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

Brian Kim, PhD
Fujimi Corporation R&D

2017 CMPUG Spring Meeting
April 13th, 2017, Portland OR

FUJIMI INCORPORATED

FUJIMI CORPORATION

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

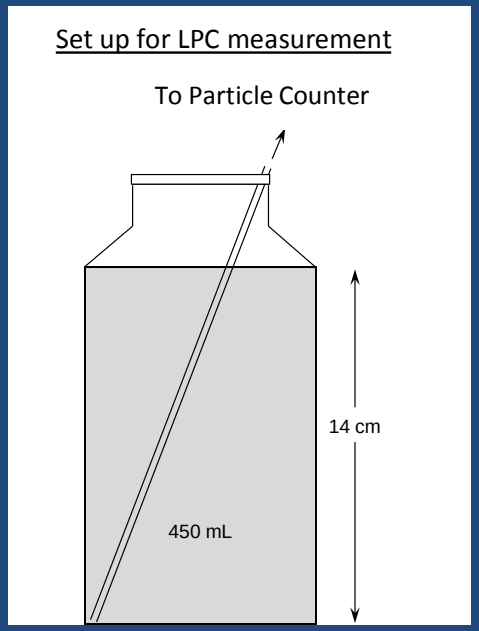
- Large Particle Count (LPC) in CMP slurries
  - Particles >0.5 um
  - LPC is typically measured to predict defects
- Possible sources of LPC:
  - In the slurry: large particles, agglomerates, micelles
  - Extraneous sources: contamination
- In this study LPC contribution from sample bottles was identified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

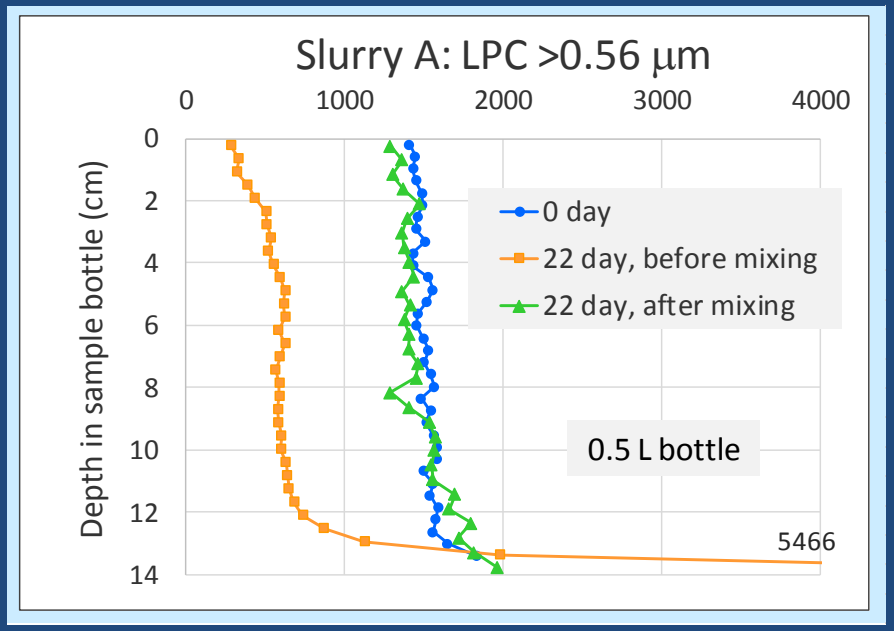
Variations in LPC Measurement

- 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

LPC as a Function of Depth in Sample Bottle

- 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





"Settling of Colloidal Silica Particles in CMP Slurry: Monitoring, Effect, and Handling" J. Lin, W.S. Rader, ***CSTIC 2017***, March 12-13, 2017, Shanghai, China.

