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Handling, Filtration and Polishing Performance Characterization of Next Generation CMP Slurries

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Overview

- Motivation and Objectives
- Why Characterize CMP Consumables?
- CMP Slurry Health Management Challenges
- Slurry Characterization, Blending and Distribution
- Slurry Filtration: Trends, Methodology and Mechanisms
- Typical Slurry Handling and Filtration Characterization Data
- Characteristics of New High-Purity Colloidal Silica Slurry
- Filtration, Polishing and Wafer Defectivity Performance Data
- Summary and Conclusions

Motivation and Objectives

- This paper reviews key considerations and challenges for CMP slurry characterization, blending, metrology, handling and filtration management, and explores characteristics of new, relatively smaller abrasive, high-purity colloidal silica slurries designed specifically for ULK dielectric layers.
- Above next generation slurries (Silco EM-5530K & EM-7530K) provide precise and consistent removal rates, minimal wafer defectivity, and maximum planarity across the wafer surface. Present study evaluates comparative performance of above slurries polishing rate, NU and particle defectivity using different CMP pads and other similar slurry products.
- Selective removal of large defect causing particles without affecting the mean particle distribution is key to effective slurry filtration. This study aimed to evaluate a series of tighter graded density depth filters (Entegris Planargard®) to determine optimum filtration scheme for the slurries bulk filtration during manufacturing as well as point-of-use applications.

Why Characterize CMP Consumables?

- Changing requirements of Chemical Mechanical Planarization
  - More complex and demanding CMP solutions for 45 nm, 32 nm and smaller nodes
  - Introduction of larger wafers, copper, ultra low-k (ULK), high-k, and noble metals
  - Improved planarity and metrology specifications in Cu/low-k, STI, and poly-si CMP
- Emerging applications/devices, new consumables and refined processes in CMP
  - Each IC solution might have unique optimized CMP and PCMP clean requirements
  - MEMS, power devices, hard disk, SOI, GaAs, 3-Dim, photonic bandgap devices
  - Changed operating parameters (much lower polishing pressures in Cu/low-k CMP)
  - Innovative PCMP clean methods (laser, gaseous aerosols, supercritical CO_2)
- Slurry vendors, system suppliers and end users more interested in collaboration
  - Ability to evaluate and fine-tune complicated CMP slurry new formulations quickly
  - Reduce CoO and minimize development/optimization time and repetition of efforts
  - Improve understanding of CMP process needs and share cost of development
- Evaluation of CMP disruptive technologies by the end users and tool suppliers
  - Fixed abrasive, Electro-CMP (ECMP), and Chemically Enhanced Planarization (CEP) may offer advantages for productivity, low stress for ULK dielectrics, and Cu loss
  - Reduced need for CMP processing, PCMP cleaning, and slurry and chemical filtration

CMP Slurry Management Challenges

- **Challenges:**
  - Tighter purity and blend accuracy requirements of next generation slurries
  - Quick settling abrasive characteristics and limited post-blending useful life
  - Variability in slurry and blend chemical properties of different lots and over time
  - Uncertainties of oxidizer and additives decay and adjustments needs with time
  - More stringent particle counts, size distribution, and filtration requirements
  - Detection and selective removal of hard large particles at small concentrations
  - Newer slurries not well defined and require fine-tuning for specific processes
  - Requirements of reducing cost of ownership of CMP process and consumables
  - Continued collaboration and consolidation, and introduction of new products
- **Slurry health or quality monitoring parameters:**
  - Large (≥ 0.56 or 1.01 micron) particle counts (LPC)
  - Mean particle size distribution (PSD) and zeta potential
  - pH, ORP (oxidation reduction potential), conductivity, viscosity and refractive index
  - Total dissolved solids (TDS), weight % solids and density (or specific gravity)
  - Oxidizer and additives concentration and ionic contamination
  - Oxide slurries: agglomeration, filtration, wt % solids, LPC and PSD
  - Tungsten, copper and STI slurries: quick settling, oxidizer level, density, LPC and PSD

CMP Slurry Benchtop Characterization

- Slurry components and blend properties
  - Conductivity, pH, density, weight % solids
  - Assay, viscosity, refractive index
  - Particle size distributions (mean PSD and LPC), zeta potential
  - Incoming, normal mix ratio
- Sensitivity analysis of blend consistency measurement parameter
  - pH, density, conductivity, viscosity, assay
- Recommended mix ratio ± 20%
- Effect of DI water dilution
- Settling characteristics of abrasive particles
  - Incoming, source drum or pail, sample bottles

CMP Slurry Handling Characterization

- Settling behavior and redispersion effort
  - Incoming, storage tank or daytank, global loop settling
  - Loop shutdown, minimum flow rates
- Lifetime testing
  - Test slurry properties daily one week: day 0, 1, 2, 3, 4, 7
  - Chart properties in individual and composite form
  - Extend testing if appropriate: day 10, 14, 21, 28
- Replenishment
  - Decay of volatile/decomposing components and replenishment rate
- Filtration
  - Point-of-use (POU), point-of-dispense and global distribution loop
- Cleaning protocol
  - CDMs/PVVs, global loop

CMP Slurry Blend Control and Distribution

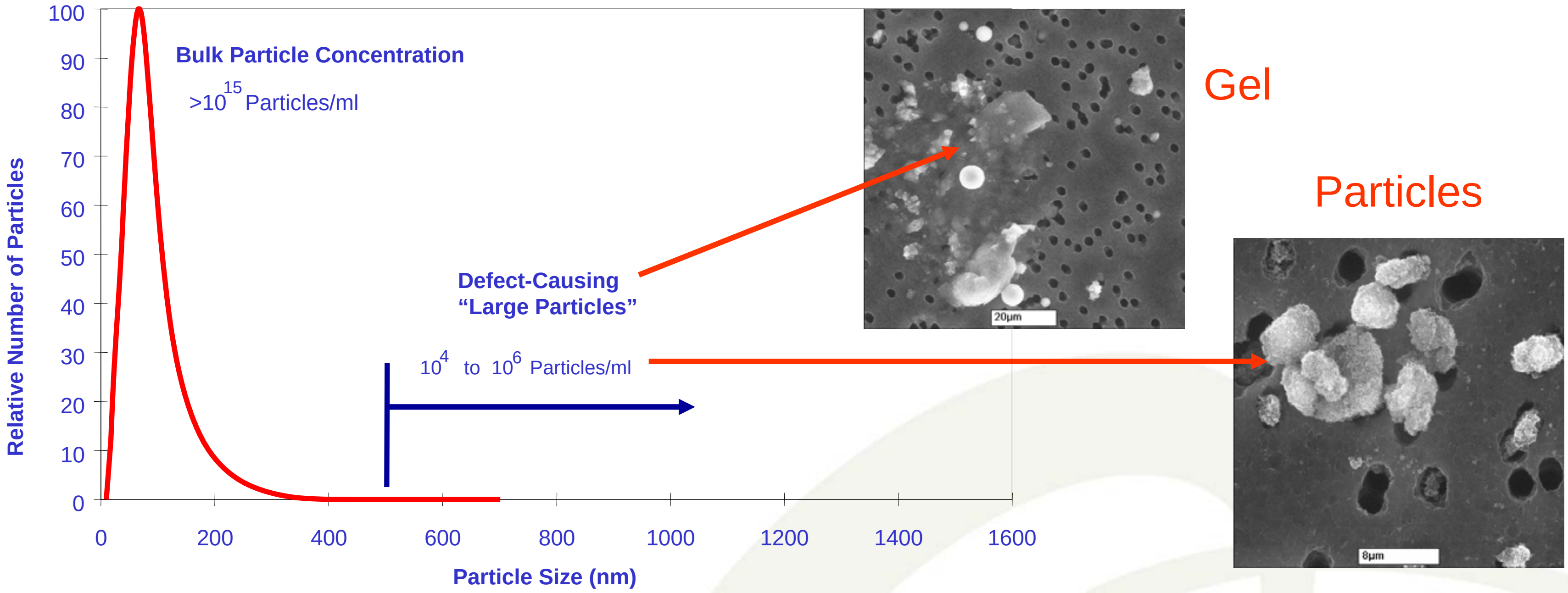
- Common blend monitoring and control parameters:
  - Density or specific gravity, Wt % solids, and oxidizer level
- Limitations of other parameters:
  - pH – slurries are chemically buffered, insignificant variation with changes in blend ratio
  - ORP (Oxidation-reduction potential) - does not change with mix ratio in most CMP slurry blends
  - Conductivity or TDS - usually has good sensitivity to blend ratio, often cannot be used as an independent control parameter, conductivity values vary in different lots of the same slurry, may also vary with aging of the same slurry lot during recommended storage life
- Silica based Oxide slurry: blends are controlled using density
- Tungsten and Copper slurry: blends controlled using an autotitrator or other concentration analyzer (ultrasonic, RI, or NIR based) - for monitoring and replenishment of the oxidizer level
- Silica slurries: slow settling characteristics
- Alumina- and Ceria-based slurries: usually have quick settling behavior
- Settling rate: can help in estimating the required minimum flow velocity of slurry in global loop

Sensitivity of Measurement Parameters to H_2O_2 Wt % Concentration in a CMP Slurry and H_2O_2 Blend

H_2O_2 % Wt	Density g/cc	Density Change	Wt % Solids	Conductivity µS/cm	pH	H_2O_2 % Vol.	% Vol. Change
2.4	1.03429	0.00026	3.036	14357	7.598	7.426	0.311
→ 2.5	1.03455	0.00026	3.025	14313	7.591	7.736	0.311
2.6	1.03481	0.00026	3.014	14270	7.584	8.047	0.311
2.7	1.03508	0.00026	3.003	14227	7.577	8.359	0.311
2.8	1.03534	0.00026	2.992	14183	7.570	8.670	0.311
2.9	1.03561	0.00026	2.981	14140	7.563	8.982	0.312
3.0	1.03587	0.00026	2.970	14096	7.557	9.293	0.312
3.1	1.03614	0.00026	2.959	14052	7.550	9.605	0.312

