Benefits and Limitations of Slurry Particle Analysis and the Need for Next Generation Capabilities

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Particle size analysis can play a major role in slurry troubleshooting, filtration and manufacturing optimization and wafer defect reduction.

Particle size analysis can also play an integral part in evaluation of various parts and slurry distribution components.

Pumps, valves, distribution systems and slurry piping designs are only a couple of examples of where particle analyzers can be beneficial in the evaluation of various components of fab systems.
Mean Particle Analysis

- Dynamic Light Scattering (DLS) also known as Photon Correlation Spectroscopy (PCS)

(Animation Courtesy of Agilent Technologies, Particle Sizing Systems Division)
Large Particle Analysis has shown to be more beneficial in slurry troubleshooting and evaluations than analyzers that only give mean results.

(Animation Courtesy of Agilent Technologies, Particle Sizing Systems Division)
### Slurry Trouble Shooting Methods

**“Large Particle Analysis – One of the Most Important Analyses”**

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Typical field CMP Issues Encountered:

- High defects/scratches on wafer
- Change in polishing performance (RR, WIWNU)
- Shorter filter lifetime or reduction in lifetime

Associated causes:

- Poor slurry handling methods (i.e. lack of humidified nitrogen blankets), which may lead to increased LPC’s and gels
- Lack of slurry distribution PM’s
- Filtration not optimized

Slurry particle size analysis plays an instrumental role in troubleshooting these issues
Terminology of Particles Observed with SEM, ESEM & Light Microscope

- **Particle**: single solid sphere or other geometry
- **Aggregate**: multiple particles chemically attached to each other (chain)
- **“Agglomerate”**: particles and aggregates come together into close-packed clumps
- **Microgel**: aggregates link together and form three dimensional network with water trapped within (Filter Plugging Problems)

Oxide Defects: “Large Particles” in Slurry May Create Micro-Scratches & Embedded Particles on Wafer

Typical defects in oxide CMP
Ref.: C. Dennison, KLA-Tencor, Micro, February 1998
Possible Sources of Large Particle Issues

Agglomerations
Silica slurry was diluted with pH adjusted water, then filtered through a 3 micron Isopore membrane.

Gels: Another Source of Large Particle Issues

Silica slurry was diluted with pH adjusted water, then filtered through a 3 micron Isopore membrane.
Large Particles & Cu-CMP Defects

- Optical image (Compass)
- SEM image (SEMVision)

- Large Chatter Marks
- Gouge
- Micro Scratch
Large Particles & Cu-CMP Defects

Large Scratch

Image Courtesy of MIC Lab

Brightfield

Darkfield

SEM
Bar = 2µ

20x DF
Possible Sources of Large Particle Issues

- Slurry shear (i.e. certain valves, pumps, piping design)
- Lack of humidification (wet N\textsubscript{2} blankets)
- Temperature issues during shipping or storage
- Settling
- Improper mixing of incoming slurry containers (pails, drums, totes)
- Large # of unnecessary turnovers
- pH shock during dilution
- Poor cleanliness of various system components (i.e. dip tubes, bung lids, filter housings systems, etc.)
- Agglomerations
Possible Sources of Large Particle Issues

- Lack of humidified nitrogen blankets

**Field Example:**
Fab unaware that system was off.
Possible Sources of Large Particle Issues

- Settling
- Improper mixing
Foreign particles (e.g. drum stirrers not properly adjusted can scrape the side of the drums and introduce large plastic particles)
Possible Sources of Large Particle Issues

- Local drying in shipping container, tank, and fittings

Field Example:
- Drum was left open to atmosphere
- A lot of dry slurry particles all over the top of the drum and the manual pump.
Possible Sources of Large Particle Issues

- Local drying of tote dip tube and lack of PM’s.
- Source of particle introduction
Another source of large particles that may be introduced during filter change out
Large Particle Counters Play Integral Part in Pump and New Technology Evaluations
Large Particle Size Plays Role in Pump Selection

- Technology has come a long way since the below graph published back in July 1998.