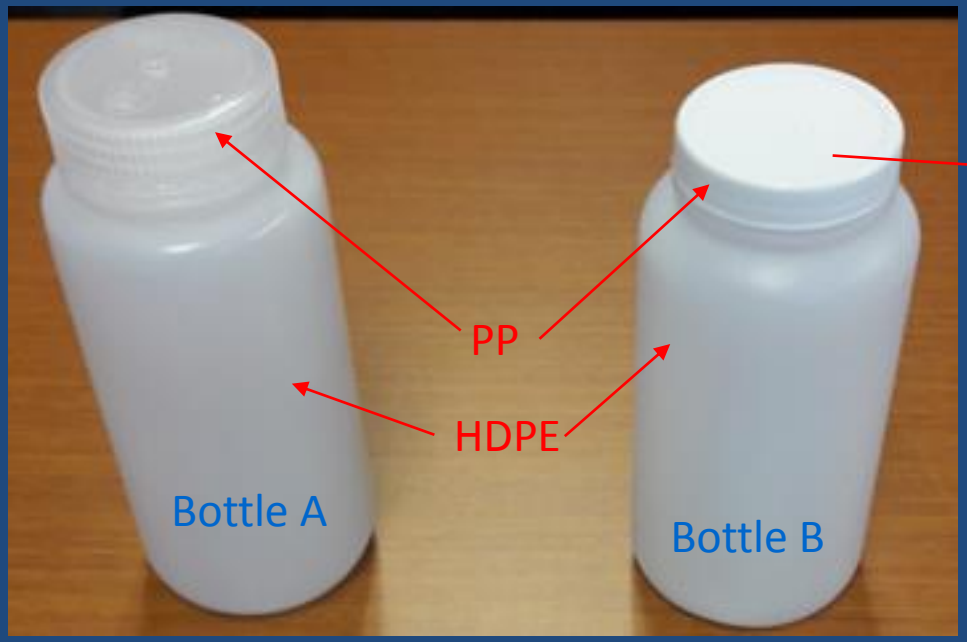
FUJIMI INCORPORATED

FUJIMI CORPORATION

Bottles Used

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

HDPE: high density polyethylene
PP: polypropylene



FUJIMI INCORPORATED

FUJIMI CORPORATION

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

Particle size (um)	Particle counts
>0.56	+/- 343
>0.99	+/- 50

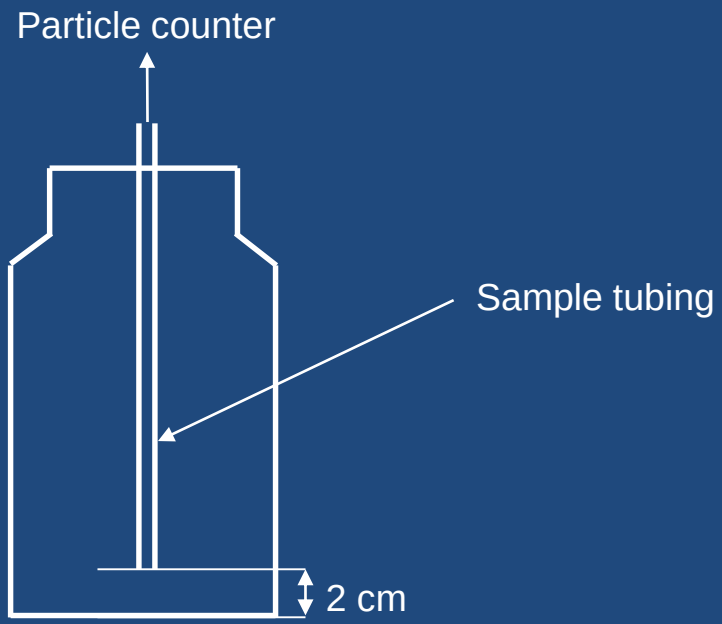
Sample Handling

