

Laboratory Scale Assessment of the Fate of CMP Nanoparticles Through Standard Waste Water Treatment Systems *(Customized Project)*

PIs:

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Other Researchers:

- Antonia Luna, Postdoctoral Fellow, Chemical and Environmental Engineering, AA
- Jim Field, Professor, Chemical and Environmental Engineering, AA

Cost Share (other than core ERC funding):

- Graduate Incentives for Growth Awards (GIGA) (\$25 K)
- Student Fellowship (Mexican Science Foundation, CONACyT) (\$20 K)

SRC/SEMATECH Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Objectives

- Investigate removal of CMP nanoparticles by interactions with the biosolids (*i.e.*, entrapment in sludge flocs, adsorption to cells, cell internalization).
- Assess the impact of municipal wastewater composition (eg. organic matter, salts, surfactants, pH, etc) on the aggregation behavior of CMP nanoparticles.
- Evaluate the removal of nanoparticles (NP) present in CMP effluents in a bench-scale wastewater treatment plant.

ESH Metrics and Impact

Reduction in emission of ESH-problematic material to environment

This study will lead to new insights on the interactions of NPs with typical components in semiconductor effluents, municipal wastewaters, and natural aquatic environments over a wide range of physicochemical conditions.

It will also provide information on the fate of NP present in CMP waste streams in municipal wastewater treatment systems. The knowledge gained can be utilized to determine if additional treatment steps are required to reduce environmental emissions of ESH-problematic chemicals, if any.

Materials and Methods

Commercial Slurries

Fumed Silica – Oxide CMP (30 nm)

Alumina – Cu CMP

Ceria – Oxide/STI CMP

Virgin Nanoparticles

Silica: 10-20 nm

Alumina: 50 nm

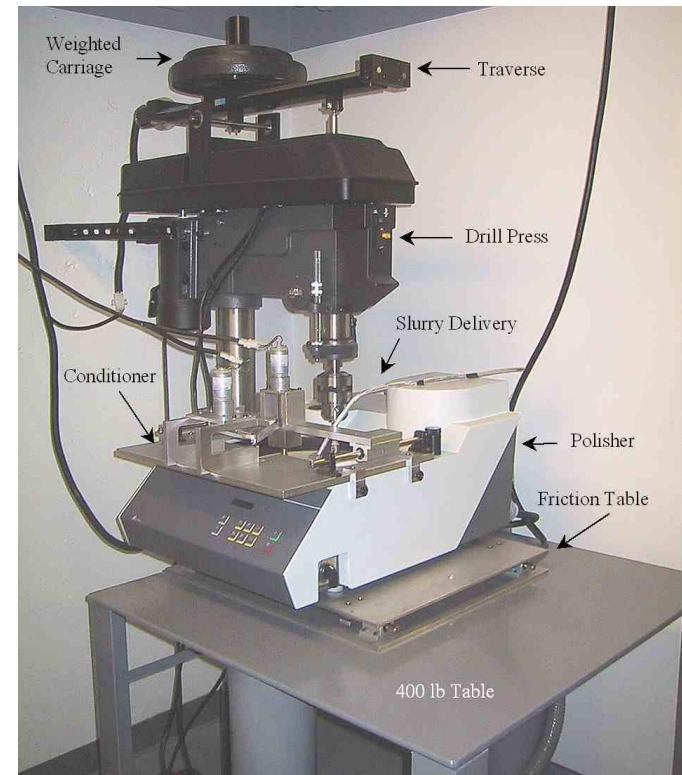
Ceria 20 or 50 nm

CMP waste:

Prepared at the pilot-scale CMP tool available at the UA

Particle size and Zeta potential:

Malvern Zeta Sizer Nano ZS



Polisher (100-mm) that will be used to prepare CMP waste effluents

Tasks

1. Impact of wastewater components on the aggregation behavior of CMP nanoparticles.
 - Organic compounds: eg proteins, aminoacids, humus, etc.
 - Polyelectrolytes
 - pH, Salts, divalent cations
2. Removal of CMP nanoparticles by biosolids
 - Adsorption isotherm experiments
 - Electron microscopy examination
3. Removal of nanoparticles in CMP effluent a lab-scale wastewater treatment plant
 - Lab-scale experiments with ceria slurry

Significant Aggregation of CeO₂, SiO₂ and Al₃O₂ Nanoparticles in Aqueous Solution

NP	Average Particle Size (nm)	
	Powder (Using TEM)	Aqueous medium (Using ZetaSizer)
CeO ₂	20	139
Al ₂ O ₃	50	175
SiO ₂	10-20	368

CeO₂, Al₂O₃ and SiO₂ NPs showed significant aggregation in aqueous solutions even at pH values considerably different from their respective isoelectric point.

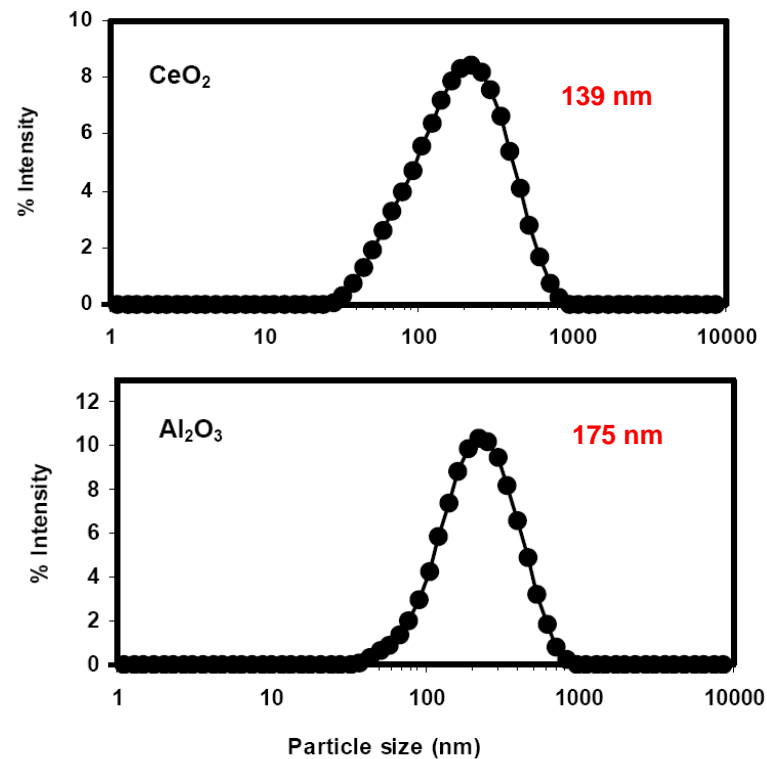


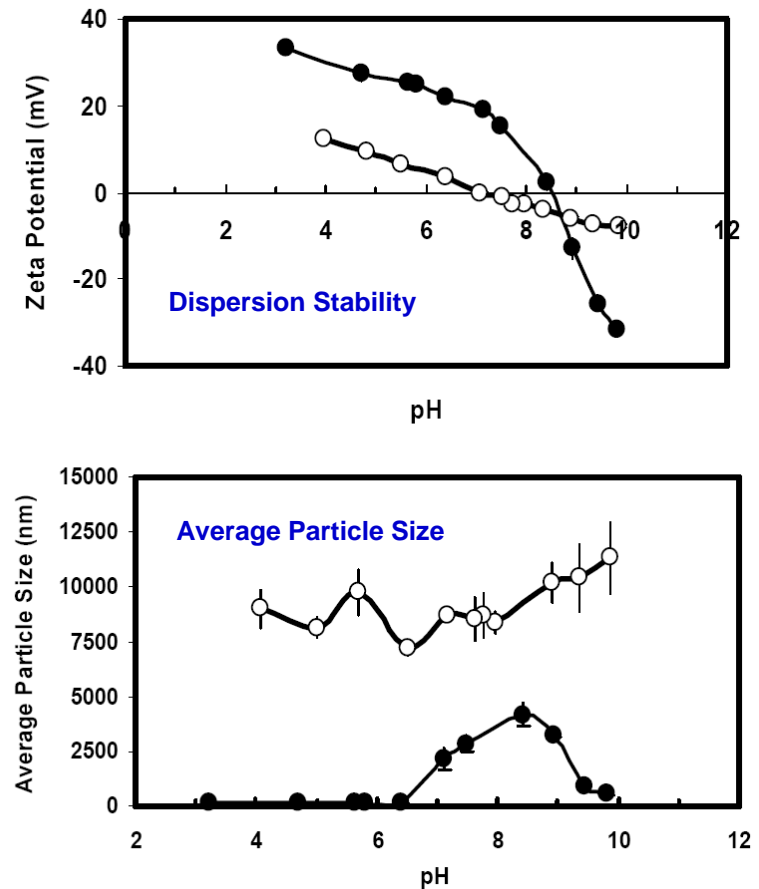
Fig. Particle size distribution of nano-CeO₂ and Al₂O₃ in pH-5.2 and pH-4.5 aqueous solutions, resp. Titration experiments showed that the dispersions exhibit high stability at these pH values.

Impact of pH and Dilution with Municipal Waste on the Stability of Al_2O_3 NP Dispersions

Z-pot: -20 to 20 mV
Dispersion is unstable

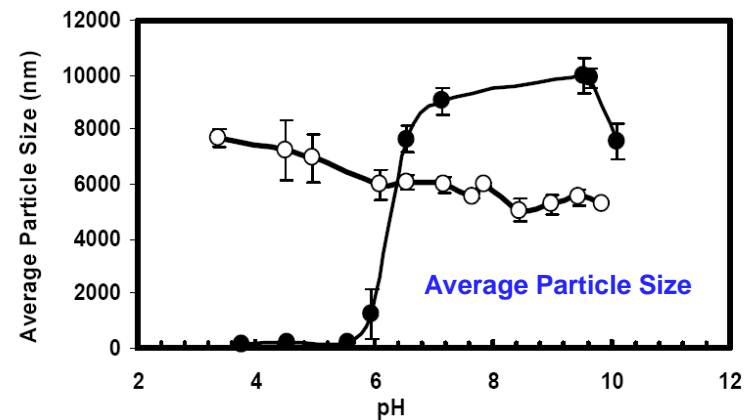
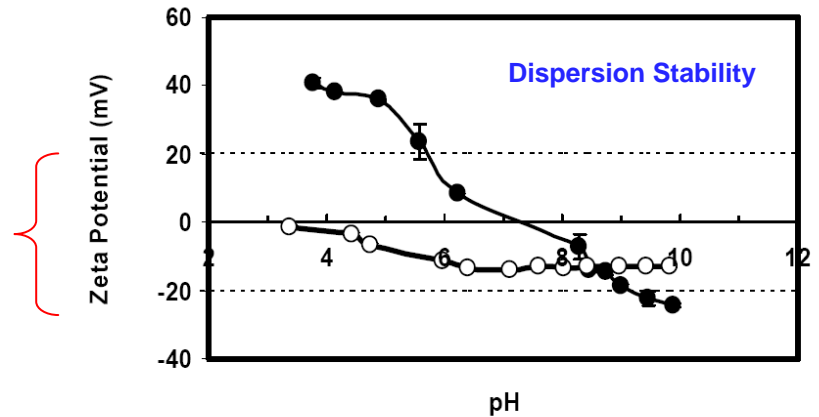
Dilution with municipal wastewater and circumneutral pH values promotes aggregation of alumina NPs.

Fig. Impact of pH on the zeta potential & average diameter of Al_2O_3 dispersions mixed with demineralized water (●) or filtered (20 nm) municipal wastewater (○).



Impact of pH and Dilution with Municipal Waste on the Stability of CeO₂ NP Dispersions

Z-pot: -20 to 20 mV
Dispersion is unstable



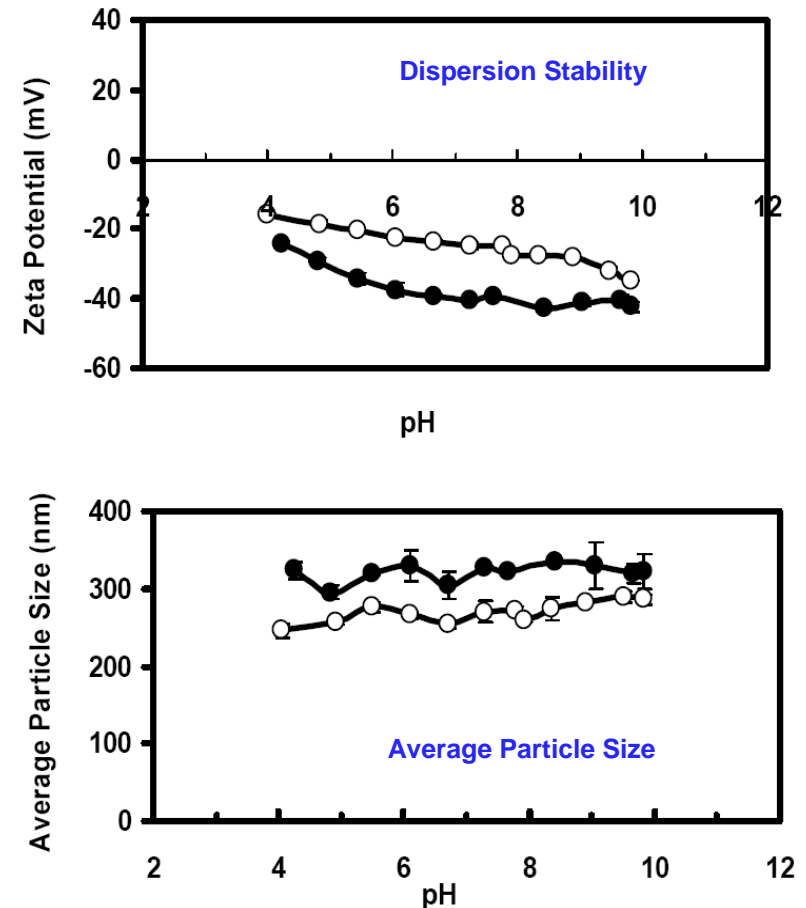
Dilution with municipal wastewater and increasing pH promotes the aggregation of CeO₂ nanoparticles

Fig. Impact of pH on the zeta potential & average diameter of CeO₂ dispersions mixed with demineralized water (●) or filtered (20 nm) municipal wastewater (○).

