The Impact of Sample Containers on Large Particle Count for CMP Slurries

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Overview

• Problem statement
• LPC sources and measurement
• Sample bottles and its evaluation
• Identification of the sources of particles
• Summary
Problem Statement

Problem: A larger variation of LPC was observed for slurry A containing surfactant when sample bottles w/ cap liner was used.
LPC and LPC Sources

- Large Particle Count (LPC) in CMP slurries
  - Particles >0.5 μm
  - LPC is typically measured to predict defects
- Possible sources of LPC:
  - In the slurry: large particles, agglomerates, micelles
  - Extraneous sources: contamination
- In this study LPC contribution from sample bottles was identified and reduced
Variations in LPC Measurement

• Sources of variations in LPC measurement
  • Taking samples
  • Handling samples
  • Preparing samples
  • Stability of LPC measurement tool
• Previous LPC studies at Fujimi:
  • Settling and re-dispersion of large particles
Settling of large particles and its re-dispersion of particle was demonstrated.

Sample preparation right before LPC measurement is important to obtain an accurate measurement.

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Bottles Used

• Bottles tested
  • Bottle w/o cap liner (Bottle A)
  • Bottle w/ cap liner (Bottle B)

HDPE: high density polyethylene
PP: polypropylene
Slurry for This Study

• Slurry A
  • HVM slurry: colloidal particle / surfactant
    • Slurry at pH 10

• A typical LPC measurement variation of slurry A including variations by LPC measurement operator and date

<table>
<thead>
<tr>
<th>Particle size (um)</th>
<th>Particle counts</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;0.56</td>
<td>+/- 343</td>
</tr>
<tr>
<td>&gt;0.99</td>
<td>+/- 50</td>
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</tbody>
</table>
Sample Handling

- Two operators collected samples directly from a production line and delivered to Quality Control (QC)

<table>
<thead>
<tr>
<th>Operator</th>
<th>Bottle handling method</th>
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</thead>
<tbody>
<tr>
<td>Operator 1</td>
<td>Bottles tipped over</td>
</tr>
<tr>
<td>Operator 2</td>
<td>Bottles kept upright from the production line to QC for LPC measurement</td>
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</tbody>
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Sample Preparation for LPC Measurement

- Sample preparation before LPC measurement
  - Slurry samples were shaken on an orbital shaker for 3 min right before sampling
  - Sampling from bottle during the measurement

- Instrument for LPC measurement: Laser-based custom system
Higher LPC was observed from slurry A in bottle B w/ cap liner.
Effect of Bottle Handling

- LPC for bottle B depended on how the sample bottle was handled.
  - Operator 1 tipped the bottle over — slurry contacted bottle cap.

Bottle B: w/ cap liner
Cap liner is demonstrated to be a significant source of LPC

Low LPC was observed from bottle B after the removal of cap liner
Large particles up to 15 μm were observed on a cap liner surface by SEM.
Cleaning Particles from Cap Liner

• Removal of particle from cap liner
  • Typical rinsing with DIW did not help to remove particles on the cap liners
Removal of Particle by Slurry A

- Particles removed by slurry A
  - Bottle B (w/ cap liner) tipped over with slurry A in the bottle
  - Large particles disappeared from cap liner
• By tipping over the bottle B particles on the cap liner could be pulled into the slurry causing higher LPC
Bottle A vs Bottle B

By using bottle A, LPC variation by sample collectors could be minimized.

<table>
<thead>
<tr>
<th>Particle size (um)</th>
<th>Bottle A</th>
<th>Bottle B</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;0.56</td>
<td>+/- 58</td>
<td>+/- 343</td>
</tr>
<tr>
<td>&gt;0.99</td>
<td>+/- 17</td>
<td>+/- 50</td>
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</table>
Summary

- LPC measurement in CMP slurry can be impacted by particles from cap liner of sample bottle
  - High LPC in slurry A was attributed to particles on a cap liner from bottle B
  - LPC from bottle B varied by how samples were collected
  - Uncertain variation by operators could be eliminated by using bottle A (w/o cap liner)

- To ensure that LPC is a valuable metric for monitoring slurry product quality, extraneous modes of LPC generation from sampling process need to be identified and eliminated
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