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

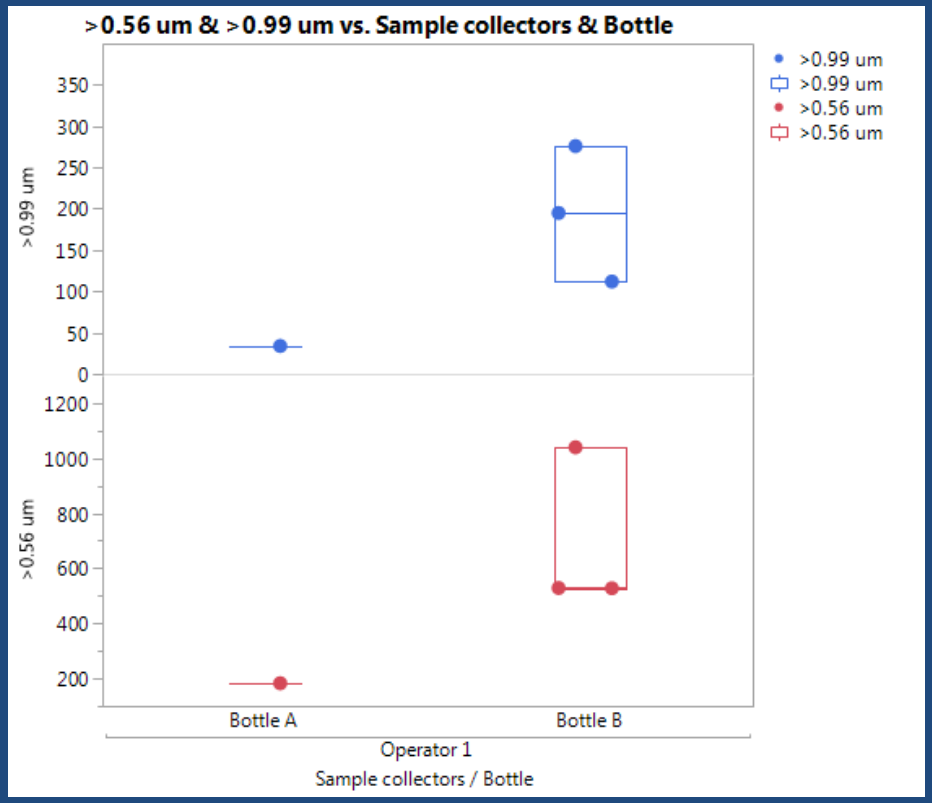
Operator	Bottle handling method
Operator 1	Bottles tipped over
Operator 2	Bottles kept upright from the production line to QC for LPC measurement

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



LPC of Slurry A from Bottles w/ or w/o Cap Liner



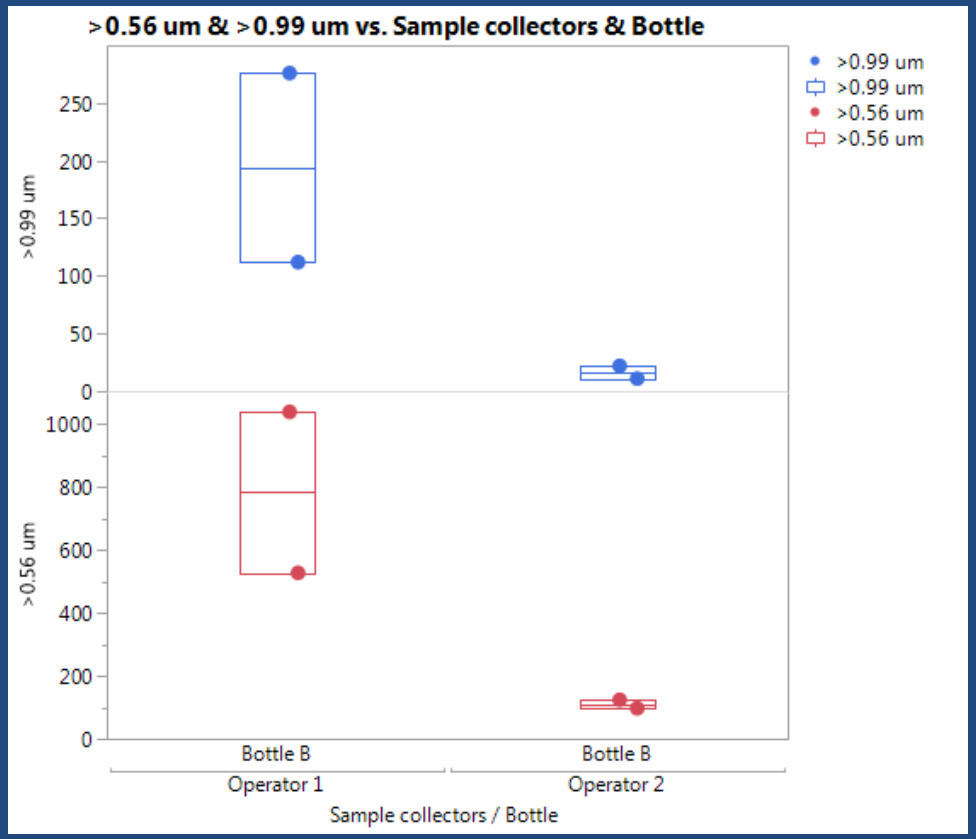
Bottle A: w/o cap liner
Bottle B: w/ cap liner