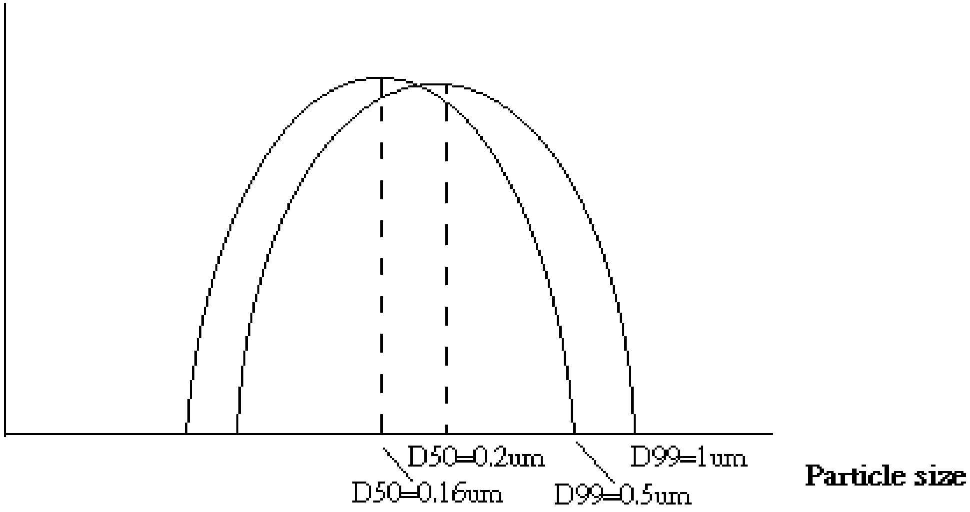
CMP Slurry Filtration: Current Trends

- Large particles (>10x of d_{50}) in slurries can cause defects (microscratches) and yield losses. Slurry suppliers employ filtration to eliminate those particles in manufacturing. Large particles tend to slowly reform due to instabilities in chemistry and handling
- Objective of CMP Slurry Filtration: Defect Reduction and Yield Improvement
  - To remove large particles and agglomerates from slurry that can cause defects, without changing slurry polishing performance



CMP Slurry Filtration: Changing Process Needs

- **Next generation slurry filtration targets:**
  - **Tighter retention of large particles at much smaller large-particle cut-off (e.g., 0.2 or 0.2 µm)**
  - **More consistent flow rate and pressure drop behavior, and longer filter lifetime**
  - **Minimal effects on the mean working particles for better local and global planarity, and consistency in the CMP processing**

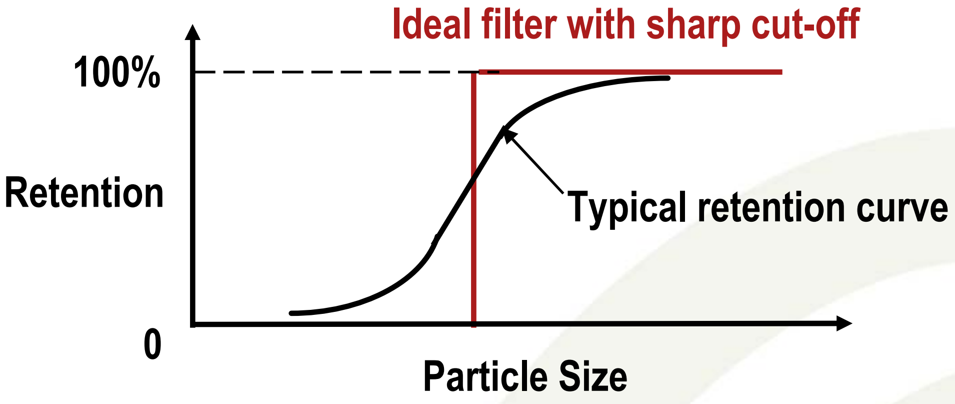


	D50 (mean size)	**D99**
Earlier	**0.20 µm**	**1 µm**
New Target	**0.07 µm**	**0.3 µm**
Typical Next Target	**0.04 µm**	**0.2 µm**

CMP Slurry Filtration: Methodology and Mechanisms

Slurry Filtration Process

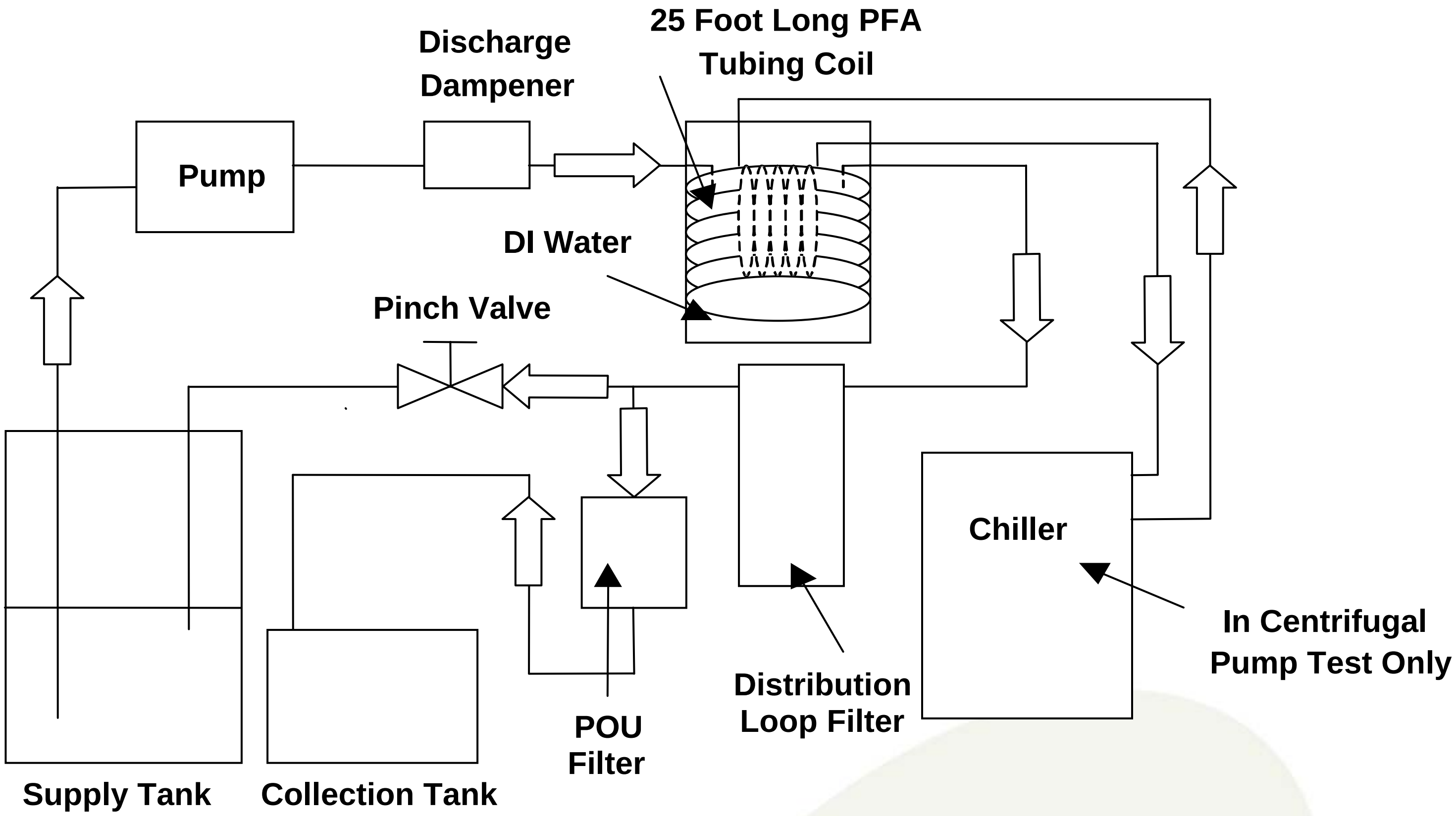
- CMP filtration is actually a separation process
- Filters have difficulty separating particles that are less than 1 order of magnitude different in size
- Don’t think of filters as strainers working only by size exclusion, there are other important mechanisms
  - Inertial impaction, Interception, Adsorption/Adhesion, Diffusion, and Settling
- There are also effects tied to how the media is arranged in the filter



Slurry Filtration Characterization

- **Retention/Flow and Pressure Drop Test**
  - Retention test conducted with PSL beads solution and CMP slurries and pressure drop tests at 0, 1, 2, 3, 4 GPM using a differential pressure unit
- **Lifetime Test**
  - Testing with CMP slurries and pressure drop and flow rate measurements till pressure drop reaches a specified limit
- **Recirculation Loop Test**
  - Evaluation of global loop and POU filters using a vacuum-pressure dispense system as well as bellows, diaphragm, a magnetically levitated centrifugal pumps
- **Collaborative Testing with Slurry Vendors and Customers**
  - Field returned filter analysis and troubleshooting
  - Extent of filter plugging/remaining lifetime by Δp and weight gain
  - SEM and ESEM (environmental SEM, for wet sample imaging) analysis
- **Filter Related Troubleshooting at Site**

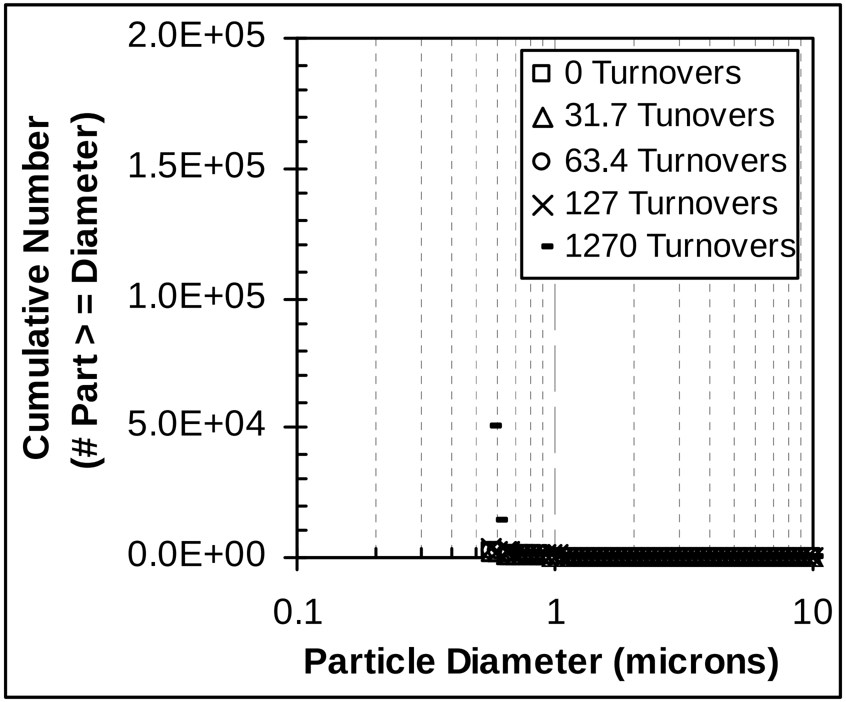
Slurry and Filter Characterization in CMP Laboratory Simulated Recirculation Loop