![Graph showing Large Particle Population Distribution with counts against particle diameter for different technologies: Control, Vacuum-Pressure Technology, Diaphragm Pump Technology, and Bellows Pump Technology.](image-url)
Correlating LPC’s to Defectivity and RR

- Large Particle Counters Play a Critical Role in Evaluating Alternative Technologies
LPC’s Key for Valve Technology Evaluations

OLD TECHNOLOGY

Tongue & Groove caused areas for Slurry to cake-up and cause premature failure and leak-by

NEW! TECHNOLOGY

- Enhanced Flow Characteristics
- No Areas for Entrapment or Agglomeration

Slide Courtesy of Parker Hannifin Corp.
Filter Ratings

- Beta Ratio
  \[ \beta = \frac{\text{Nu}}{\text{Nd}} \]
  where Nu is the number of particles / mL from the unfiltered upstream
  Nd is the number of particles / mL from the filtered downstream

- LRV (Log Reduction Value)
  \[ \text{LRV} = \log\left(\frac{\text{[Feed]}}{\text{[Filtrate]}}\right) \text{ in particles/mL} \]

- Percent Retention or Retention Efficiency
  \[ \% \text{ Retention} = (1 - \frac{\text{Filtrate}}{\text{Feed}}) \times 100 \]
  or if know beta ratio is same as \( (1 - \frac{1}{\beta}) \times 100 \)

- Efficiency can be derived from a known Beta ratio
  \[ \text{Ex} = (\beta x - 1) / \beta x \times 100 \]
  where Ex is the efficiency for a given particle size

Note: All these methods rely on large particle analysis
Large Particle Analysis is Critical for Creating Retention Curves

- Filter Retention Curves Play an Integral Role in Filter Selection

(Retention Curves Courtesy of Entegris and ZenPure)
Large Particle Role in Filter Optimization

- **Blue** – Unfiltered Sample
- **Red** – Single Pass Global Loop Filtered
- **Black** – Multiple Pass Global Loop Filtered

Particle Concentration Diff. Dist.

Diam. (um) ->

noFilt201 ISISD0.201 ISISD8.201
Defectivity Correlates with Large Particle Analysis

- Note: However, this is not always the case. It is possible to have large soft particles that do not increase wafer defects.
Limitations and Future Needs
Some Limitations

- There are many particle counters available on the market today (i.e. disc centrifuge, light scattering, light extinction, acoustic spectroscopy and CHDF technologies).

- Some counters are not as user-friendly and there is a lot of art involved in getting good repeatable data. Data can vary greatly from operator to operator. Critical to have good training programs covering techniques and SOP’s.

- Many tools lack good SOP’s and therefore on many occasions need to be developed in house.

- Some tools are prone to cavitation and degassing liquids more than others. This can skew results and decrease repeatability.

- Challenge when comparing data from tool to tool.

- However, with that said, Large Particle Analysis is still one of the best analytical methods of choice:
  - To aid in slurry trouble-shooting
  - Slurry filter optimization and recommendations
  - Equipment and component evaluations
Future Needs

- Analyzers that can detect large particles at lower micron ranges.
- Sub 0.1µ detection capability is needed for future nodes of chip technology with high resolution capability.
- Better tool-to-tool repeatability is needed.
- One area of limitation: particle hardness. (Large soft particles that do not impact wafer defectivity)
- Soft vs. hard particles is one area missed by large particle counters. Therefore, can’t assume a larger particle tail will automatically give poor wafer results. Furthermore, cannot assume the slurry is bad because the LPC’s were higher on one tool compared to another.
- Optimized liquid stream handling for improved results and efficiency.
- A method to determine particle hardness in a liquid stream would be of great benefit to the industry if one can be developed.
References


ROHM HAAS | ELECTRONIC MATERIALS

- Circuit Board Technologies
- CMP Technologies
- Display Technologies
- Microelectronic Technologies
- Packaging and Finishing Technologies