Betapure™ CMP Filter Cartridges and Capsules** – Betapure CMP filters are high capacity filters optimized for oxide and metal slurries used at point-of-use for chemical mechanical planarization applications. Betapure CMP filters are composed of all-polypropylene components and features a multi-zone “graded porosity” design for the peak level of particle classification. This novel construction provides enhanced flow characteristics, including low pressure drop, to minimize shearing of the slurry while providing superior filter life. Betapure CMP capsules are offered with Flaretek® and NPT fitting connections in lengths of 4”, 10”, 15”, and 20”. For more information please ask for 3M Purification literature LITCCMPCTG1 and LITCCMPCAP1.
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