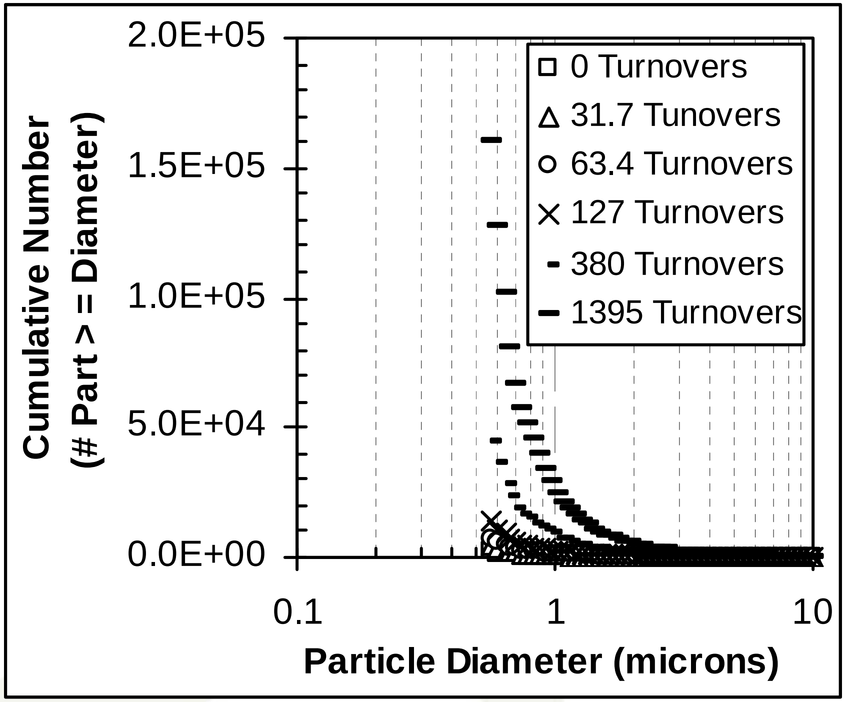
Schematic of Slurry Recirculation Loop Test Set-Up

Example 1 - LPC data for Silica Slurry-A under extensive handling in a magnetically levitated centrifugal pump and a diaphragm pump

8000 rpm, 28 psi back pressure, 8 lpm, 63.4 turnovers/hr, 20 hr test, BPS-3 pump

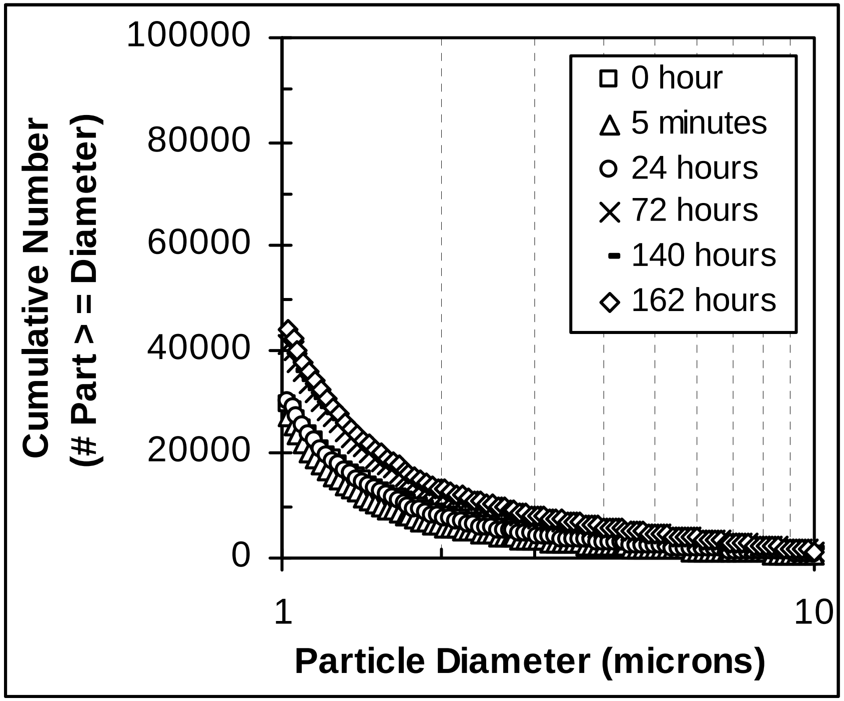


28 psi back pressure, 8 lpm, 63.4 turnovers/hr, 24 hr test, diaphragm pump

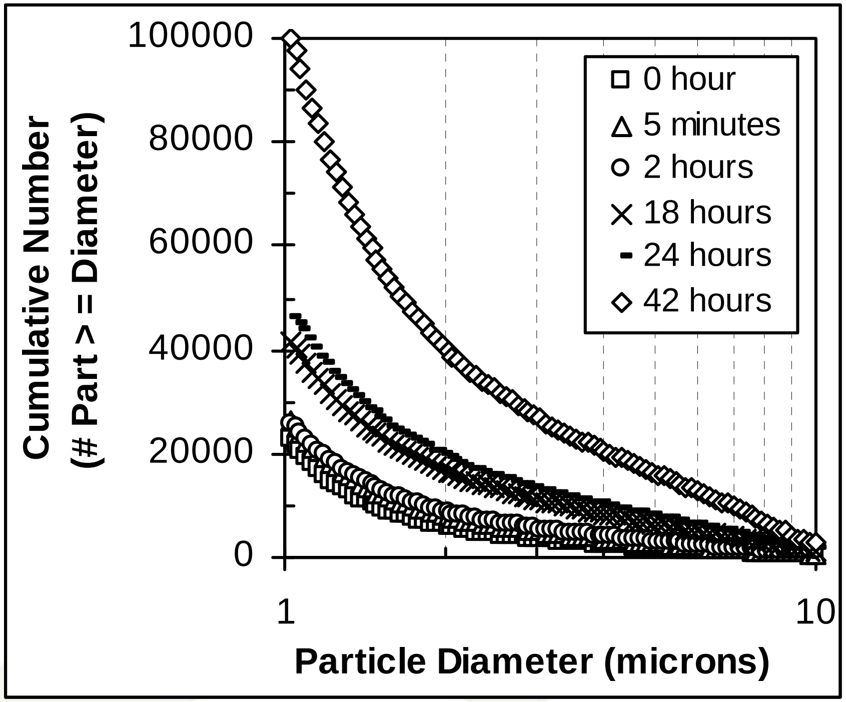


Example 2 - LPC data for Silica Slurry-B under extensive handling in a vacuum-pressure dispense system and a bellows pump loop

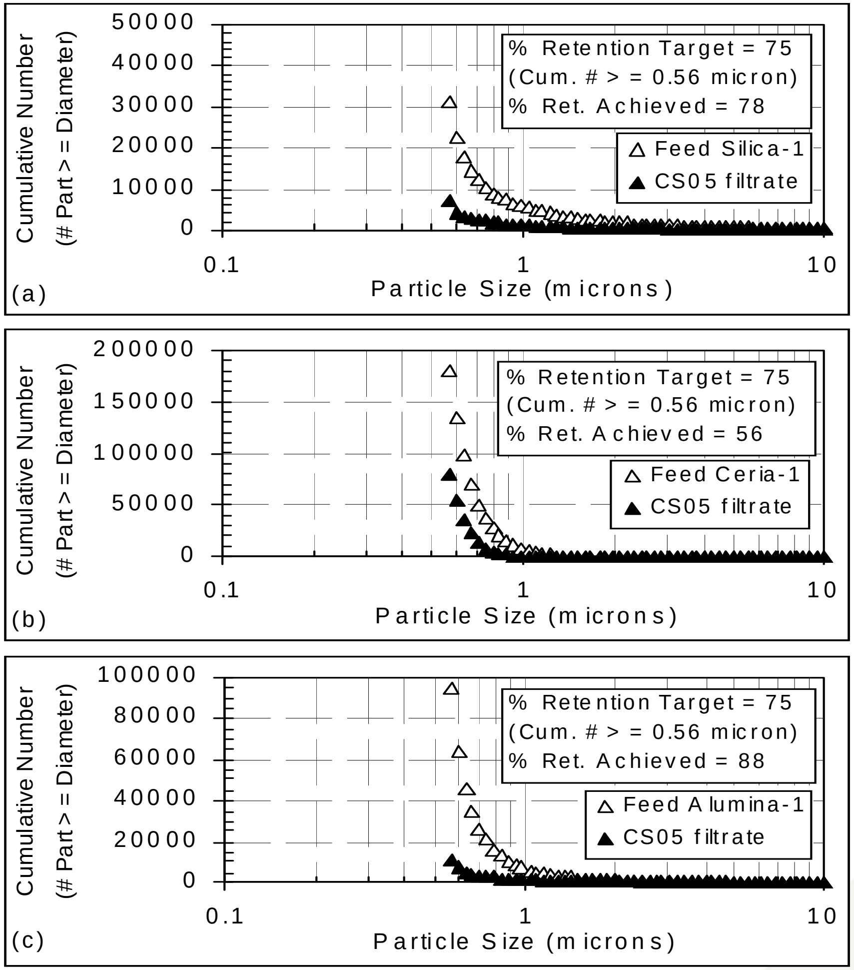
LPC data for Silica Slurry 1 in a vacuum-pressure dispense system at 17.1 turnovers/hr



LPC data for Silica Slurry 1 in bellows pump recirculation loop at 60 turnovers/hr



LPC data for different abrasive slurries under single-pass tighter filtration using Entegris Planargard® CS05 (0.5 μm) depth filter



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.

LPC data, pressure drop and flow rate for different slurries with Entegris Planargard® CS05 (0.5 μm) and Planargard® CMP1 (1 μm) depth filters

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

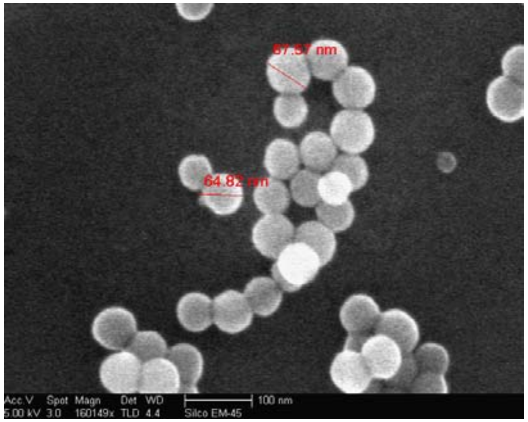
Slurry / Challenge Solution	**CMP1 (Cumulative % LPC reduction for particles ≥1.01 μm)**	**Pressure Drop Δp (psi)**	**Flow Rate (ml/min)**
Silica-1	63	7.8	423
Ceria-1	53	5.2	519
Alumina-1	71	3.5	535
Alumina-2	69	5.2	531
PSL Bead Solution	36	4.4	535

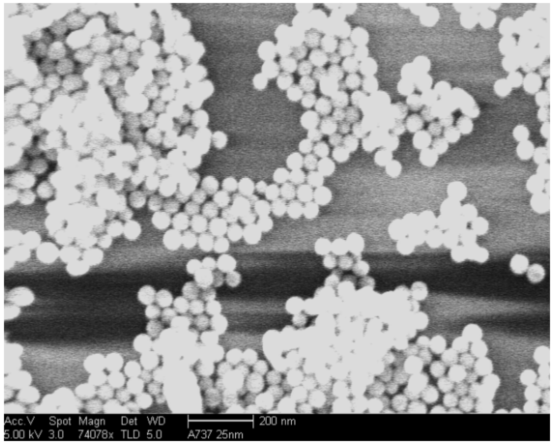
Table 1. Filters show large slurry dependent variations in performance.