- Higher LPC was observed from slurry A in bottle B w/ cap liner

FUJIMI INCORPORATED

FUJIMI CORPORATION

Effect of Bottle Handling



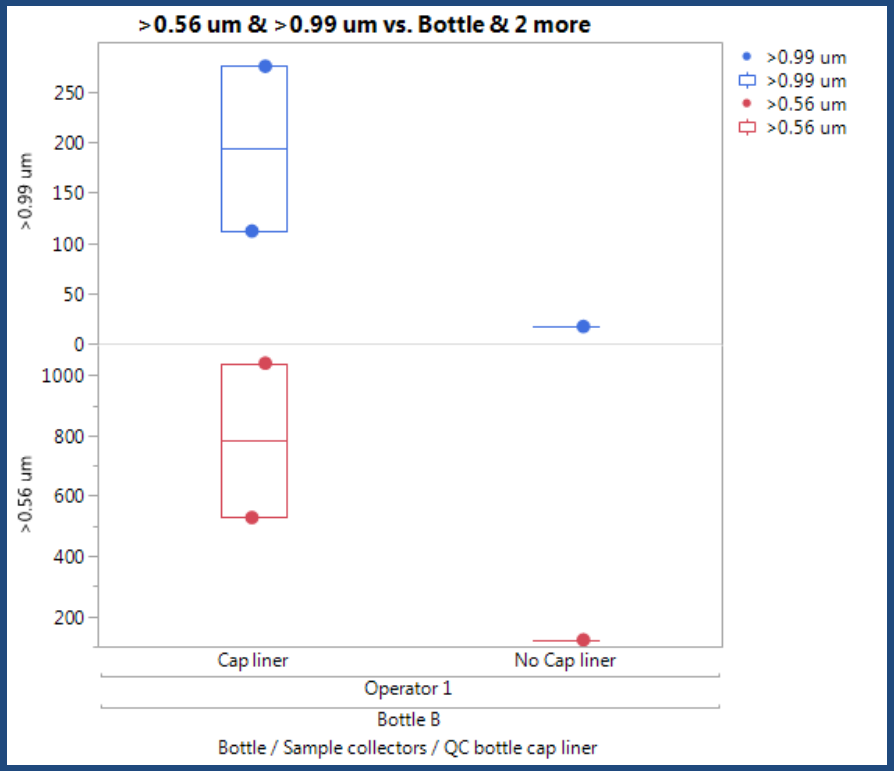
Bottle B: w/ cap liner

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

FUJIMI INCORPORATED

FUJIMI CORPORATION

Bottle B after Removal of Cap Liner

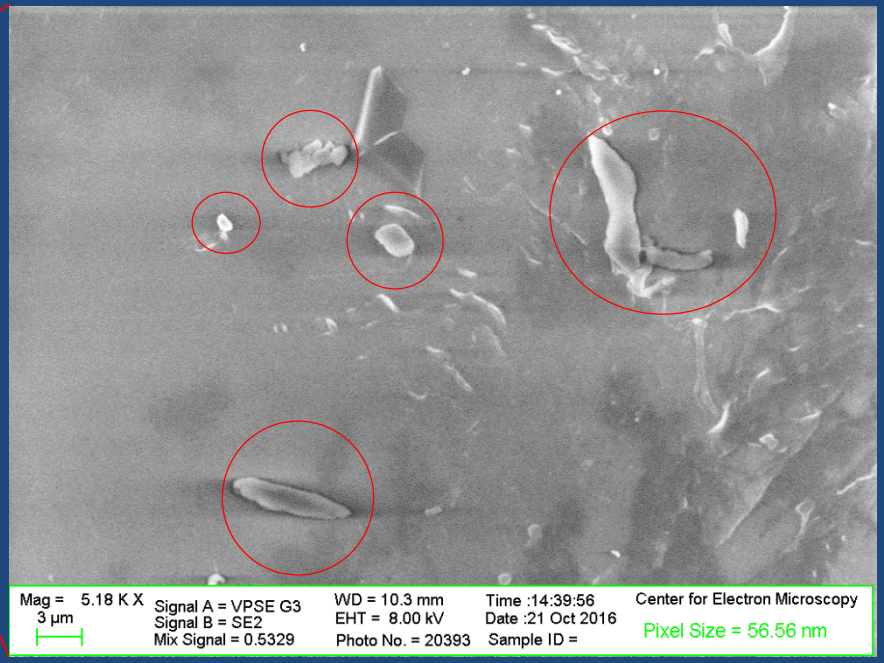


Bottle B: w/ cap liner
Bottle B: w/o cap liner

- Cap liner is demonstrated to be a significant source of LPC
  - Low LPC was observed from bottle B after the removal of cap liner