Impact of pH and Dilution with Municipal Waste on the Stability of SiO₂ NP Dispersions

The stability of SiO₂ NP dispersions was little affected by changes in pH or dilution with municipal wastewater

Fig. Impact of pH on the zeta potential & average diameter of SiO₂ dispersions mixed with demineralized water (●) or filtered (20 nm) municipal wastewater (○).

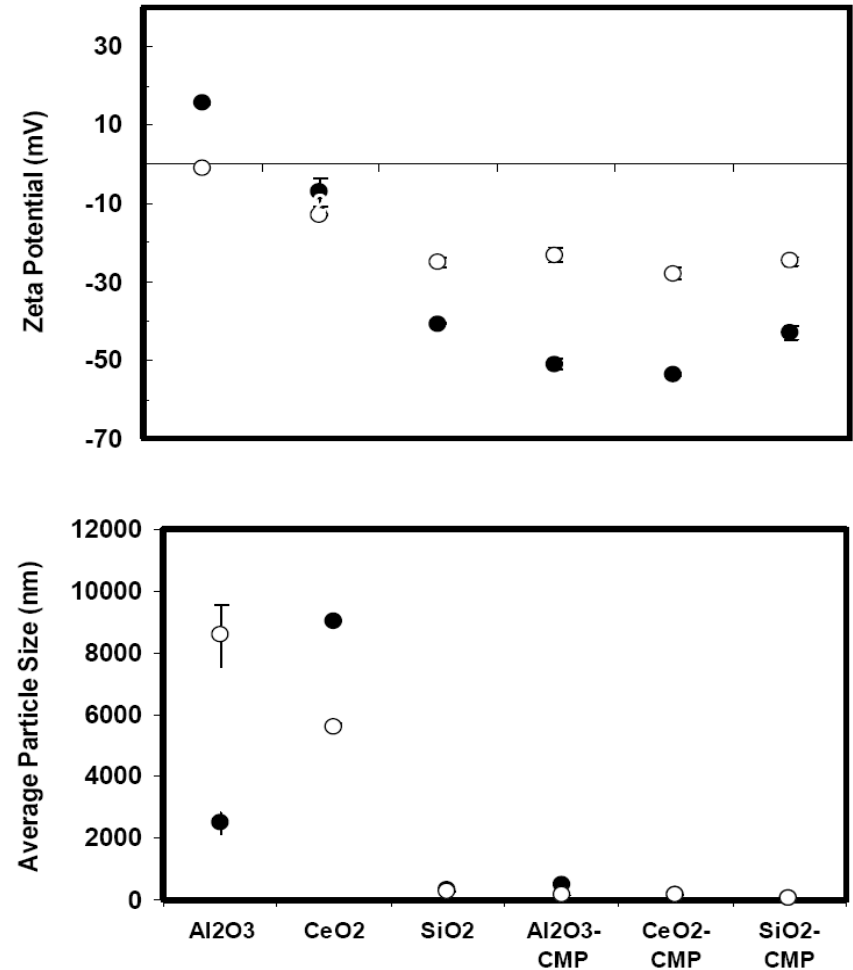


Impact of Dilution with Municipal Waste on the Stability of Al_2O_3 , CeO_2 , and SiO_2 NP Dispersions

Dilution of **CMP waste** with domestic wastewater (1:1) only caused a moderate decrease in the dispersion stability, probably due to high levels of surfactants in the slurry.

Considerably higher wastewater dilution resulting in increased destabilization of the NP dispersion is expected in the sewer.

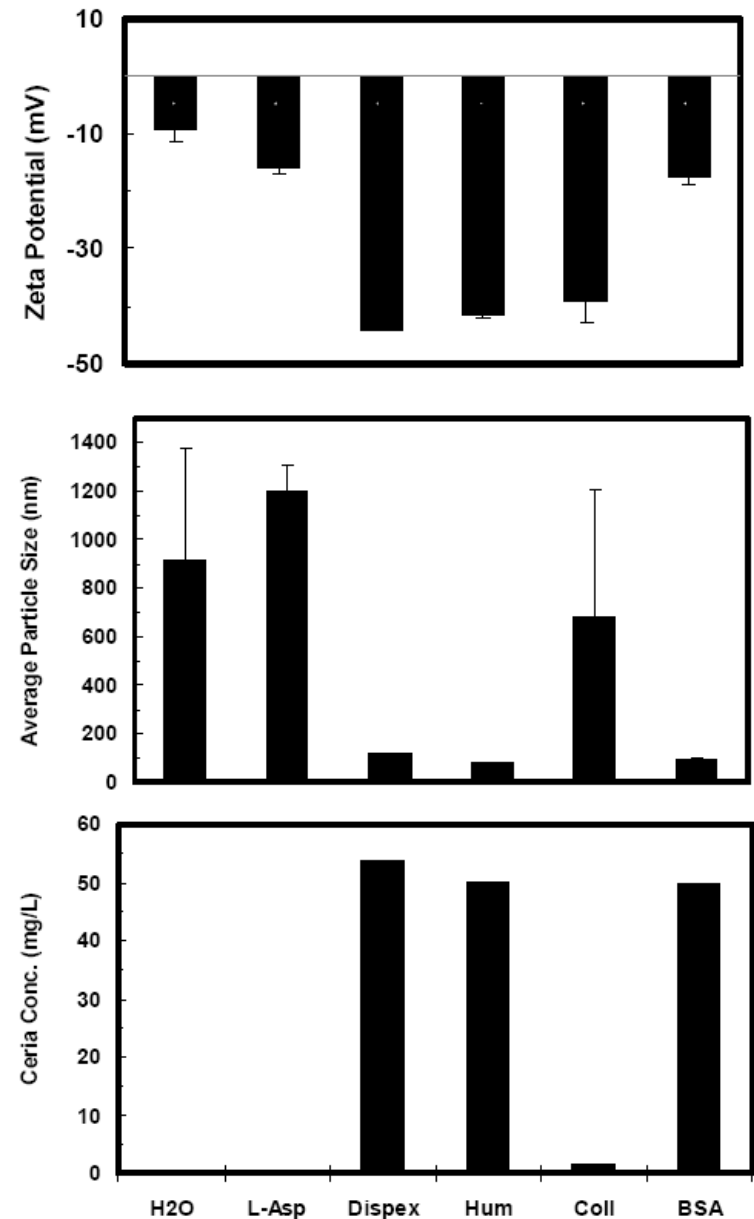
Fig. Stability of nano-sized CeO_2 , Al_2O_3 and SiO_2 dispersions, and CeO_2 , Al_2O_3 and SiO_2 -based CMP slurries diluted (1:1, v/v) with deionized water (●) and membrane-filtered (20 nm) municipal wastewater (○) at circum-neutral pH values.



Wastewater Components and NP Stability

Constituents in municipal wastewater alter the stability of the inorganic oxide dispersions. Some proteins, humic substances and synthetic dispersants stabilize NP dispersions (CeO_2 , Al_2O_3 , SiO_2) in the neutral to mildly alkaline range which is typical of municipal wastewaters.

Fig. Zeta potential (Top panel), average particle size (Middle panel), and residual ceria concentration in dispersions (Lower panel) after dilution (1/1, v/v) of a nano-sized ceria dispersion with solutions of different additives (200 mg/L). The final pH of the diluted dispersions was 6.8. Additives: L-Asp= L-aspartic acid; Dispex= Ammonium polyacrylate dispersant; Hum= Humic acid; Coll= Collagen; BSA= Bovine serum albumin.



Removal of CMP Nanoparticles by Activated Sludge

Aerobic activated sludge has a relatively high affinity for CeO_2 and Al_2O_3 NPs

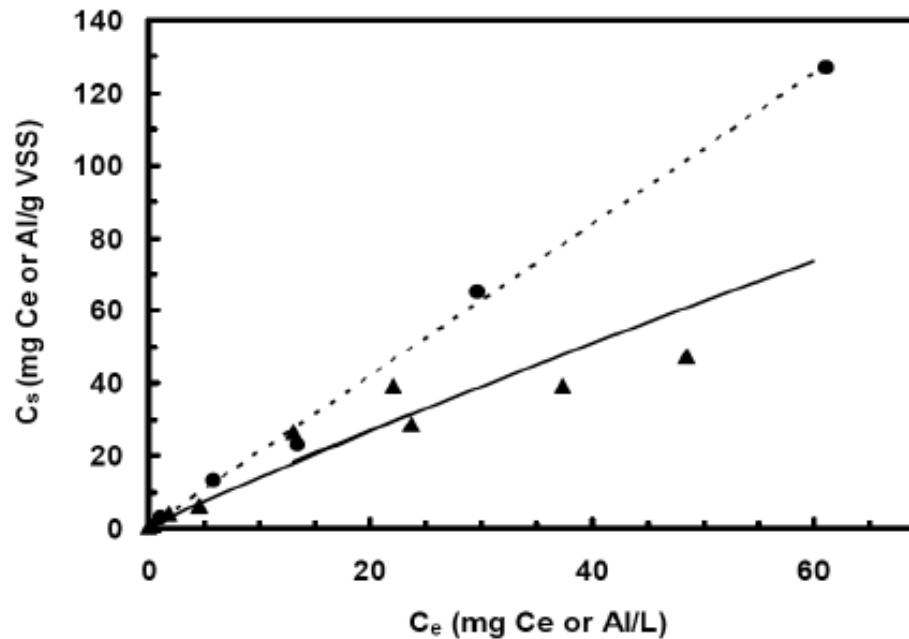
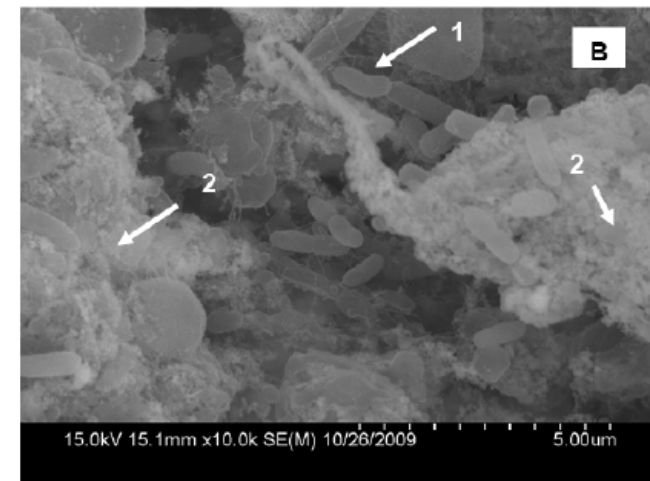
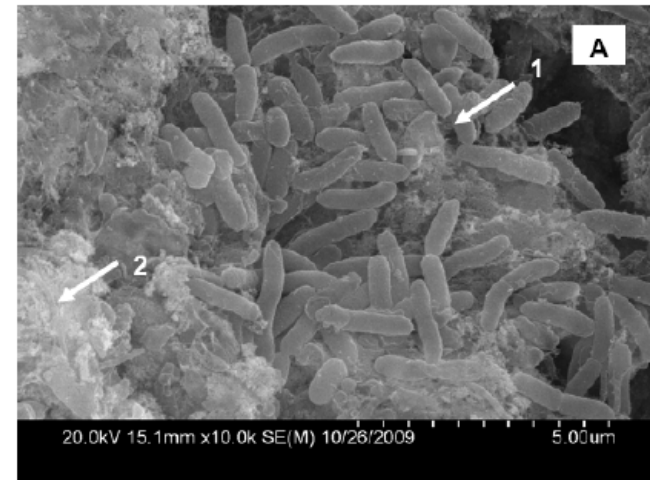


Fig. Partitioning of ceria (●) and alumina (▲) onto activated sludge collected from a municipal wastewater treatment plant. Experimental data fit to Freundlich model for ceria (dashed line) and alumina (solid line).

Removal of CMP Nanoparticles by Activated Sludge

SEM images of activated sludge collected from the bioreactor system fed with a wastewater containing CeO_2 NPs. The arrows indicate areas with bacterial cells (labeled 1), and areas where extracellular deposits (labeled 2) that are likely to contain large fractions of agglomerated ceria.