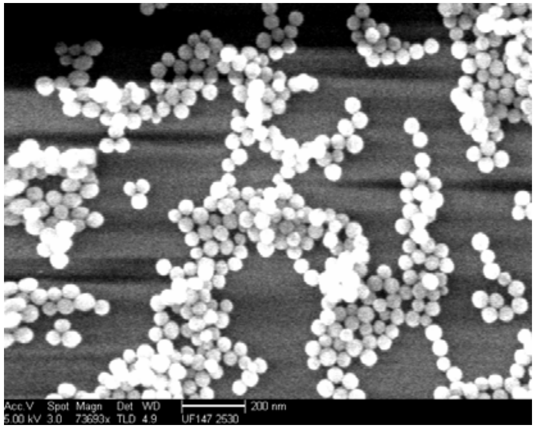
Characteristics of a New Colloidal Silica Slurry

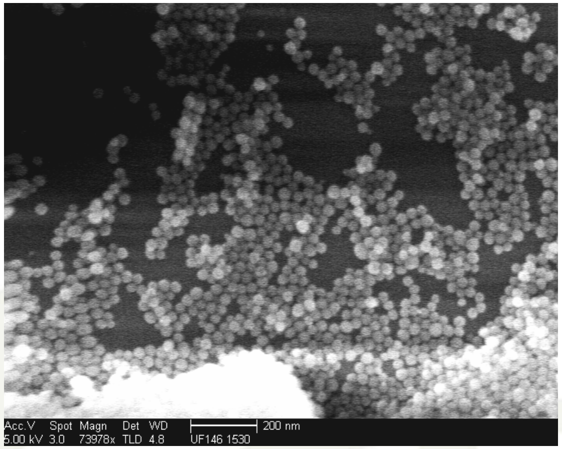
- Unique pH-Stable Colloidal Slurry Products
- Consistent Particle Size Distribution
- More Stable CMP Process Window
- Very High Purity… < 10 PPM Sodium
- Very Low Metals, No Chlorides
- Excellent Product Stability
- Monodisperse Low-pH Particles
- Lower Defect Counts !

Typical Colloidal Silica Particles



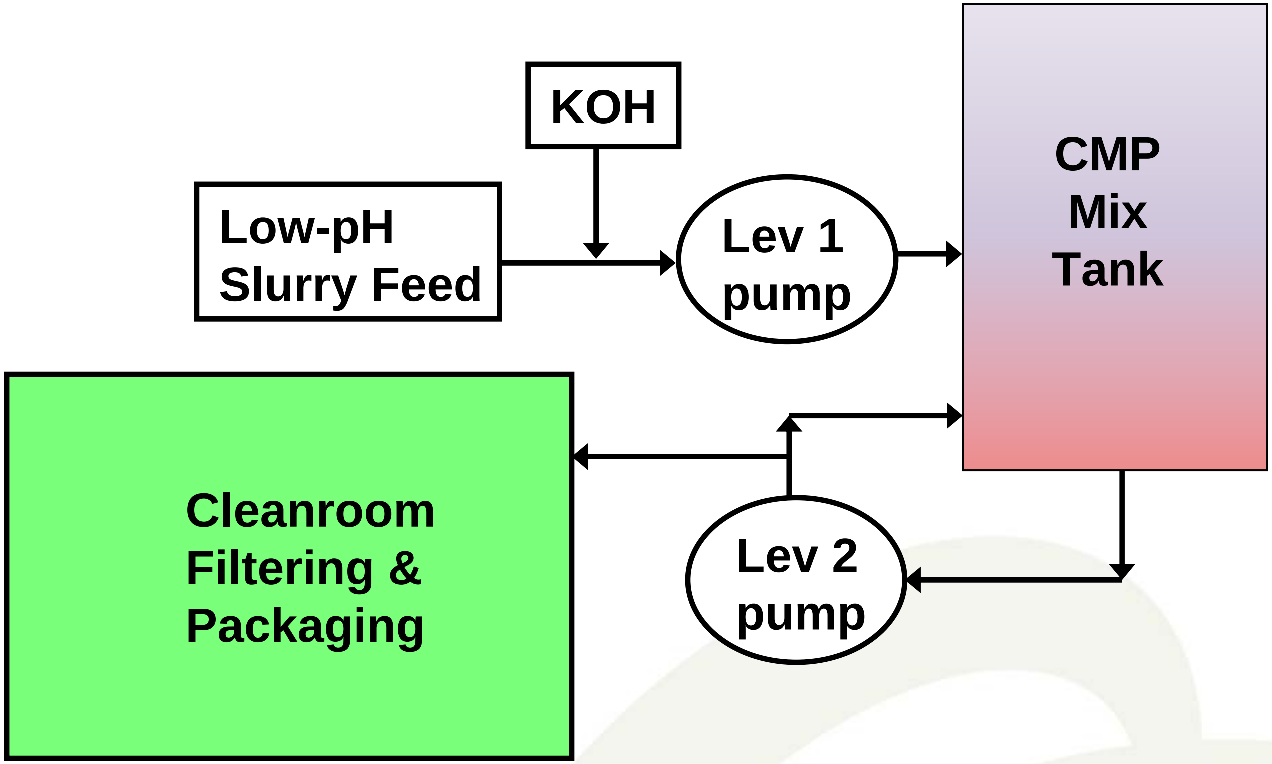






pH Stabilization Process

pH Stabilization Process


KOH

Low-pH Slurry Feed

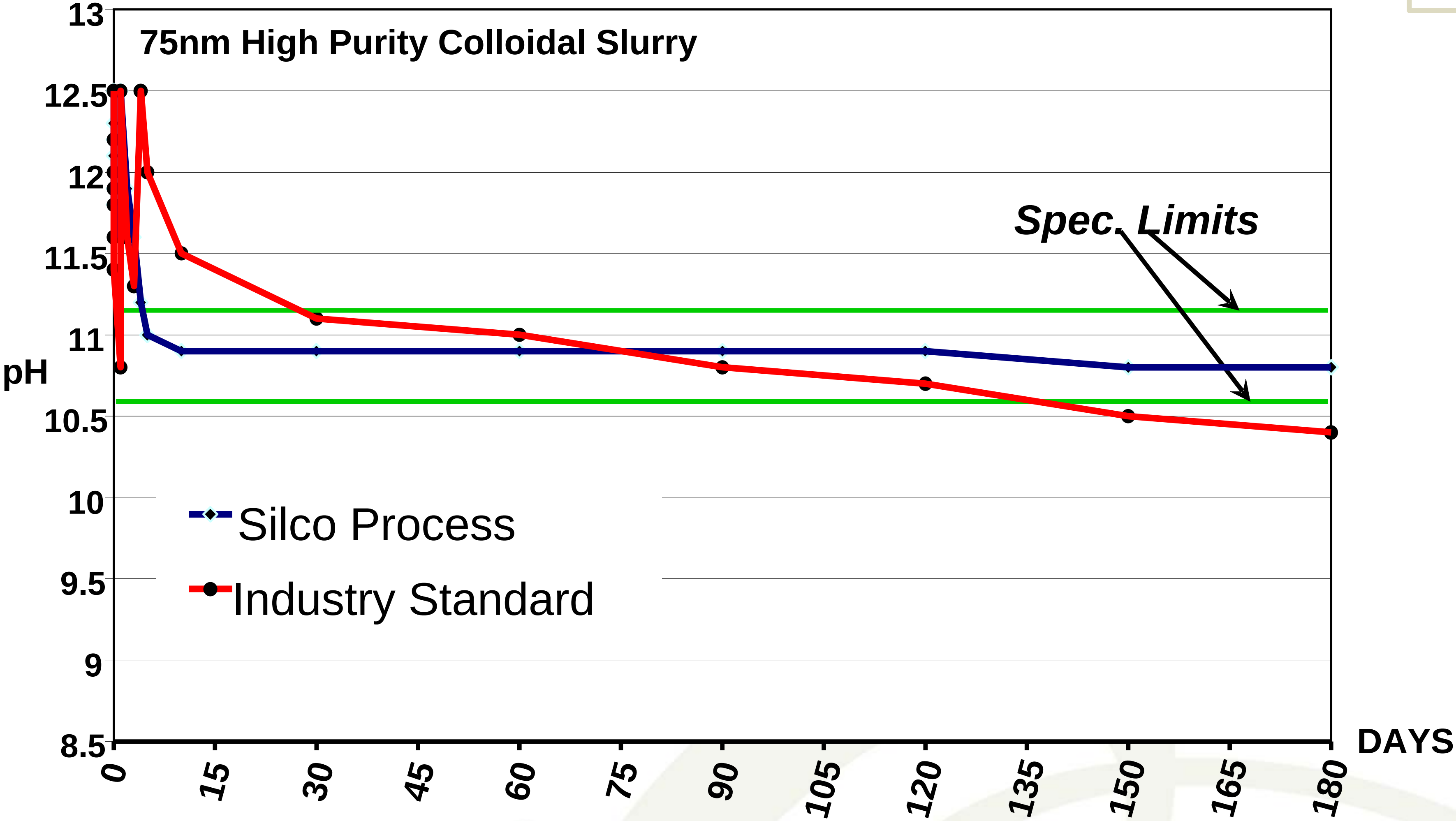
Lev 1 pump

CMP Mix Tank

Lev 2 pump

Cleanroom Filtering & Packaging

pH Stabilization Study



Handling and Filtration of CMP Slurries
Levitronix CMP Users Conf., Feb. 11, 2008

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Effect of Trace Metals on ILD Polish Performance

Slurry	[Al]	[Ca]	[Cr]	[Fe]	[Ni]	[Na]	Normalized Defects & Microscratches
Silco	**<1**	**<0.1**	**<0.1**	**<1**	**<0.1**	**<10**	**198** **164**
Standard	<100	<1.8	NA	<6.5	<10	<50	**588-658** **268-316**
Supplier X	NA	NA	NA	NA	NA	NA	**233-277** **451-533**
Typical	<50	<5	<1	<20	<100	<100	NA

- **Experiments run by a volume IC Fab**
- **All metals are specs and units in ppm**
- **All slurries are based on colloidal silica particles**
- **Comparable removal rate and uniformity**

Silco 75nm ILD Slurry Performance

Slurry	Down Force (psi)	Uniformity	Normalized Defects & Microscratches	Polish Rate (A/min)
Silco	**7.0**	**6.0%**	**198** **164**	**3800**
Standard	7.0	7.4%	588-658 **268-316**	3700
Alternative	7.6	5.5 to 8.0%	233-277 **451-533**	3650