SEM Images for Surface of Cap Liner

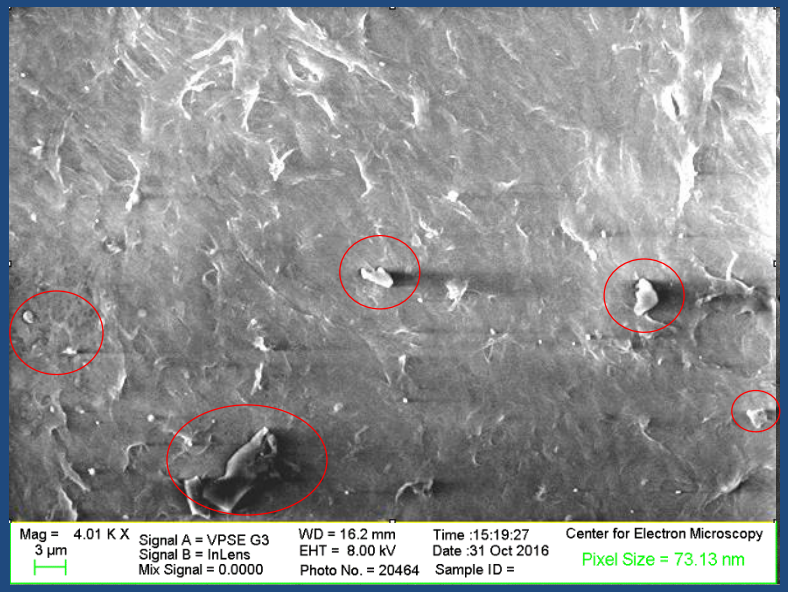
Cap liner



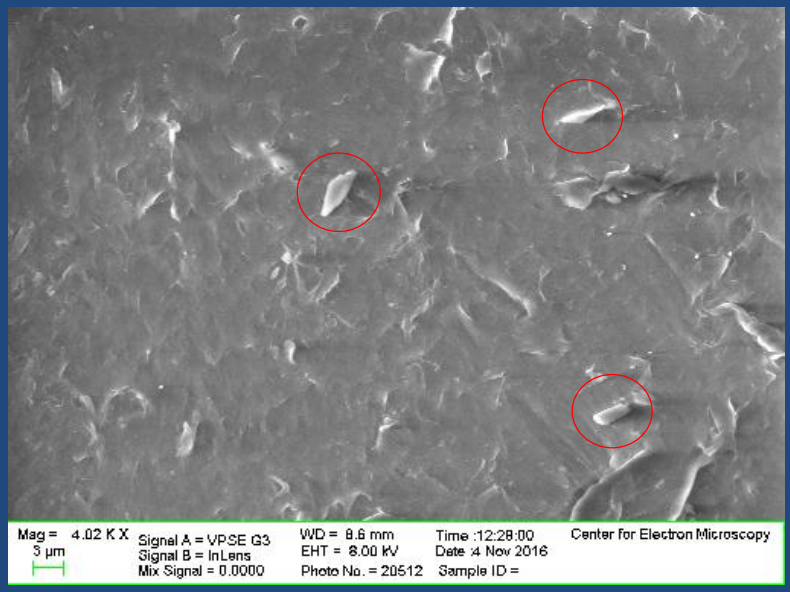
- Large particles up to 15 um were observed on a cap liner surface by SEM