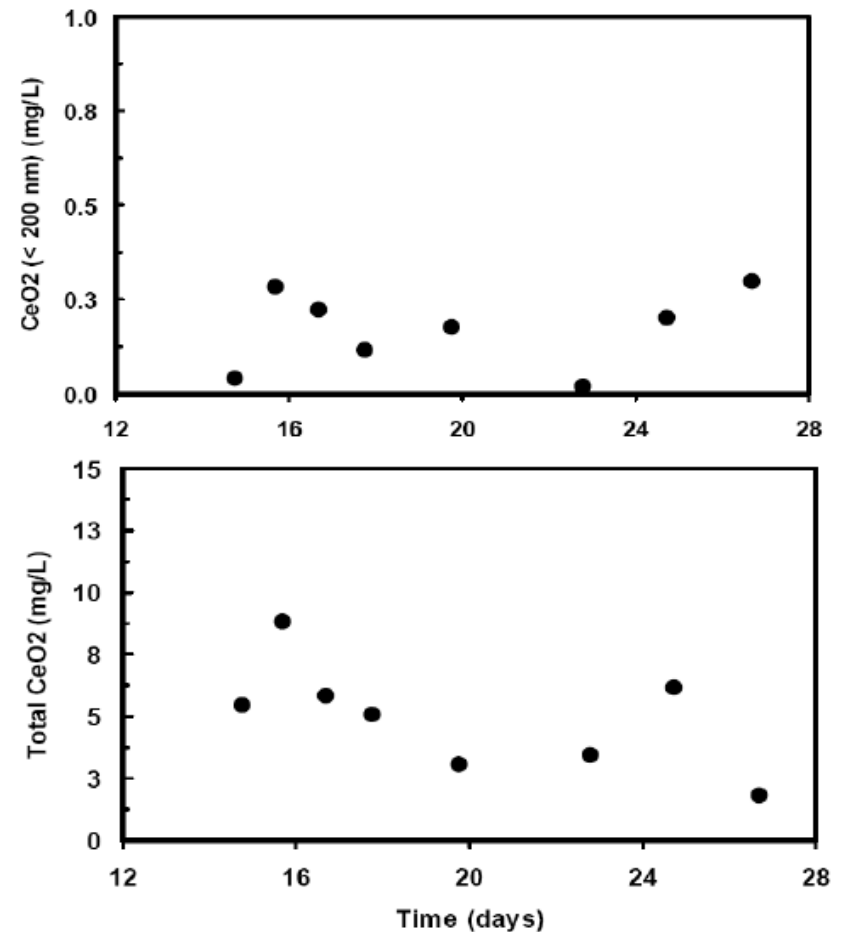


Removal of CMP Nanoparticles During Conventional Wastewater Treatment (Activated Sludge Process)

Conventional biological treatment provides high removal of CeO₂ NP.

NPs removed by adsorption onto the bacterial cell wall and entrapment into sludge flocs.

Fig. Concentrations of ceria (< 200 nm) (Top panel) and total ceria (Lower panel) in the treated effluent of a continuous lab-scale activated sludge system fed with nano-sized CeO₂ (75 mg/L)



Conclusions

- Treatment of effluents containing ceria or alumina NPs in conventional wastewater treatment plants is expected to result in a significant removal of the inorganic oxides from the treated effluent.
- Destabilization of the NP dispersions by constituents in the wastewater as well as association of the inorganic oxides with the activated sludge appears to be implicated in the removal mechanisms.
- SiO₂ NP dispersions were considerably more stable, even when diluted with municipal wastewaters or solutions containing proteins or amino acids that promoted aggregation of ceria and alumina NPs. These results suggest that a high fraction of SiO₂ NPs could escape wastewater treatment. Additional research is needed to test this hypothesis.

Industrial Interactions and Technology Transfer

- **Laurie Beu (ISMI / Sematech)**
- **Steve Brown (Intel)**
- **Reed Content (AMD)**
- **Art Fong (IBM)**
- **Peter Maroulis (Air Products)**
- **Brian Raley (Global Foundries)**
- **Tim Speed (IBM)**
- **Steve Trammel (ISMI / Sematech)**
- **Tim Yeakly (TI)**

Future Plans

Next Year Plans

- **Writing scientific publications resulting from the project**
- **This 7-month project was completed on Nov. 2009**
- **Funding is being sought to continue research work on:**
 - **Fate and transport of SiO₂ and CeO₂ nanoparticles in aquatic environments, including wastewater and surface water**
 - **Innovative methods for the removal of abrasive nanoparticles from CMP wastewaters.**

Publications, Presentations, and Recognitions/Awards

- Sierra-Alvarez, R., F. Shadman, A. Philipossian. 2009. Laboratory-Scale Assessment of the Fate of Chemical Mechanical Polishing (CMP) Nanoparticles Through Standard Wastewater Treatment Systems FATE Technology Transfer 09115058A-ENG. International SEMATECH Manufacturing Initiative, Inc. Austin, TX. Dec. 14, 2009.
- Three papers accepted for presentation by students at the 32nd Annual SESH International High Technology ESH Symposium and Exhibition. Scottsdale, AZ, April 26-29, 2010.
- Sierra-Alvarez, R et al. 2010. Removal of CeO₂ Nanoparticles in Semiconductor Manufacturing Effluents during Activated Sludge Treatment. 2010. 7th Leading Edge Conference on Water and Wastewater Technologies, Phoenix, AZ (June 2-4). (Accepted for presentation)
- Sierra-Alvarez, R., Barbero, I., M. Rodriguez, J. Rottman, F. Shadman, J.A. Field, R. Fate of Chemical Mechanical Polishing (CMP) Nanoparticles in Activated Sludge Processes (Paper in preparation)
- Field JA, Sierra-Alvarez. 2010. Nanoparticle Interaction with Biological Wastewater Treatment Processes. Brown Bag Water Sustainability Series, UofA Maricopa County Cooperative Extension. Phoenix, AZ. Jan 20.
- Barbero, I., M. Rodriguez, J. Rottman, F. Shadman, J.A. Field, R. Sierra-Alvarez. 2009. Evaluation of the Fate of Nanoparticles in CMP Wastewater during Standard Wastewater Treatment. 2009 Graduate Symposium, Dept Chemical and Environmental, UA, Oct. 10 (Poster, 1st prize)

Reducing Water and Energy Usage in Batch and Single-Wafer Rinsing Tools

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Xu Zhang², Junseok Chae², Bert Vermeire² and Farhang Shadman¹**

¹ University of Arizona

² Arizona State University

Co-Sponsored by:

**ERC, Environmental Metrology Corp (EMC),
Freescale Semiconductor Inc., and Samsung Electronics**

Objective and Approach

Objective:

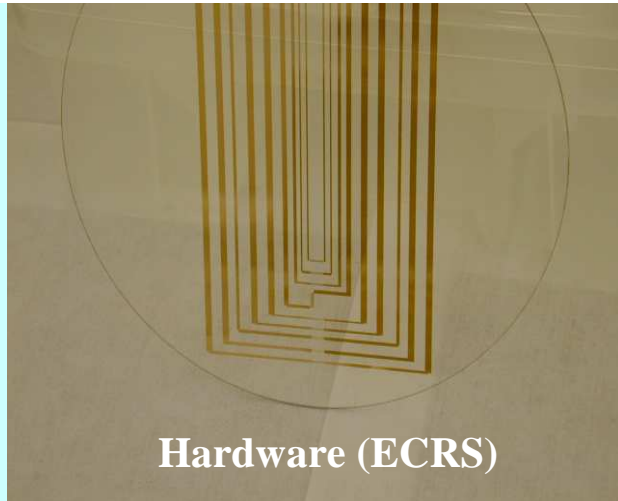
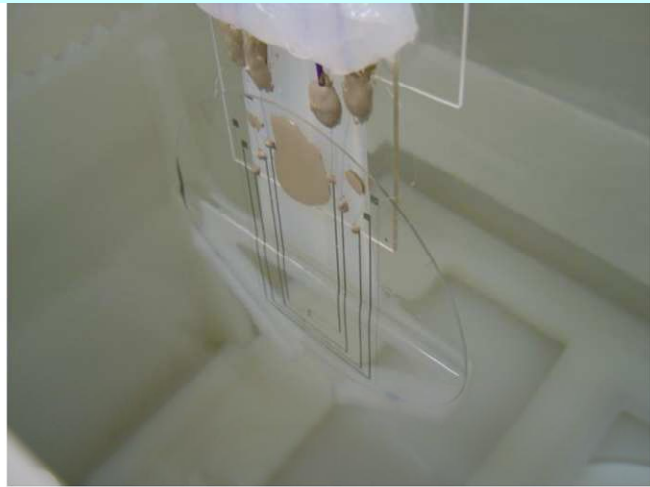
- Investigate the fundamentals of cleaning, rinsing, and drying of micro- and nano-structures; develop new technologies (hardware, process models, and process recipes) to reduce water, chemicals, and energy usage during these processes.

Method of Approach:

- Apply the novel Electro-Chemical Residue Sensor (ECRS) metrology method for in-situ and real-time monitoring of the dynamics of batch and single-wafer surface preparation.
- Combine metrology with process modeling to identify the controlling steps (bottlenecks) in the cleaning, rinsing, and drying of micro- and nano- structures.

Background: In-situ Metrology

Electro-Chemical Residue Sensor (ECRS)



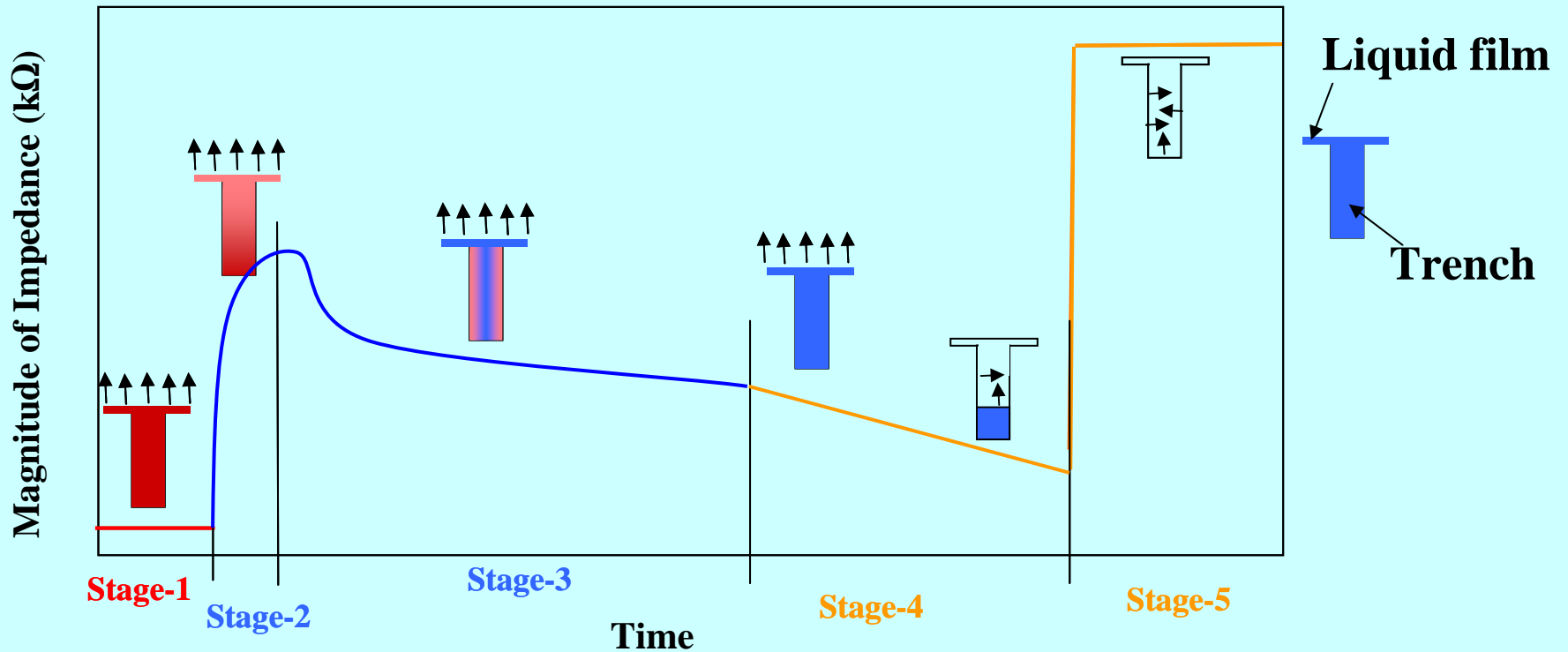
Hardware (ECRS)

Solution (pH)	UPW (pH=7)	HCl(pH=6)	HCl(pH=5)
Resistivity (M Ω)	18	2.3	0.23
Resolution (ppt)	5	30	400

Key Features

- Real Time
- In-situ
- Online
- High Sensitivity
- Non-destructive
- Quick Response

Monitoring Capabilities of ECRS



Stage-1: Chemical Exposure

Stage-2: Rinsing -Purging of the Trench

Stage 3: Rinsing: Surface Reaction

Stage-4: Bulk Drying

Stage-5: Surface Drying

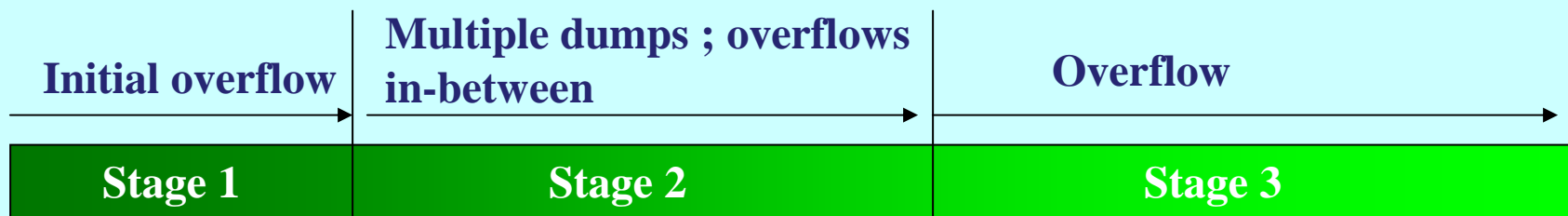
ECRS Application in Over-Flow and Quick-Dump Rinse Tools

Joint work with Freescale and EMC* on development of new low-water rinse processes using ECRS and process modeling.