- Experiments run by a volume IC Fab
- All slurries are based on colloidal silica particles
- All slurries have same solids content of silica

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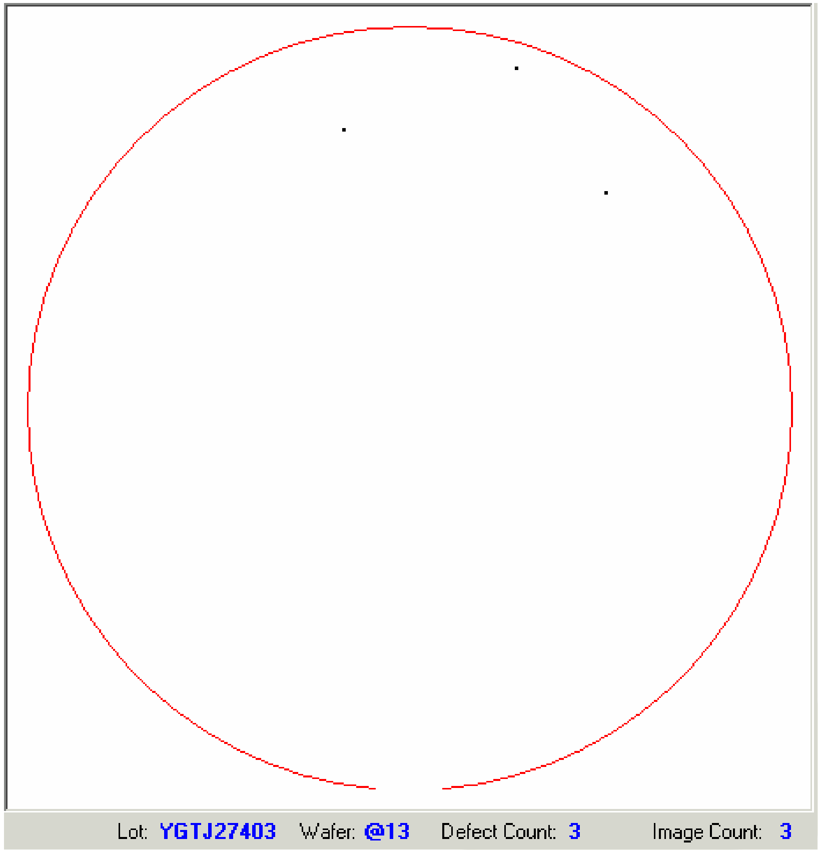
ILD Polish Objectives

- Compare defect performance of Silco EM products vs POR slurry and alternate slurry in qualification using blanket furnace TEOS wafers
- Perform 1000A HF etch to highlight and provide insight into microscratch performance
- Compare blanket polish rates and non-uniformity using Silane based oxide film
- Tests performed on Novellus Momentum and Applied Materials Mirra platforms

Results

– Scratch Monitor: Control, HF Highlight without a polish



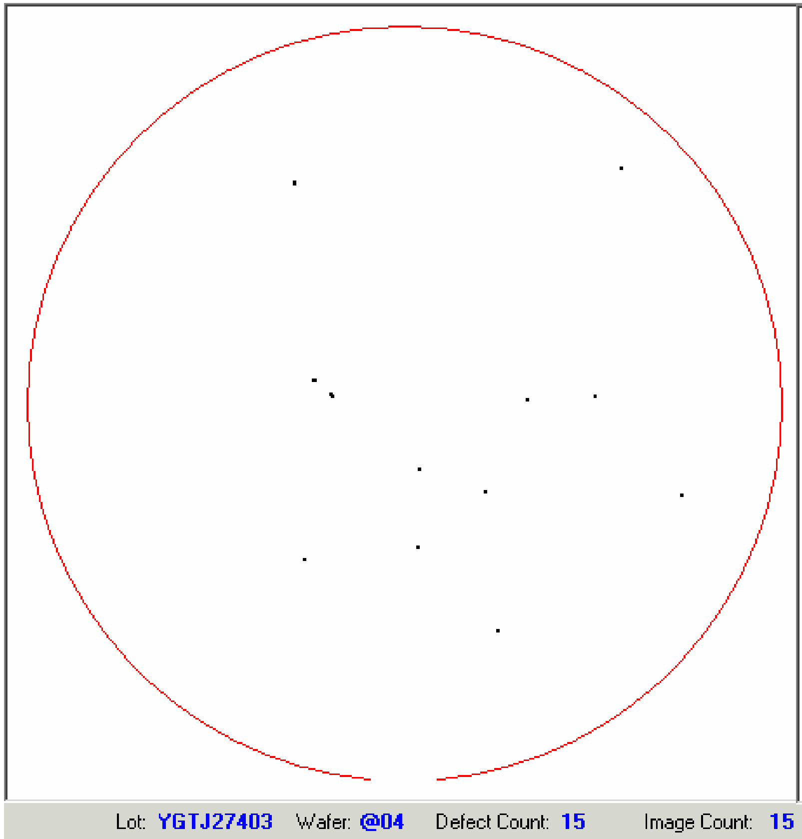
Lot: YGTJ27403 Wafer: @13 Defect Count: 3 Image Count: 3

HF Highlight added 3 particles

Results Cont.

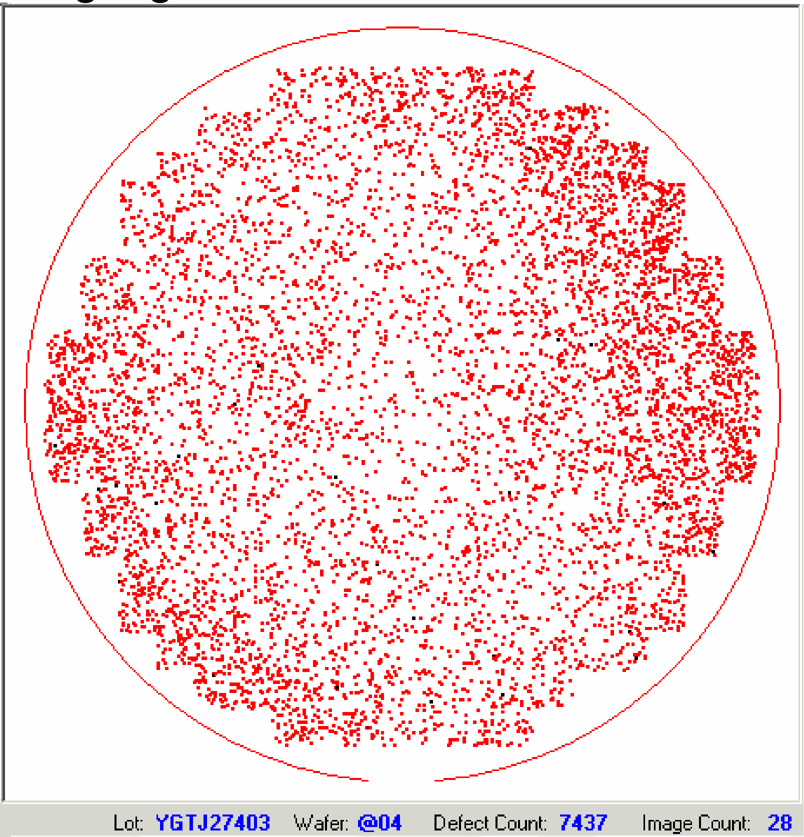
– Scratch Monitor: Slurry 1, Novellus w/ IC1000 Pads, Wafer #1

Particle count post polish



Lot: YGTJ27403 Wafer: @04 Defect Count: 15 Image Count: 15

Particle count post 1000Å HF highlight for micro-scratches



Lot: YGTJ27403 Wafer: @04 Defect Count: 7437 Image Count: 28

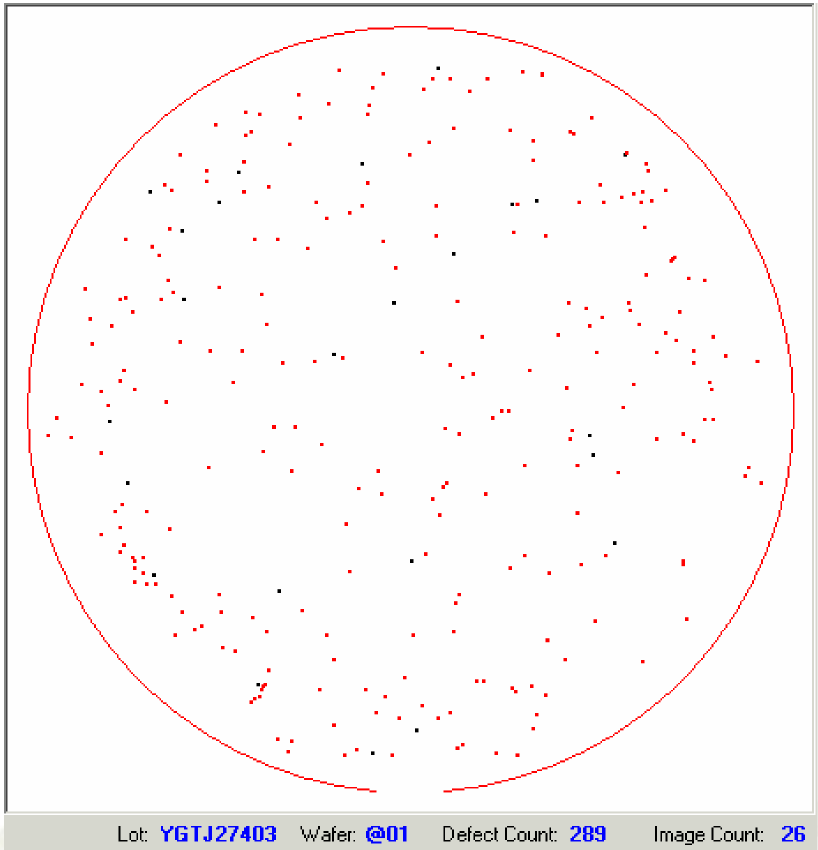
Results Cont.