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



Before cleaning



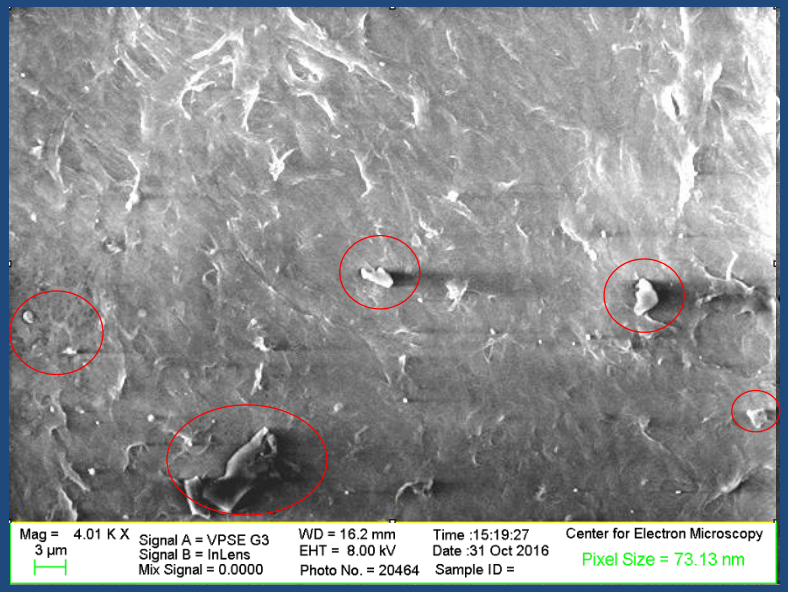
After cleaning

FUJIMI INCORPORATED

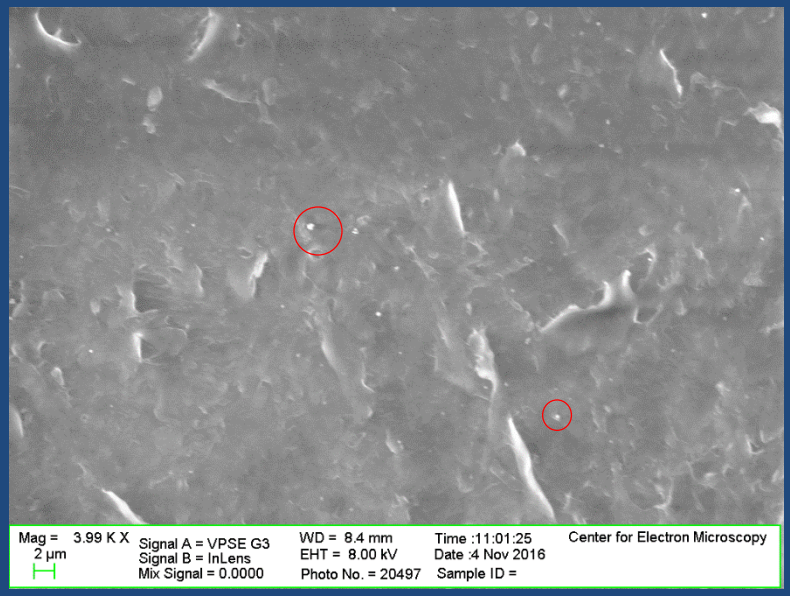
FUJIMI CORPORATION

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



Before contacting slurry A

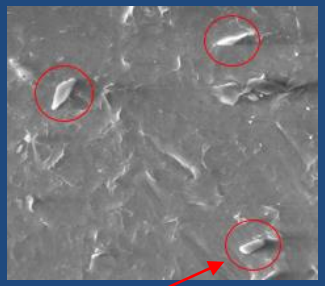


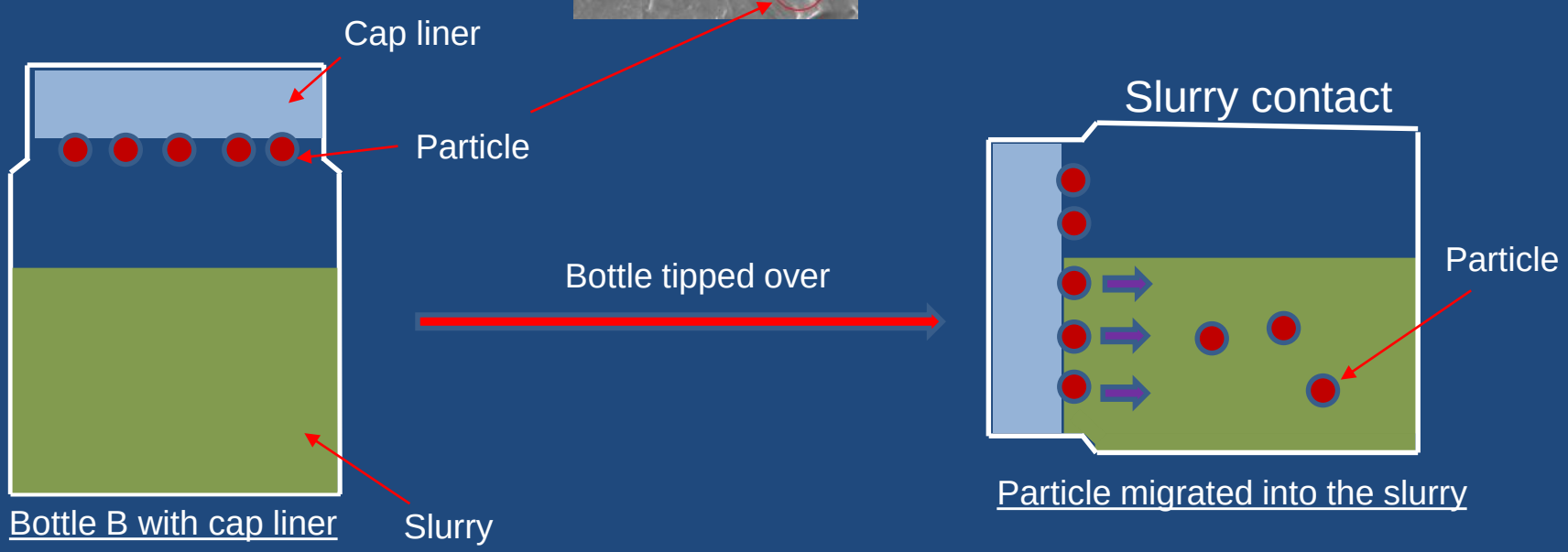
After contacting slurry A

FUJIMI INCORPORATED