Co-Investigators and Liaisons: Hsi-An Kwong, Marie Burnham, Tom Roche, Amy Belger, Stuart Searing, and Georges Robert

** EMC is a Engineering Research Center spin-off company that is formed for tech transfer and commercialization of ECRS technology*

Sample Results: Optimization of Rinse Recipe in Over-Flow and Quick-Dump Rinse Combination



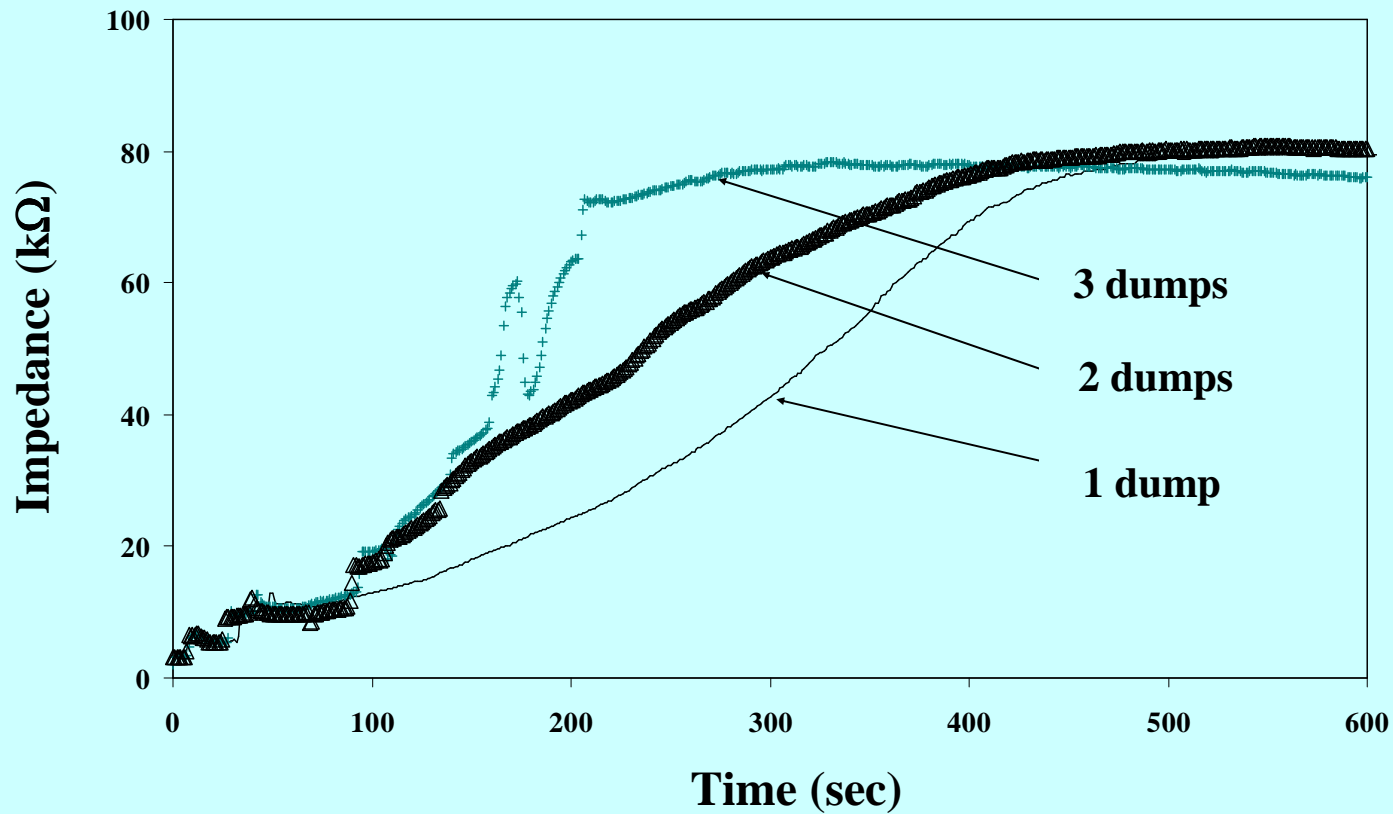
Process Parameters

- Flow rates at different stages
- Time for every session of overflow rinse
- Number of quick dumps
- Water temperature at every session

Collaboration with Freescale to optimize existing rinse recipes is ongoing.

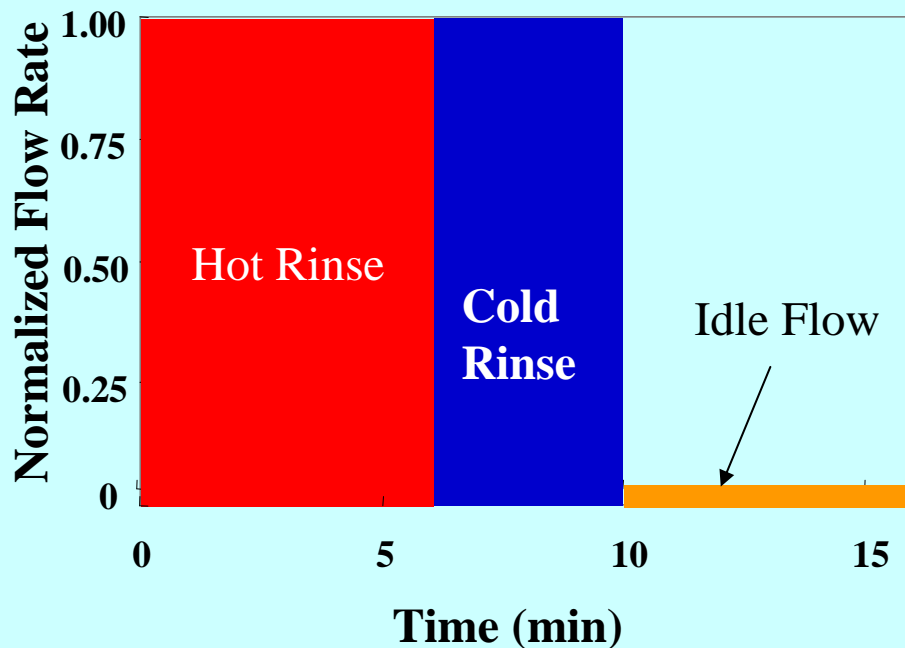
Effect of the Number of Dumps on the Post-APM Rinsing

Initial overflow for 5 sec; overflow in between for 5 sec;
flow rates are high flow at all stages; 32 °C

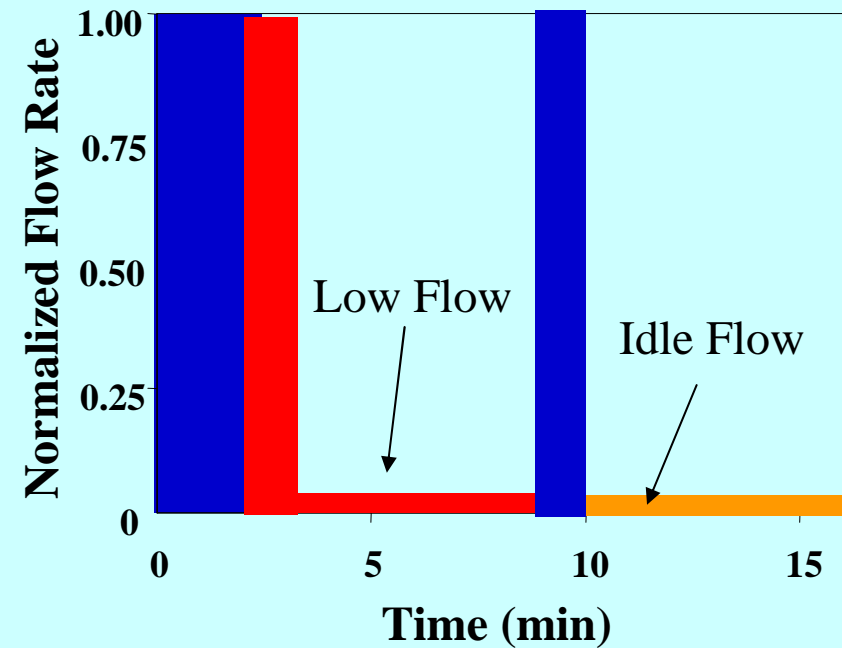


Example of a New Hot Rinse Schedule

Conventional Rinse



ECRS Enabled Rinse



- Use initial cold rinse to flush tank
- Use hot water to finish flush and heat wafers
- Cycle time is not increased
- Savings: ~ 25% cold water and ~ 80% hot water

ECRS Application in Single-Wafer **Rinsing and Drying**

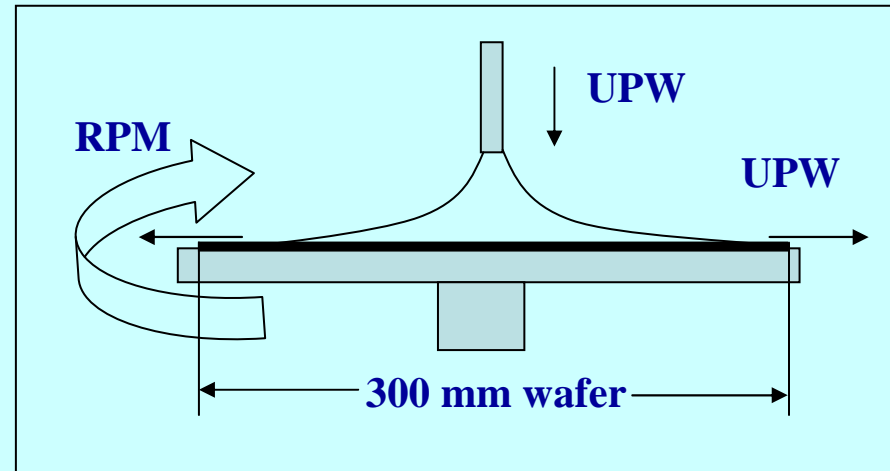
Joint work with Samsung and EMC on analysis of the dynamic of spin rinse and development of new low-water rinse processes.

**Samsung Co-Investigators and Liaisons:
Jeongnam Han, Seung-Ki Chae, Pil-Kwon Jun**

Application of ECRS to Single-Wafer Spin Rinsing



Experimental Setup



Process Model Schematic

- *A single wafer tool equipped with ECRS is designed and set up.*
- *Combination of experiments and process model is used to study the effect of various process parameters.*

Process Model for Spin Rinsing

Multi-component species transport equations :

$$\frac{\partial C_i}{\partial t} = \nabla \cdot (D_i \nabla C_i + z_i F \mu_i C_i \nabla \phi) - u \nabla C_i$$

$$u_r = \frac{\rho \omega^2 r h^2 (1 - (1 - \frac{z}{h})^2)}{2\mu} \quad h = 0.782 \left(\frac{Q\mu}{\rho \omega^2 r^2} \right)^{1/3}$$

Surface adsorption and desorption:

$$\frac{\partial C_{S2}}{\partial t} = k_{a2} C_2 (S_{02} - C_{S2}) - k_{d2} C_{S2}$$

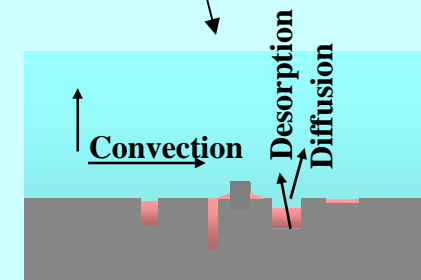
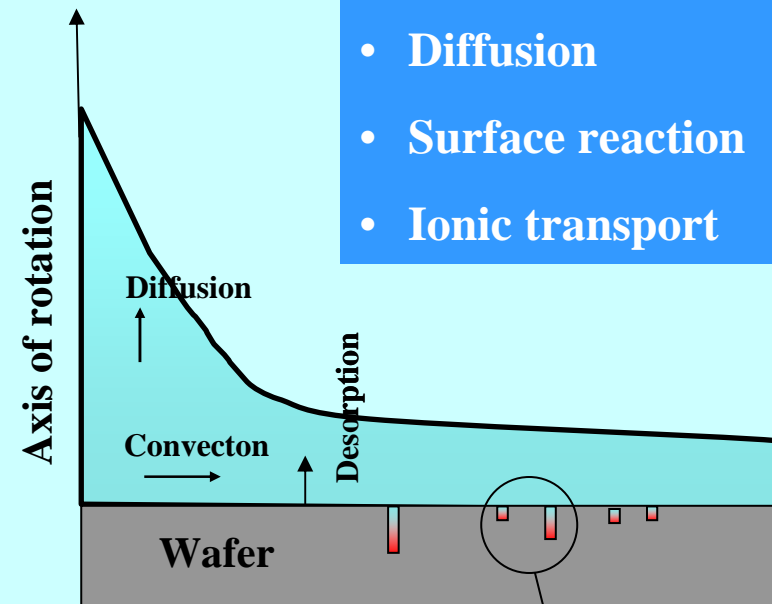
Poisson equation: $\nabla^2 \phi = -\frac{\rho}{\epsilon}$