– Scratch Monitor: Slurry 2, Novellus w/ IC1000 Pads, Wafer #1

Particle count post polish

No defects found

Particle count post 1000Å HF highlight for micro-scratches

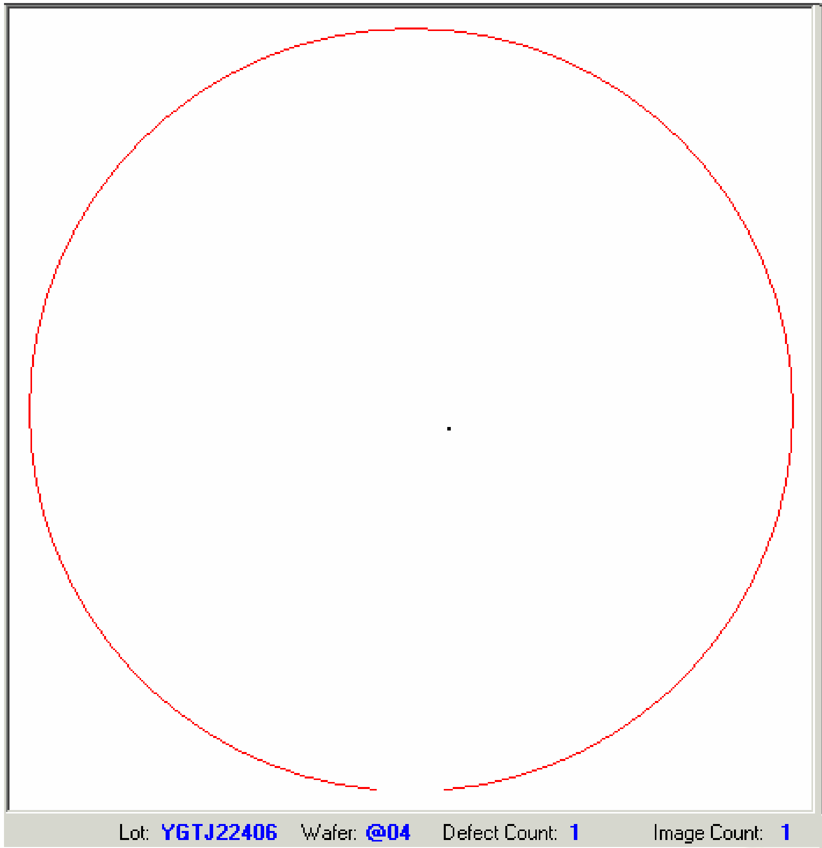


Lot: YGTJ27403 Wafer: @01 Defect Count: 289 Image Count: 26

Results Cont.

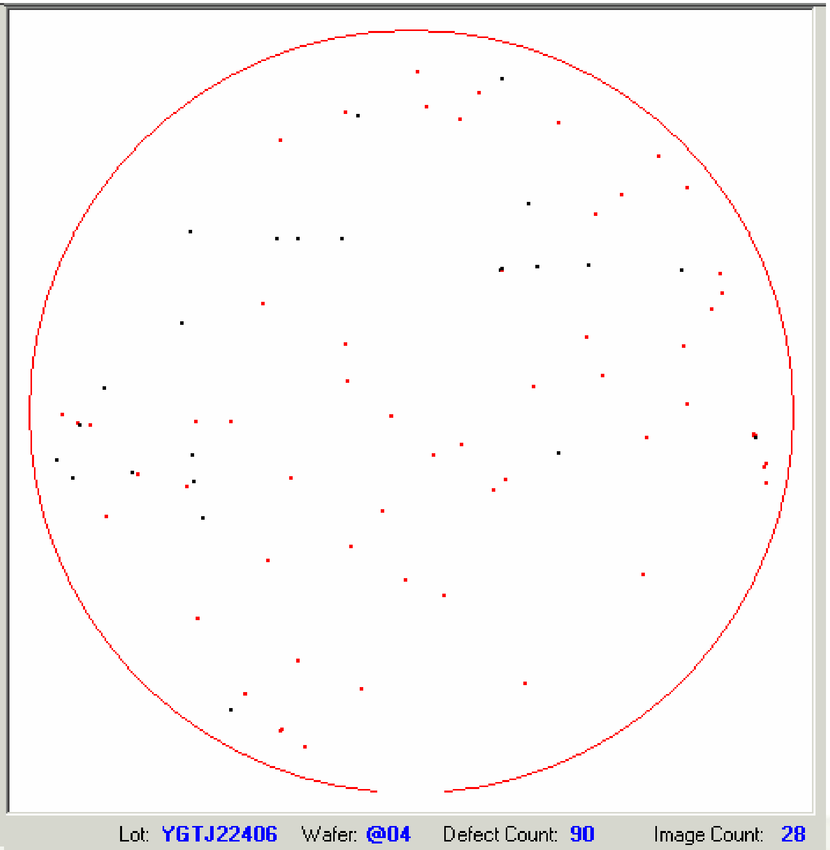
- Scratch Monitor: Silco EM-7530K, Novellus w/ IC1000 Pads, Wafer #1

Particle count post polish



Lot: YGTJ22406 Wafer: @04 Defect Count: 1 Image Count: 1

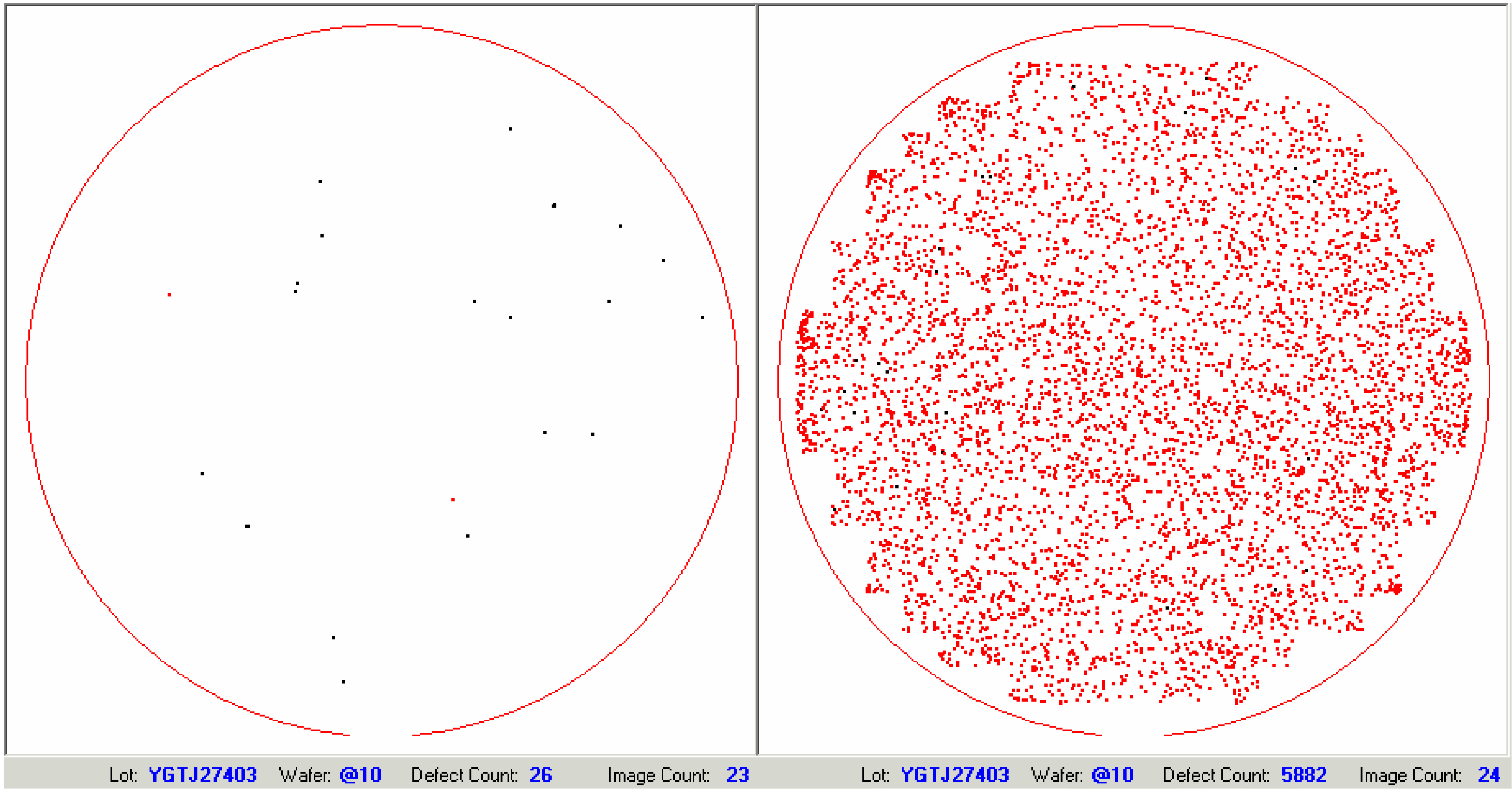
Particle count post 1000Å HF highlight for micro-scratches



Lot: YGTJ22406 Wafer: @04 Defect Count: 90 Image Count: 28

Results Cont.

– Scratch Monitor: Slurry 1, AMAT w/ IC1010 Pads, Wafer #1



Particle count post polish

Particle count post 1000Å HF highlight for micro-scratches

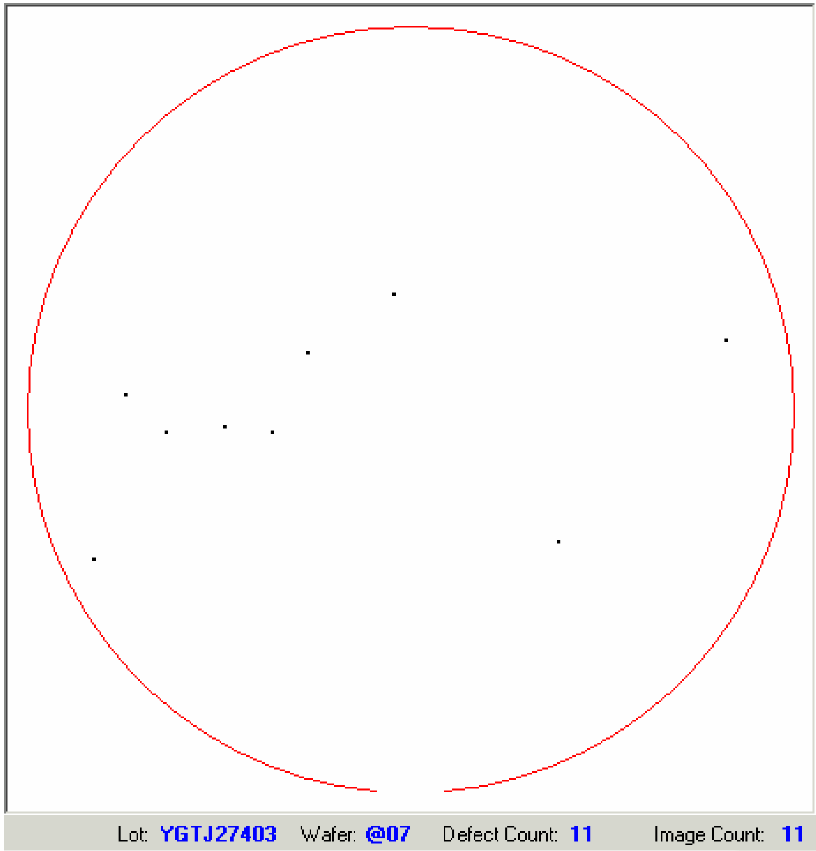
Lot: YGTJ27403 Wafer: @10 Defect Count: 26 Image Count: 23

Lot: YGTJ27403 Wafer: @10 Defect Count: 5882 Image Count: 24

Results Cont.