FUJIMI CORPORATION

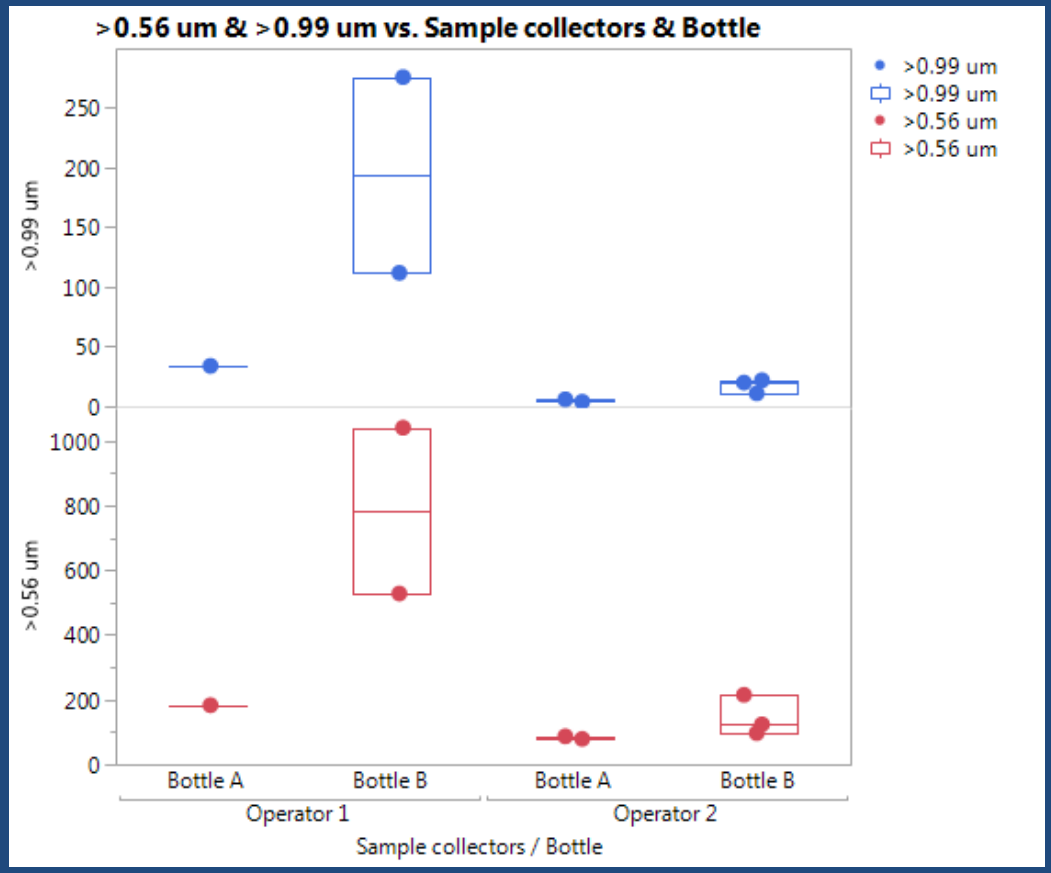
Particle from Cap Liner of Bottle B on LPC





- 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



Bottle A: w/o cap liner
Bottle B: w/ cap liner

Measurement variation of slurry A

Particle size (um)	Particle counts	
	Bottle A	Bottle B
>0.56	+/- 58	+/- 343
>0.99	+/- 17	+/- 50

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

FUJIMI INCORPORATED

FUJIMI CORPORATION

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

Bottle A: w/o cap liner
Bottle B: w/ cap liner

Acknowledgements

- The author thank colleagues at Fujimi Corporation, particularly Dr. Jie Lin, Dr. Haiyan Li, Dr. Jimmy Granstrom, Annette Schaper, Dr. Hisashi Takeda, Dr. Charles Poutasse, Dr. Anne Miller, and Dr. Scott Radar, for detailed discussions

FUJIMI INCORPORATED

FUJIMI CORPORATION