where charge density: $\rho = F \sum_i z_i C_i$

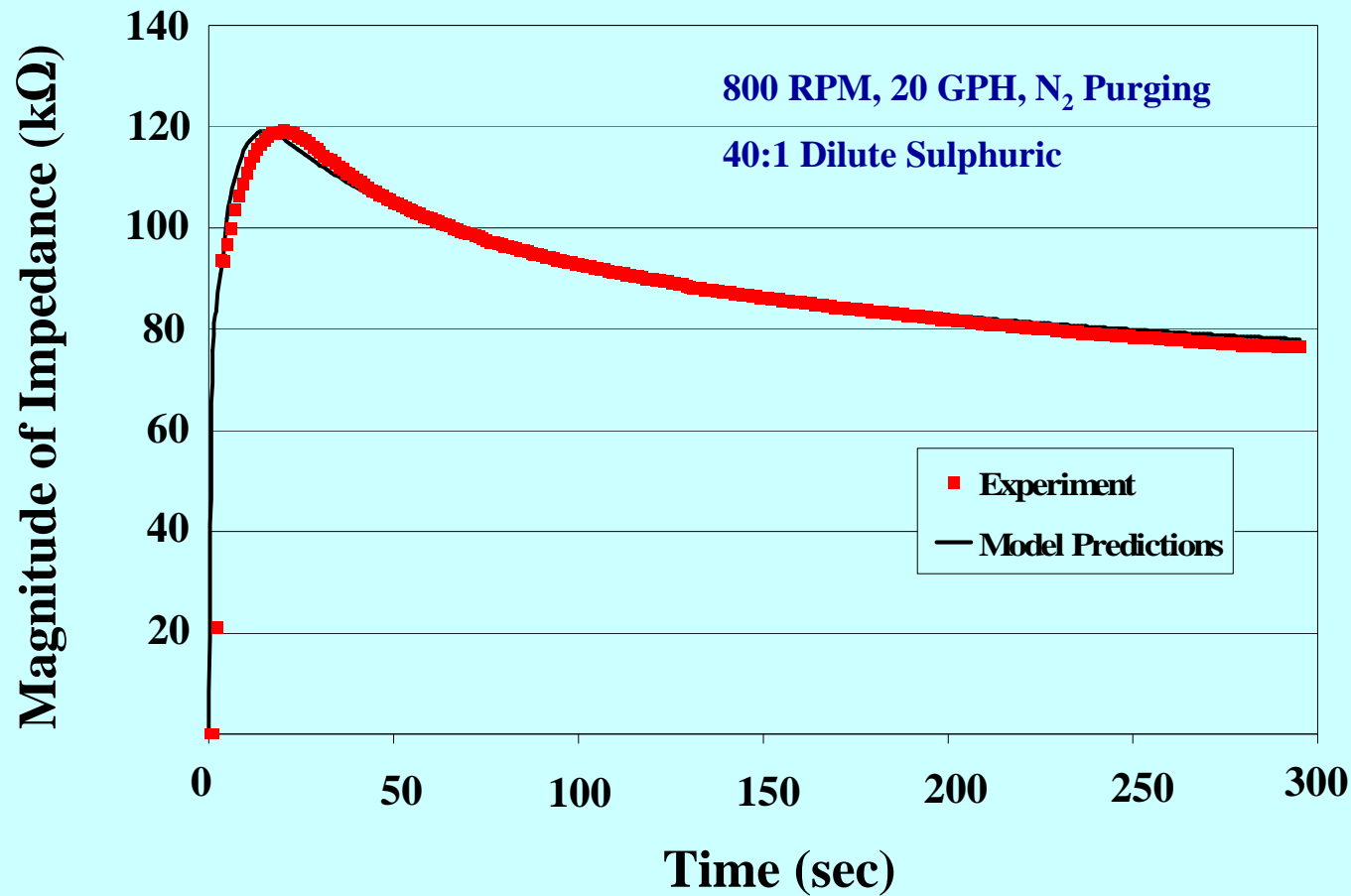
Ohm's law: $\vec{J} = \sigma \vec{E} \quad \nabla \times \vec{E} = 0$

where electrical conductivity: $\sigma = \sum_i \lambda_i C_i$

- Convection
- Surface Charge
- Diffusion
- Surface reaction
- Ionic transport



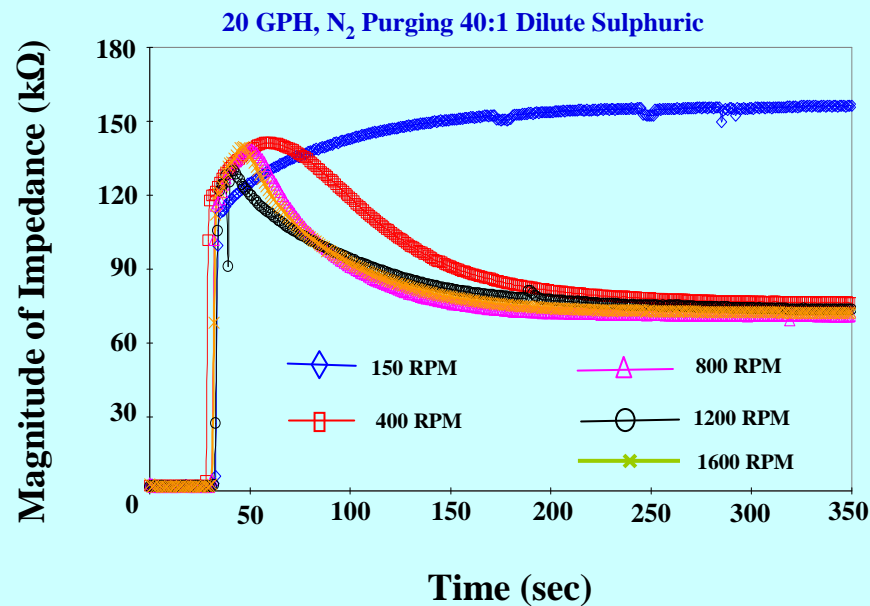
Validation of Process Simulation



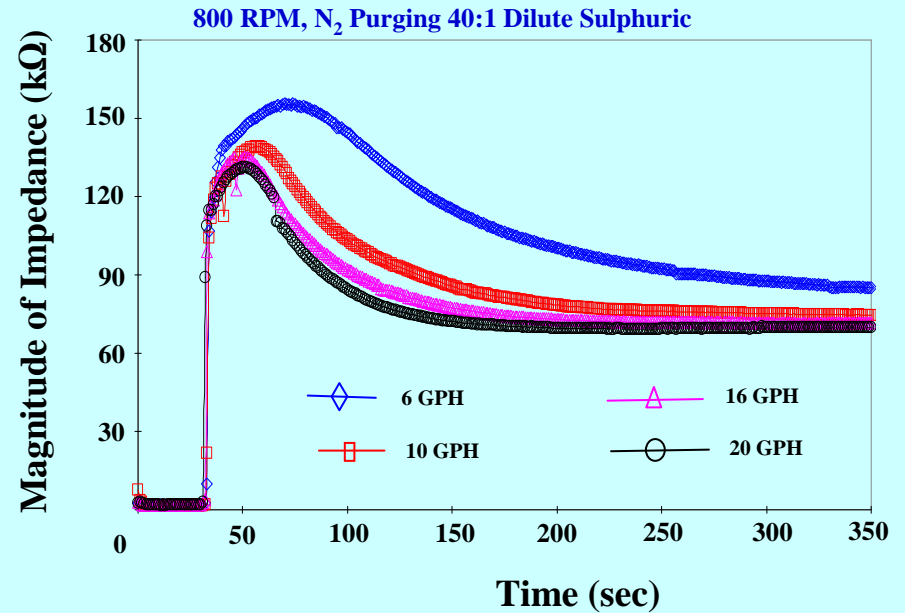
Simulation results are in good agreement with the experimental data

Effect of Process Parameters

Effect of Speed of Rotation

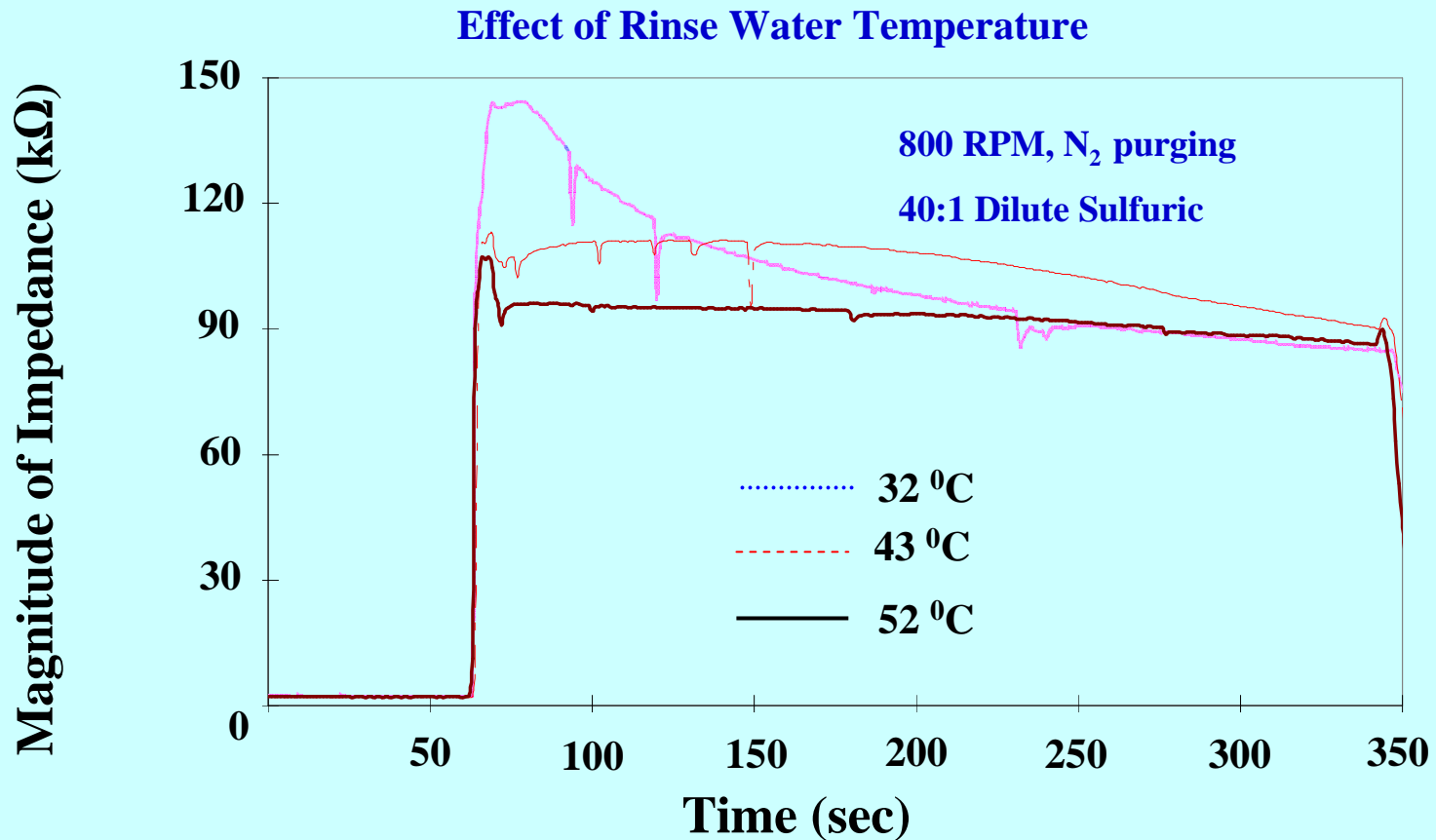


Effect of Flow Rate



- *Gain in rinse efficiency diminishes as we reach an optimum speed of rotation and rinse-water flow rate.*
- *Speed of rotation and water flow rate can be optimized to reduce rinse time and increase throughput.*

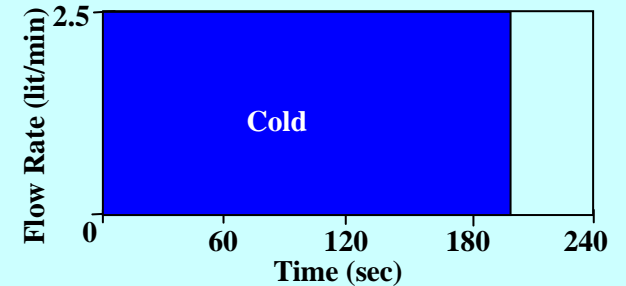
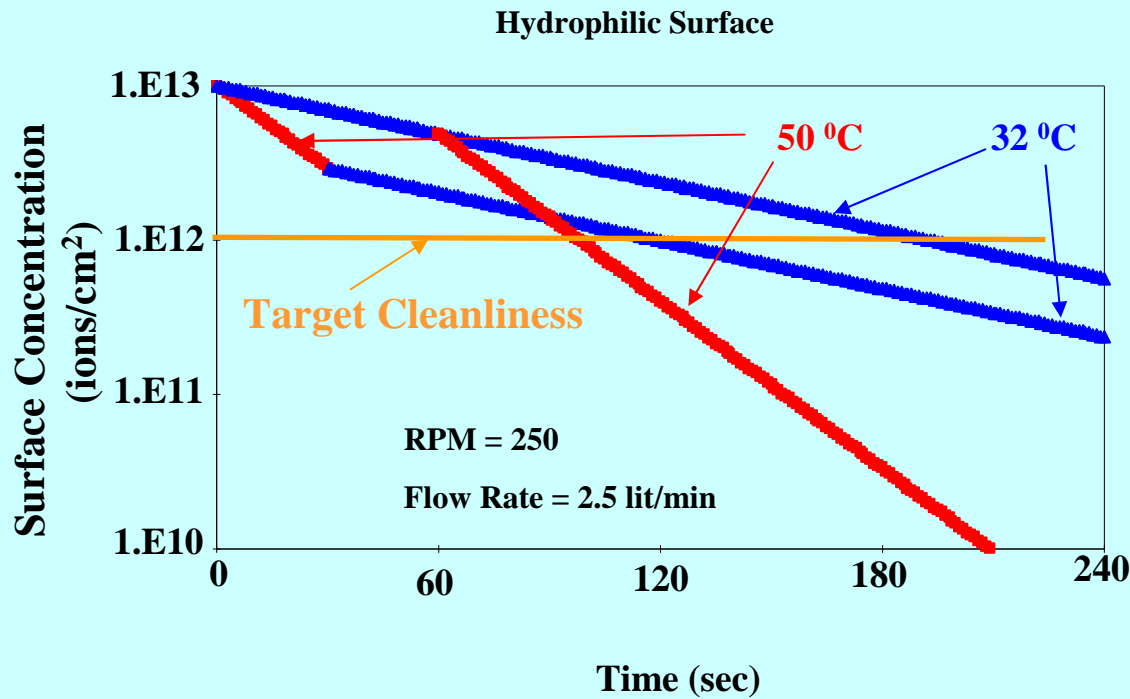
Effect of Process Parameters



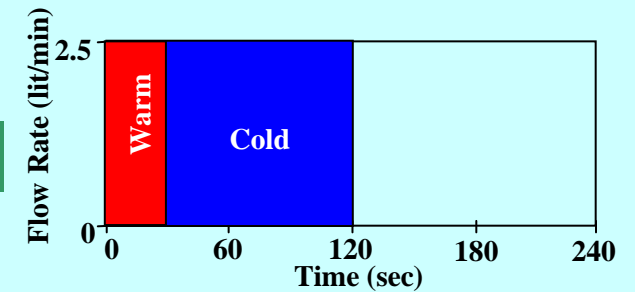
Temperature has significant effect on enhancing both the early stage and the final stage of the rinse process.