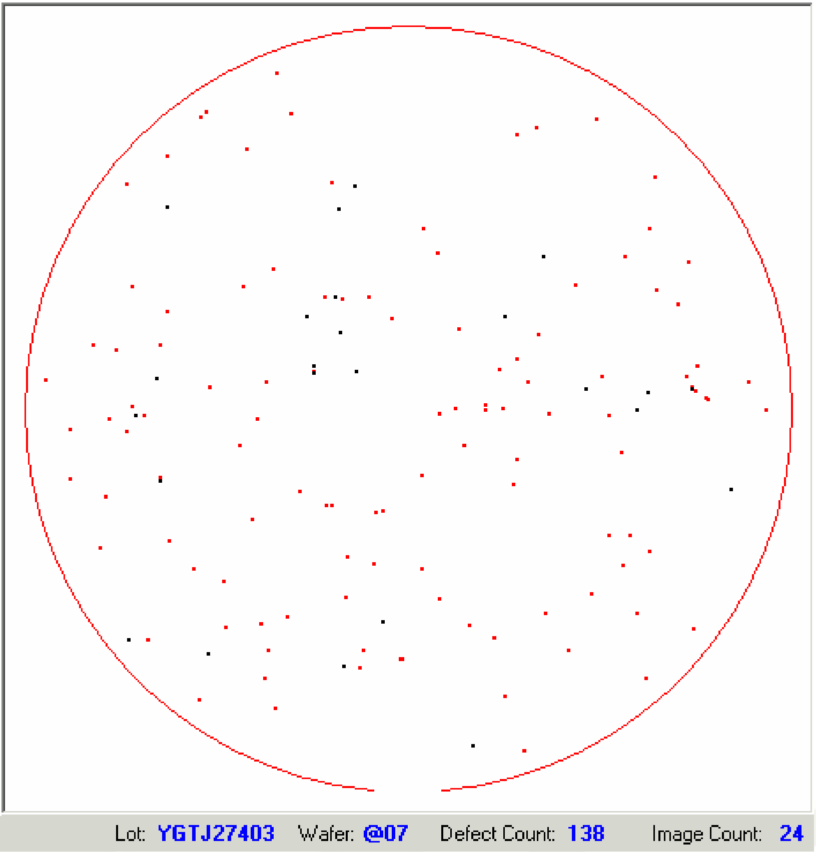
– Scratch Monitor: Slurry 2, AMAT w/ IC1010 Pads, Wafer #1

Particle count post polish



Lot: YGTJ27403 Wafer: @07 Defect Count: 11 Image Count: 11

Particle count post 1000Å HF highlight for micro-scratches

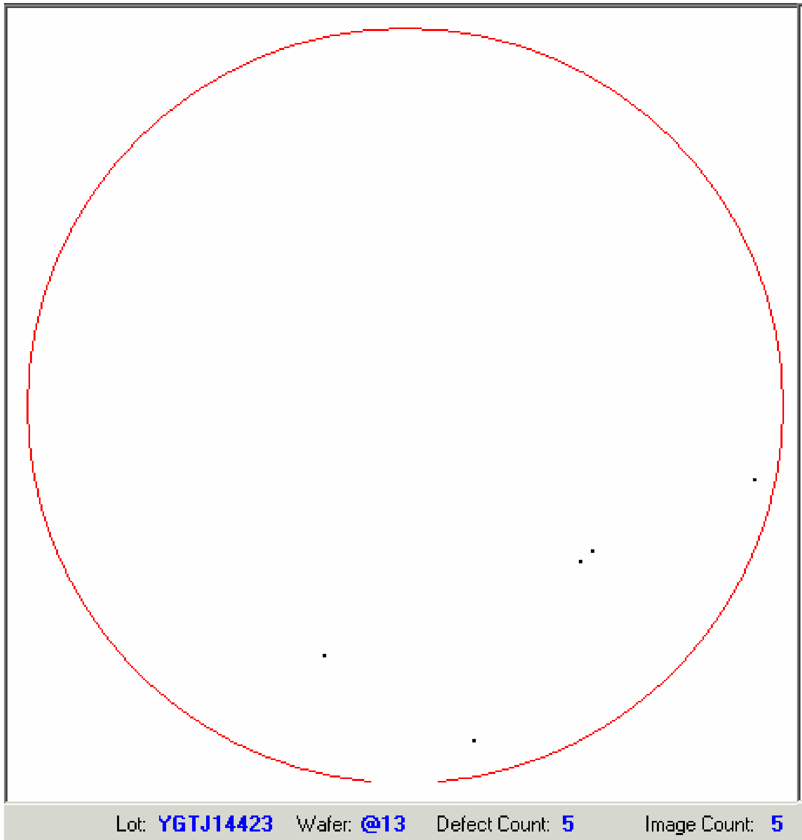


Lot: YGTJ27403 Wafer: @07 Defect Count: 138 Image Count: 24

Results Cont.

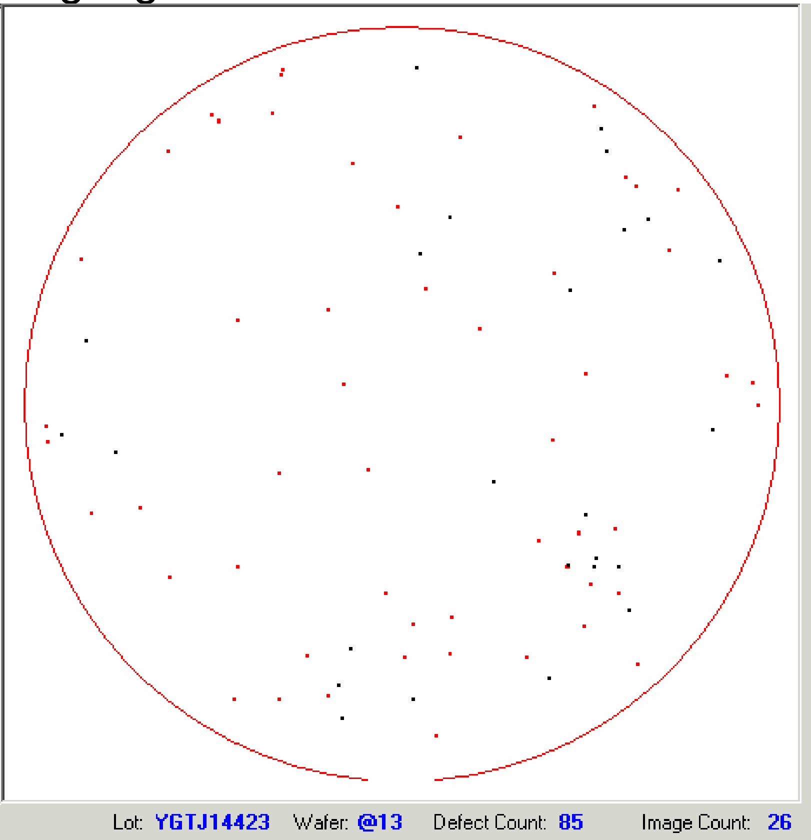
- Scratch Monitor: Silco EM-7530K, AMAT w/ IC1010 Pads, Wafer #1

Particle count post polish



Lot: YGTJ14423 Wafer: @13 Defect Count: 5 Image Count: 5

Particle count post 1000Å HF highlight for micro-scratches



Lot: YGTJ14423 Wafer: @13 Defect Count: 85 Image Count: 26

EM-5530K and EM-7530K Wafer Polishing Rate, NU and Particle Data Summary

Platform	Pad	Slurry	Rate	NU	Particles
Novellus	IC1000	EM-5530K	2496	3.8	2.67
Novellus	IC1000	EM-7530K	2221	1.5	1.33
Novellus	IC1000	Slurry 1	2364	6.8*	14.67
Novellus	IC1000	Slurry 2	2366	6.9*	0.67
AMAT	IC1010	EM-5530K	3143	6.38	16
AMAT	IC1010	EM-7530K	2862	6.57	2.67
AMAT	IC1010	Slurry 1	2990	5.37*	19.67
AMAT	IC1010	Slurry 2	3098	5.01*	12.3
AMAT	PPG	EM-5530K	3243	4.43	4
AMAT	PPG	EM-7530K	3066	4.66	4.67

Green = Silco

Yellow = Slurry 1

Blue = Slurry 2

ILD Polish Observations

- Defect performance of Silco EM-7530K is favorable compared to alternate colloidal slurries on both the Novellus Momentum and Applied Materials Mirra platforms
- Removal rate and non-uniformity are comparable on both Novellus and Applied platforms
- Silco EM-5530K exhibited slightly higher removal rate relative to EM-7530K

Results of Filtration Study with Entegris High-Retention Graded Density Depth Filters

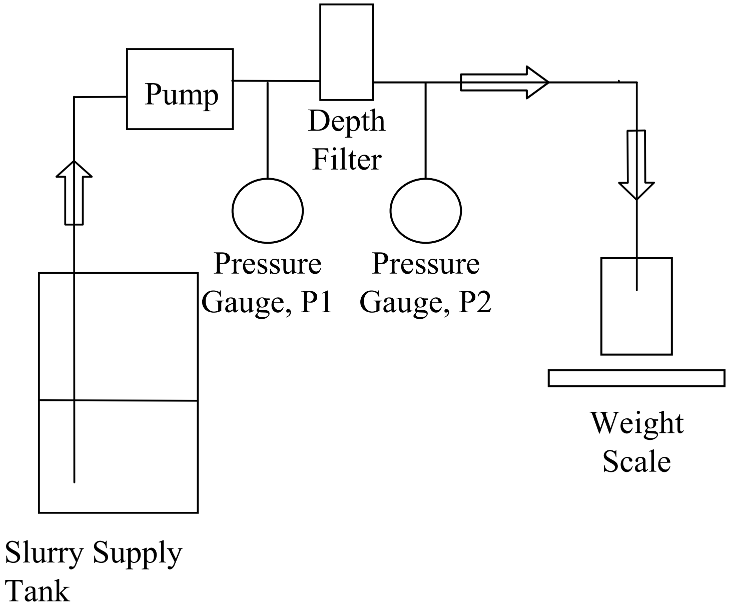


Figure 1. Schematic of Filter Test Set-Up.