Benefits of Staged Rinsing

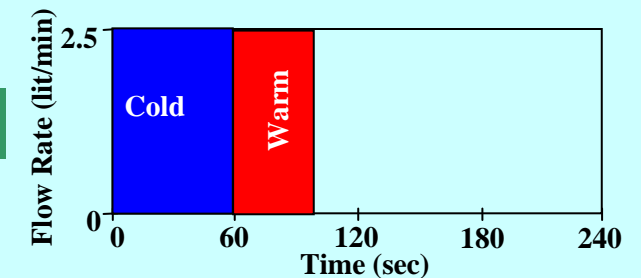
Post SC-1 Rinsing



1



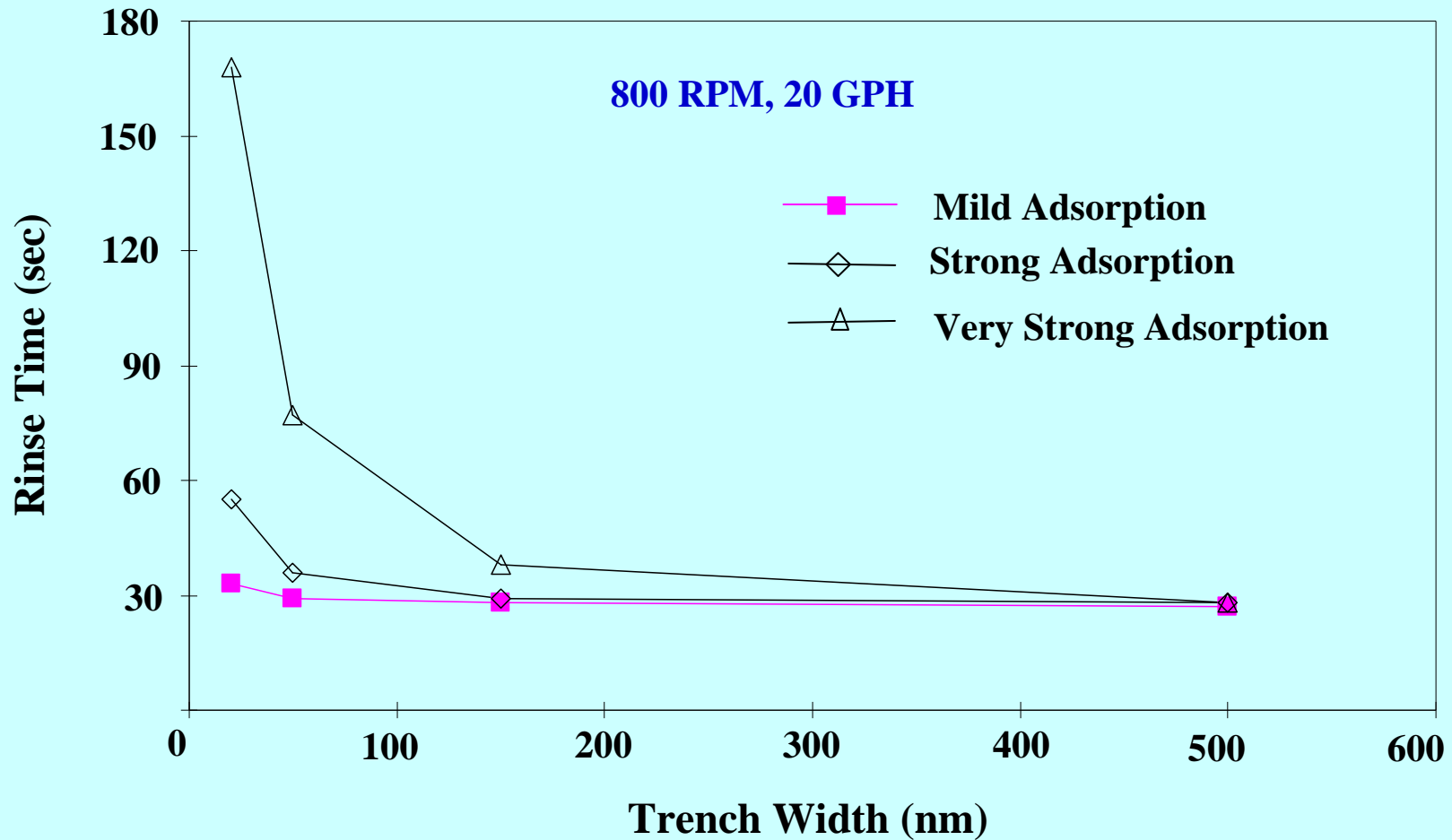
2



- *Staging temperature of UPW decreases rinse time without sacrificing cleanliness*
- *To reach 1.E12 ions/cm², staged rinsing leads to water savings of 40% for staged rinse “1” and 50% for staged rinse “2”.*

Effect of Feature Size

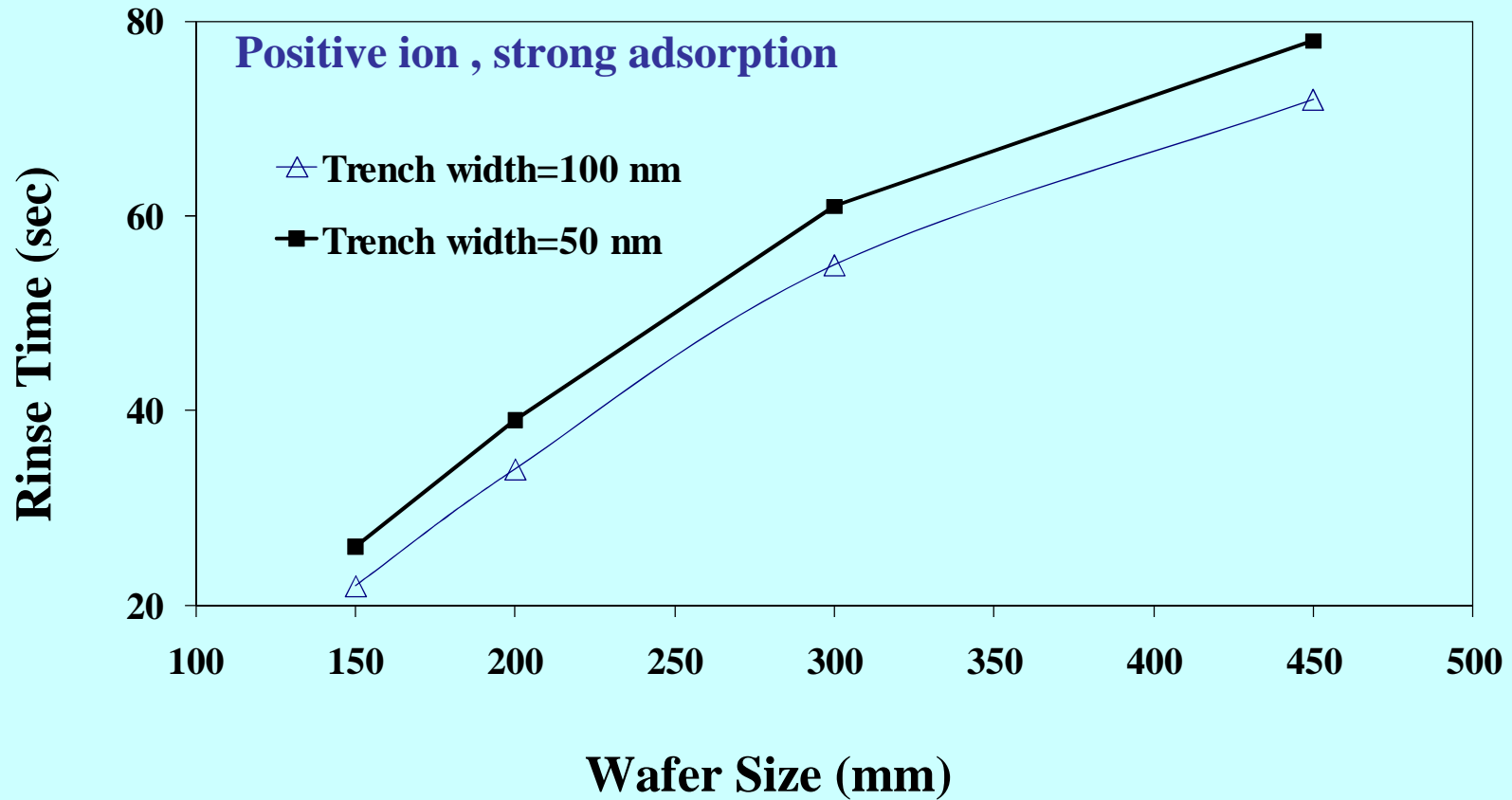
Rinse time to reach 10^{12} ions/cm² on the surface of the trench.



Required rinse time increases as feature size decreases.

Effect of Wafer Size

Rinse time to reach 10^{12} ions/cm² on the surface of the trench.



Required rinse time increases as wafer size increases.

Summary

- **Developed a new method for integrating ECRS with single-wafer tools.**
- **Developed a comprehensive process simulator and verified it for single-wafer rinsing; the simulator is applicable to rinsing and cleaning of fine structures on patterned wafers.**
- **Applied ECRS to determine the effect of key process parameters such as speed of rotation, flow rate, rinse water temperature, feature size, and wafer size (a unique and first-time study of these effects).**
- **The ECRS technology and the new process simulator were used to develop rinse recipes that result in significant reduction in usage of water and energy (hot water) for both batch and single-wafer rinse tools (joint work with Freescale and Samsung).**
- **Work is continuing on the wireless version of the ECRS (details on the poster)**
- **Future work will also include applying this new technology to drying processes.**

ECRS: Winner of "Best Product 2009" Award

6...



Semiconductor International Announces 2009 Editors Choice Best Product Award Winners

Wed Jul 15, 8:32 AM

[Email Story](#) [IM Story](#) [Printable View](#)

OAK BROOK, Ill.--(BUSINESS WIRE)--*Semiconductor International* bestowed its 2009 Editors' Choice Awards for excellence in semiconductor manufacturing on 15 commercially proven industry products. These awards will be presented at a ceremony in San Francisco on Wednesday, July 15, during SEMICON West. The 2009 winners exemplified state-of-the-art equipment and materials installed and used in numerous fabs around the world.

The 2009 Editors' Choice Best Product Award winners are:

AMEC	Primo D-RIE dielectric etcher
Applied Materials Inc.	SEMvision G4 defect review platform
ASM Technology Singapore	IDEALcompress encapsulation system
ATMI Inc.	AutoClean ion implant cleaning process
ATMI Inc.	Safe Delivery Source (SDS) cylinder
Cabot Microelectronics Corp.	Epic D100 CMP pad
Carl Zeiss SMT AG	ULTRA plus field-emission SEM
CI Semi	WetSpec200 in-line chemical analyzer
Environmental Metrology Corp.	Electro-Chemical Residue Sensor (ECRS)
Linde Group	Generation-F 80 on-site fluorine generator
Nikon Precision Inc.	NSR-S610C ArF immersion scanner
Nova Measuring Instruments Ltd.	NovaMARS optical CD software
Octect Technologies	ChemetriQ 3000 inspection system
Tec-Sem AG	Pr@ctor 300 mm single wafer management
W.L. Gore & Associates Inc.	GORE ultrapure water filters

"The Editors' Choice Best Products awards program acknowledges products, materials and services that are proven in the manufacturing environment," said Laura Peters, Editor-in-Chief of *Semiconductor International* (SI). In the evaluation process, SI's editors consider the products based on feedback from

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Press Release
Semiconductor International Announces 2009 Editors' Choice Best Product Award Winners
 07.15.09, 08:30 AM EDT

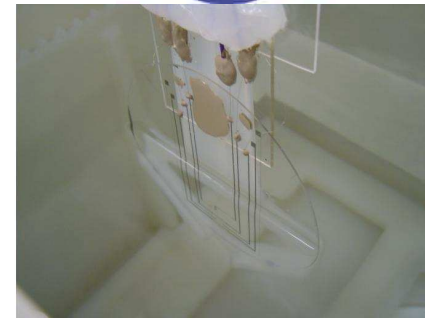
BusinessWire - Semiconductor International bestowed its 2009 Editors' Choice Awards for excellence in semiconductor manufacturing on 15 commercially proven industry products. These awards will be presented at a ceremony in San Francisco on Wednesday, July 15, during SEMICON West. The 2009 winners exemplified state-of-the-art equipment and materials installed and used in numerous fabs around the world.

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 Singapore IDEALcompress encapsulation system ATMI Inc.
 AutoClean ion implant cleaning process ATMI Inc. Safe Delivery Source (SDS) cylinder Cabot Microelectronics Corp. Epic D100 CMP pad Carl Zeiss SMT AG ULTRA plus field-emission SEM CI Semi WetSpec200 in-line chemical analyzer Environmental Metrology Corp. Electro-Chemical Residue Sensor (ECRS) Linde Group Generation-F 80 on-site fluorine generator Nikon Precision Inc. NSR-S610C ArF immersion scanner Nova Measuring Instruments Ltd. NovaMARS optical CD software Octect Technologies ChemetriQ 3000 inspection system Tec-Sem AG Pr@ctor 300 mm single wafer management system W.L. Gore & Associates Inc. GORE ultrapure water filters

"The Editors' Choice Best Products awards program acknowledges products, materials and services that are proven in the manufacturing environment," said Laura Peters, Editor-in-Chief of *Semiconductor International* (SI). In the evaluation process, SI's editors consider the products based on feedback from actual customers in the field and only the most highly recommended ones are honored each year.

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Seed Project: Understanding, Characterizing and Controlling Pad Debris Generation during Copper CMP

PIs:

- **Takenao Nemoto, New Industry Creation Hatchery Center, Tohoku University**
- **Ara Philipossian, Chemical and Environmental Engineering, UA**

Graduate Students:

- **Yubo Jiao, PhD candidate, Chemical and Environmental Engineering, UA**
- **Zhenxing Han, PhD candidate, Chemical and Environmental Engineering, UA**

Other Researchers:

- **Yasa Sampurno, Research Associate, Chemical and Environmental Engineering, UA**
- **Yun Zhuang, Research Associate, Chemical and Environmental Engineering, UA**
- **Xun Gu, Ph. D. candidate, Graduate School of Engineering, Tohoku University**
- **Siannie Theng, Research Technician, Chemical and Mechanical Engineering, UA**

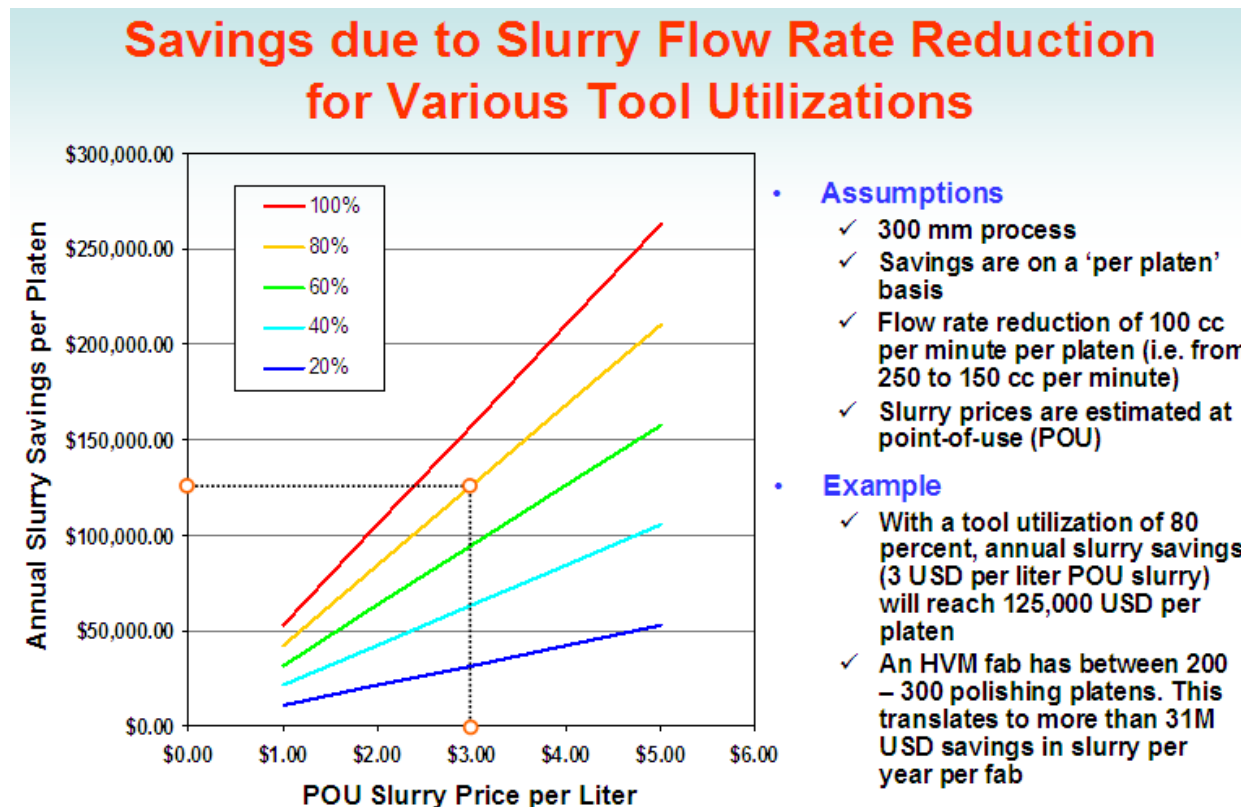
Objectives

Reduce consumables in CMP by extending pad life and by reducing slurry flow

- **Quantify the extent of pad debris generated during conditioning using different industry-standard pads and diamond disc conditioners**
- **Compare the polishing performance of standard vs. novel slurry injection methods on the extent of pad debris resident on the pad surface, polish performance and wafer defects during copper CMP**

ESH Metrics and Impact

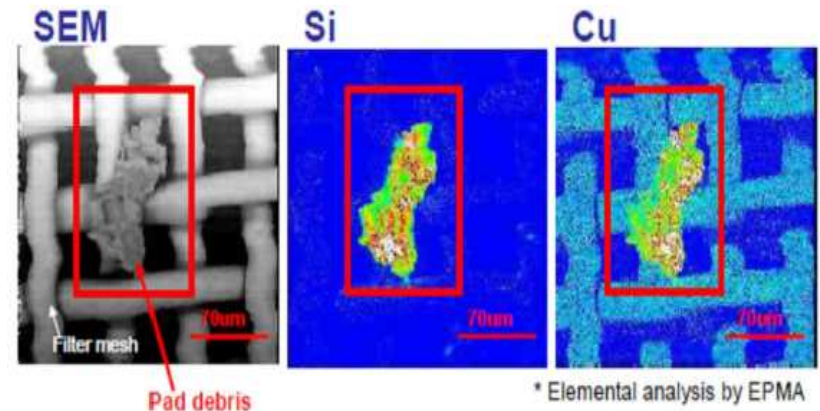
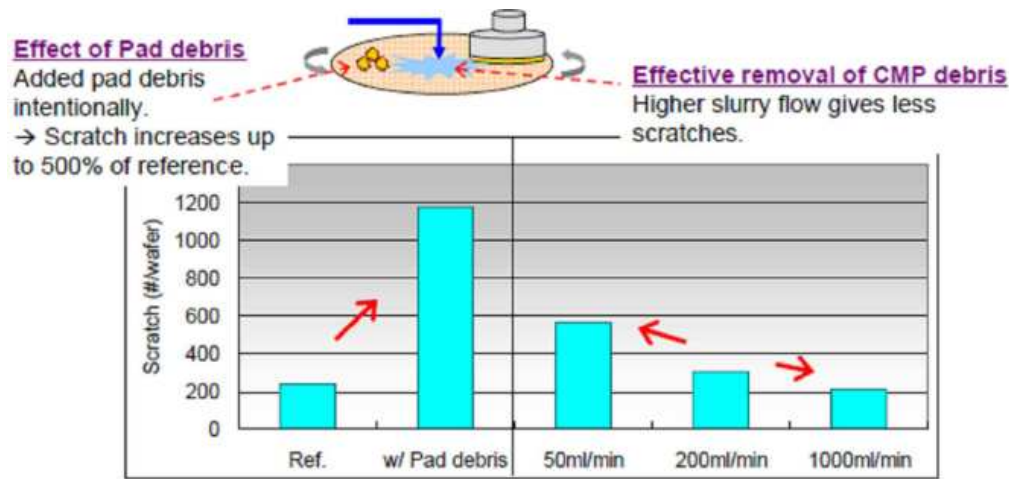
1. Increase yield by reducing wafer-level defects
2. Reduce slurry consumption (and associated waste) by 25 – 50 %
3. Increase pad life by more effectively removing conditioner-induced pad debris during polishing



Approach

- **Define and Implement a systematic characterization method for quantifying pad debris generated as a result of conditioning on the pad surface.**
- **Quantify the extent of pad debris generated during pad conditioning using different industry-standard pads and conditioning discs.**
- **Select a pad and a disc combination based on the above study. Perform polishing to determine whether extent of pad debris generation is correlated to wafer-level defects.**
- **Determine whether a novel slurry injection method (that also requires 2X less slurry) is effective in preventing pad debris from entering the pad-wafer interface.**
- **Determine whether the above novel injection method also reduces wafer-level defects.**

Effect of Pad Debris on Wafer-Level Defects



- ✓ Pad debris exhibits apparent effect on scratch generation.
- ✓ Scratch count decreases with higher slurry flow. Efficient debris removal would be one of the factors of scratch reduction.

Elemental analysis suggests adhesion of silica particle and Cu-complex on the pad debris.

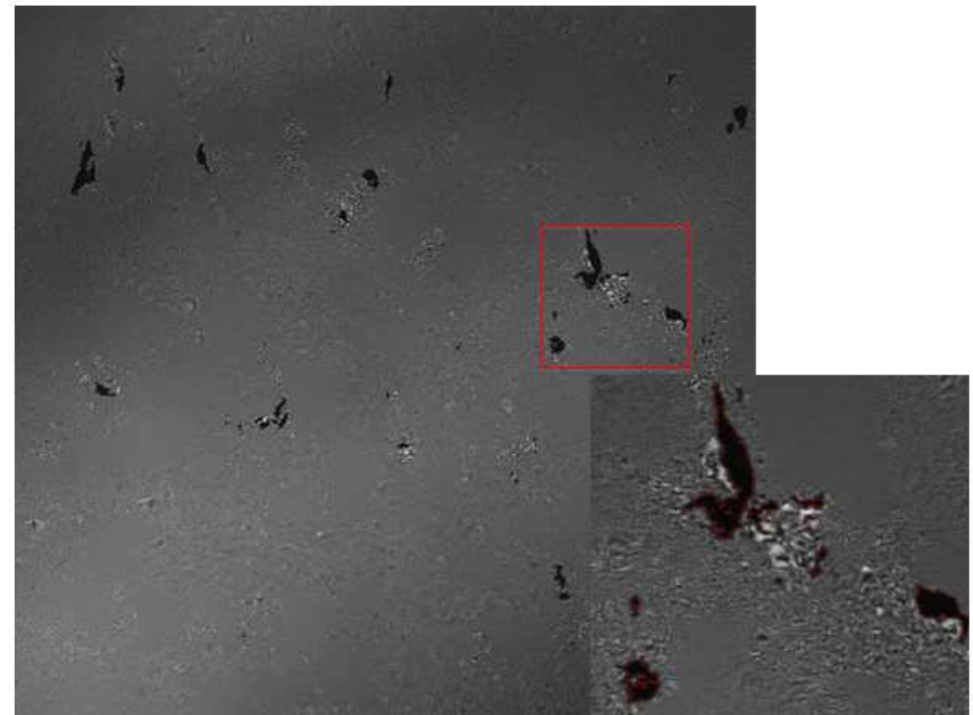
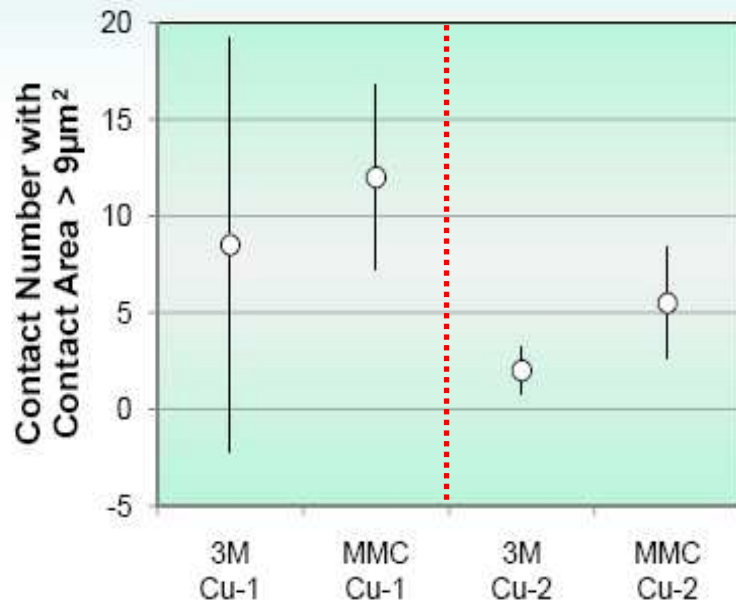
Source: JSR Corporation (2008)

- Pad debris have been shown to be sources of scratch defects during CMP
- JSR's proposed solution is to reduce pad debris by increasing slurry flow rate by 5X
- This goes against EHS and COO objectives