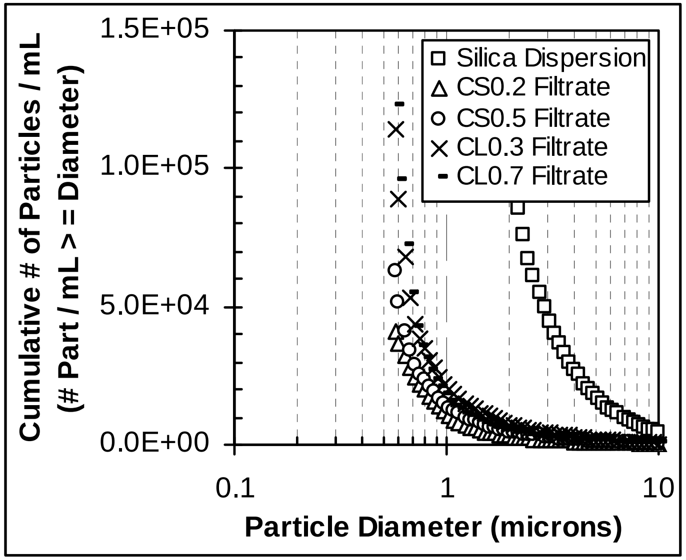


Figure 2. Colloidal Silica Dispersion source and Planargard® high retention filters LPC distribution.

Results of Filtration Study with Entegris 1 µm Nominal Rating Graded Density Depth Filters

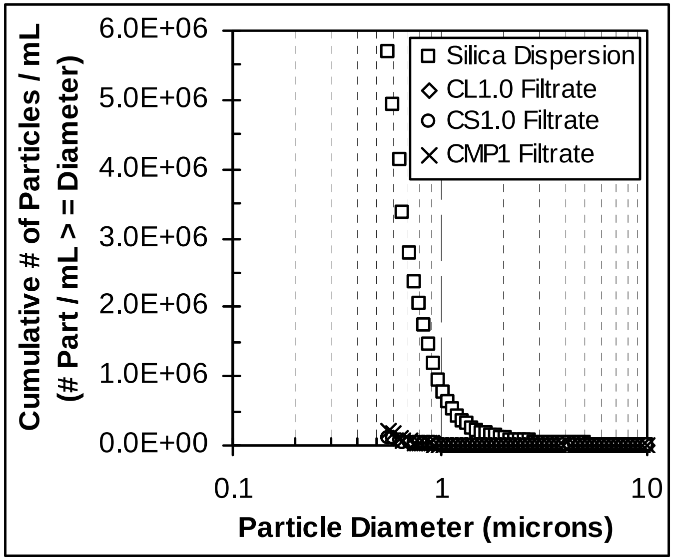


Figure 3a. Colloidal Silica Dispersion source and Planargard® 1 micron filters LPC distribution.

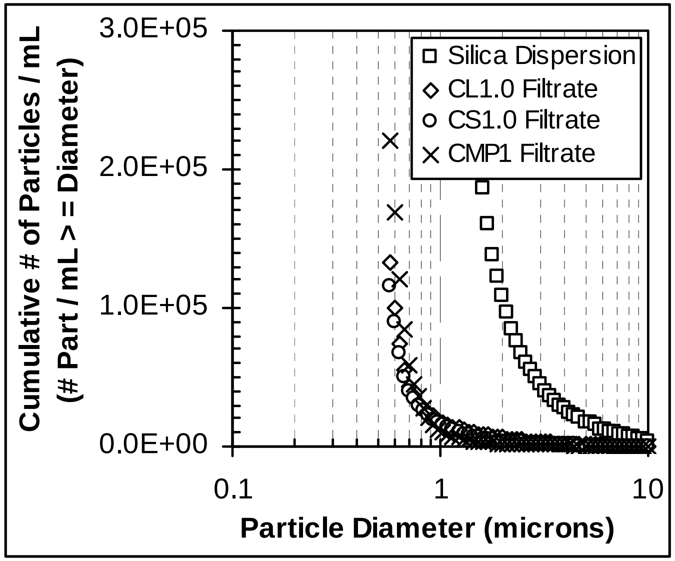


Figure 3b. Colloidal Silica Dispersion source and Planargard® 1 micron filters LPC distribution.

Results of Filtration Study with Entegris High-Retention Graded Density Depth Filters

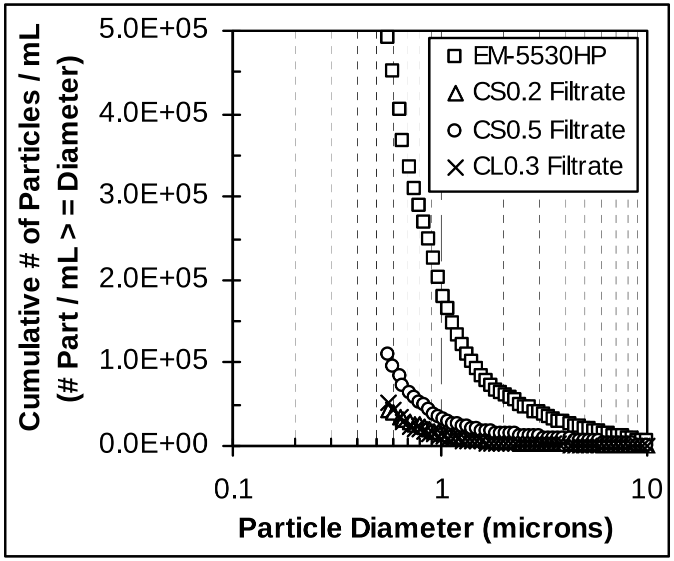


Figure 4a. EM-5530HP oxide silica slurry source and Planargard® high retention filters LPC distribution.

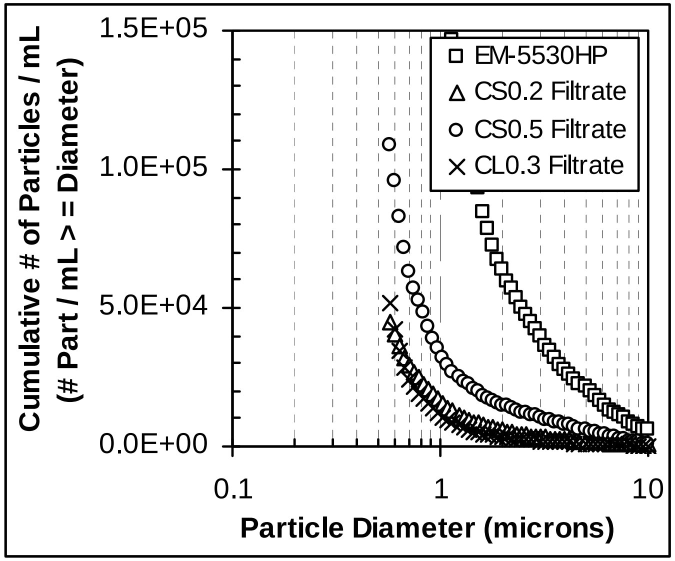


Figure 4b. EM-5530HP oxide silica slurry source and Planargard® high retention filters LPC distribution.

Results of Filtration Study with Entegris High-Retention Graded Density Depth Filters

Filter Type (2” sample)	Pr. Drop Δp (psi)	Flow Rate Q(mL/min)	#/mL Part. Conc. ≥0.56 μm size	#/mL Pt. Retention ≥0.56 μm size (%)	Weight % of Solids
Silica Dispersion (Filtrate Properties)					
Planargard® CS0.2	44	466	41312	99.3	32.0
Planargard® CS0.5	16	474	62764	98.9	32.0
Planargard® CL0.3	11.8	469	114332	98.0	32.1
Planargard® CL0.7	12.1	467	123110	97.8	32.3
Planargard® CL1.0	10.5	476	133722	97.7	32.3
Planargard® CS1.0	11.1	479	115540	98.0	32.3
Planargard® CMP1	7.0	479	222172	96.1	32.5
EM-5530HP Oxide CMP Slurry (Filtrate Properties)					
Planargard® CS0.2	21.4	481	44534	90.9	32.5
Planargard® CS0.5	9.5	488	108934	77.7	32.3
Planargard® CL0.3	7.9	487	51620	89.4	32.4
Source Silica Dispersion and EM-5530HP Slurry Properties					
Silica Dispersion			5696650		32.7
EM-5530HP Slurry			489000		32.6

Results of Filtration Study with Entegris High-Retention Graded Density Depth Filters

Filter Type (2" sample)	Filtrate Mean Particle Size, µm	pH	Conductivity (µS/cm)	TDS (ppm)	ORP (mV)
Silica Dispersion					
Planargard® CS0.2	0.0945	3.08	394.5	264.3	361
Planargard® CS0.5	0.0951	3.09	394.6	264.3	328
Planargard® CL0.3	0.0950	3.07	395.1	264.5	369
Planargard® CL0.7	0.0947	3.07	393.7	263.6	353
Planargard® CL1.0	0.0946	3.08	398.2	266.5	375
Planargard® CS1.0	0.0948	3.10	400.5	268.3	364
Planargard® CMP1	0.0945	3.11	400.0	268.4	360
EM-5530HP Oxide CMP Slurry					
Planargard® CS0.2	0.0981	10.48	2413	1758	112
Planargard® CS0.5	0.0983	10.55	2414	1759	121
Planargard® CL0.3	0.0977	10.45	2413	1758	105
Source Silica Dispersion and EM-5530HP Slurry Properties					
Silica Dispersion	0.0957	2.96	397	269	348
EM-5530HP Slurry	0.0976	10.40	2386	1753	70

Summary and Conclusions

- Effective CMP slurry management should consider abrasive type and composition, oxidizer and chemical additives, LPC, mean PSD, pH, conductivity, wt % solids, viscosity, filter particle retention, pressure drop, flow-rate and lifetime, slurry usage schedule and turnover rate, and the blending and distribution system "the pump" characteristics. Slurry characterization and metrology studies help in identification of sensitive parameters to blend slurry accurately and monitor its health during usage and replenishment.
- Diaphragm and bellows pump handling tests show that silica-based CMP slurries are shear sensitive and generate significant number of large particles under extensive pump turnovers. A magnetically levitated centrifugal (MLC) pump generated far fewer large particles (size > 1 micron) as compared to double diaphragm pumps in silica slurry under comparable turnovers.
- Present study evaluates comparative performance of Silco EM-5530K & EM-7530K slurries in terms of polishing rate, NU and particle defectivity using different CMP pads and other similar slurry products. These next generation slurries provide precise and consistent removal rates, minimal wafer defectivity, and maximum planarity across the wafer surface.
- This study aimed to determine the optimum filtration scheme for Silco Electronic Materials EM-5530 silica slurry. A series of Entegris Planargard® graded density depth filters were tested to quantify their effectiveness in removing defect causing large particles from the slurry.
- Planargard® CS0.5 and Planargard® CL0.3 (nominal ratings 0.5 and 0.3 micron, respectively) filters provided the required reduction in the cumulative LPCs ≥ 0.56 micron. This study shows the importance of CMP consumables comparative laboratory and fab evaluations to generate optimum slurry quality/health management information.

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- Levitronix CMP Users Conf. Organizers for the opportunity

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