Confocal Microscopy Images of Pad Debris

Based on an earlier joint study by UA and Tohoku University

Pad debris is defined as a contact area greater than $9 \mu\text{m}^2$

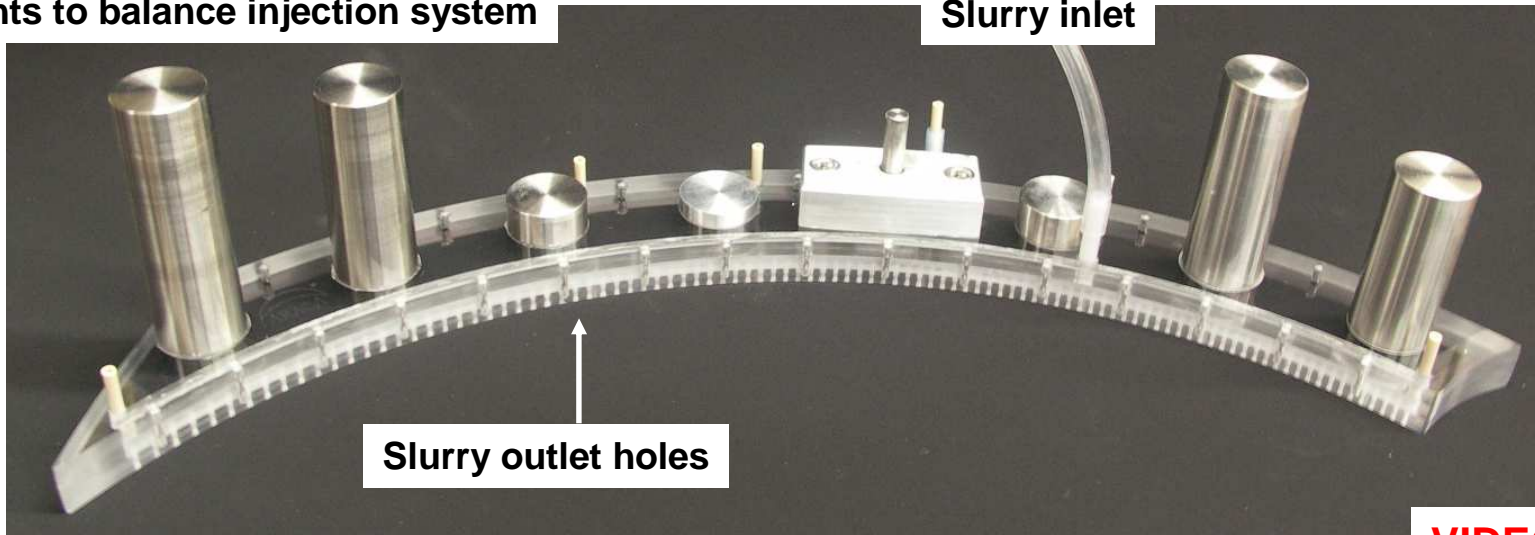


- MMC disc generates more pad debris than 3M disc.
- We believe that pad debris is the source of defects during CMP.
- Cu - 2 process generates less pad debris than Cu - 1.

Novel Slurry Injection System

Weights to balance injection system

Slurry inlet



Slurry outlet holes

VIDEO NO. 1

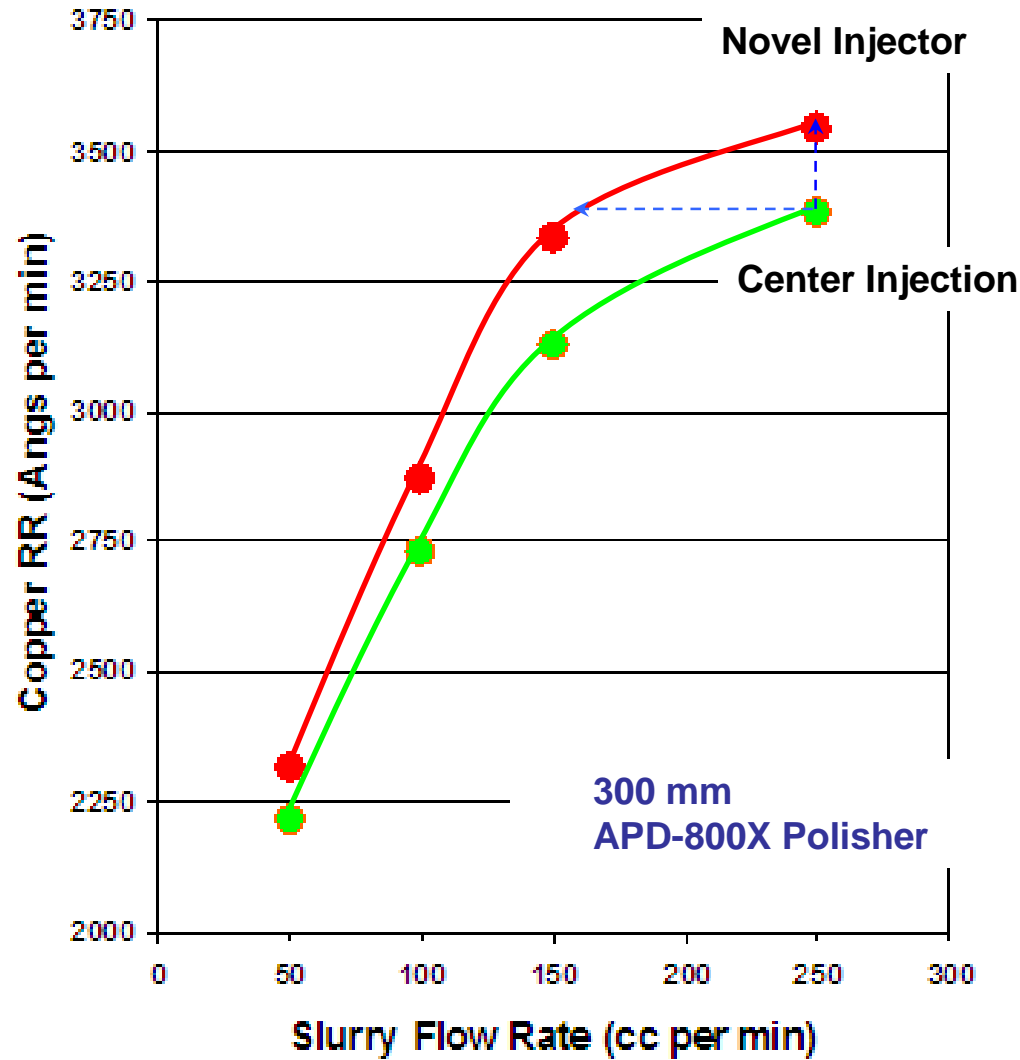


exhaust

Pad

conditioner

RR vs. Flow Rate Data Comparison



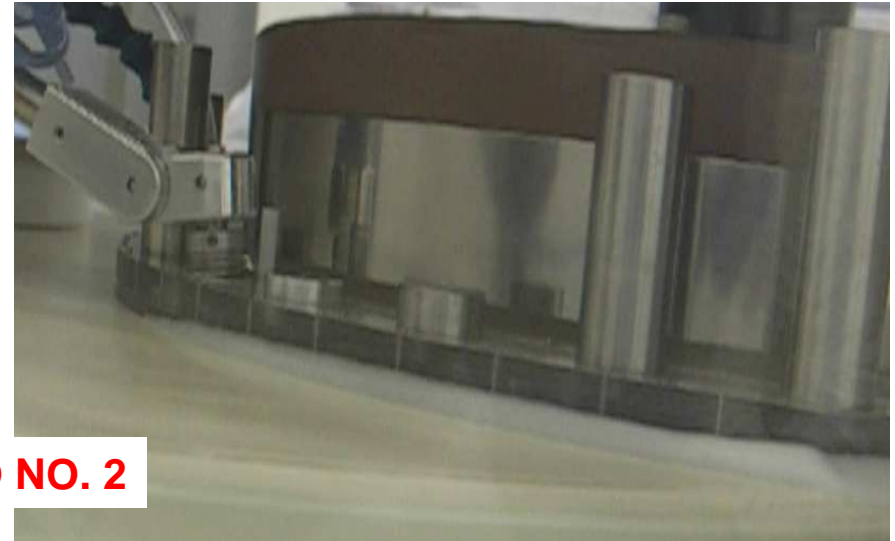
Benefits of Novel Slurry Injection System

- **Fresh slurry is applied exactly where it is needed**
- **The application method minimizes fresh slurry loss from the wafer or the retaining ring bow wave**
- **The device minimizes mixing of rinse water and used slurry with fresh slurry**
- **Preliminary results show that wafer-level defects are reduced**
- **The device can partially block foamed slurry from getting in the pad-wafer interface during polishing**
- **The design reduces slurry residue build-up on the pad**

It is not clear if the injection method will also be effective in preventing pad debris from getting into the pad-wafer interface during polishing – This study aims to address this matter

Novel Slurry Injection System

Removing Slurry Foam to Improve Defects

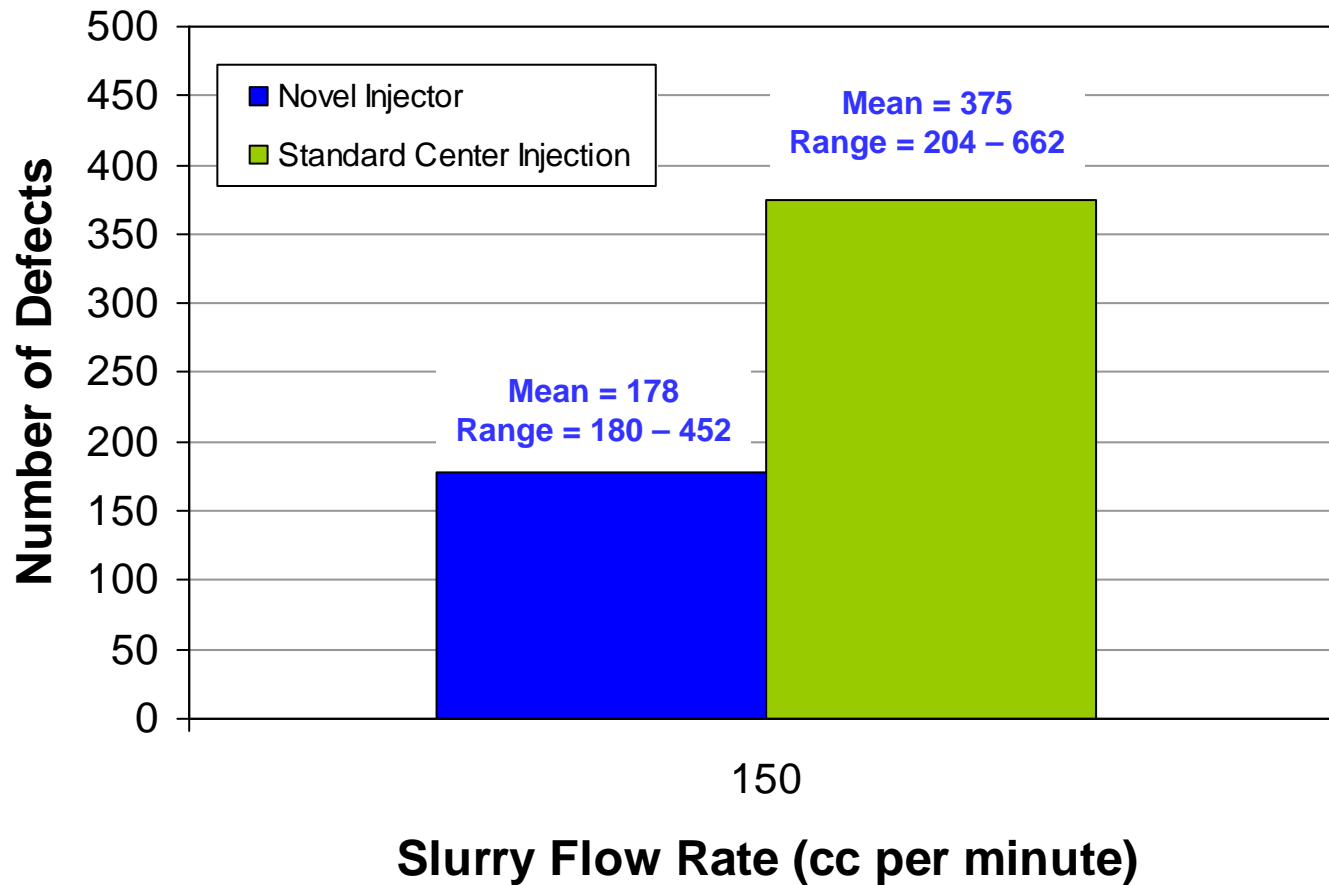


VIDEO NO. 2

- Slurry foam is formed throughout polishing process
- Slurry foam is a major issue in IC manufacturing since most fabs are forced to change polishing parameters to suppress foam formation
- Novel slurry injection system can effectively screen out the foam from entering the polishing region without modifying polishing parameters

Wafer-Level Defects Comparison

200 mm COPPER
on APD – 800X Polisher
(SP1 defects > 250 nm at 5 mm EE)



Industrial Interactions and Technology Transfer

Industrial mentors and contacts:

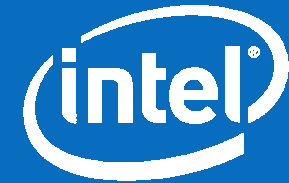
- **Leonard Borucki (Araca Incorporated)**
- **Tadahiro Ohmi (Tohoku University)**
- **SK Lee and Peter Ojerholm (Ehwa)**
- **Shiro-san (Toray)**
- **Chris Wargo (Entegris)**
- **Cliff Spiro (CMC)**

Plans (Only 1 Year Project)

- **Define and Implement a systematic characterization method for quantifying conditioner-induced pad debris.**
- **Quantify the extent of conditioner-induced pad debris using different industry-standard pads and conditioning discs.**
- **Select a pad and a disc combination based on the above study and perform polishing studies to determine whether pad debris generation correlates to wafer-level defects.**
- **Determine whether a novel slurry injection method (that also requires 2X less slurry) is effective in preventing pad debris from entering the pad-wafer interface.**
- **Determine whether the above novel injection method also reduces wafer-level defects.**



ERC – Intel Customized Joint Pilot Program



*Expected Outcome: New Technologies on Environmentally–
Friendly High-Volume Nano-Manufacturing*

Gopal Rao, Intel

Intel/ERC Steering Committee: Gopal Rao (Co-Chair), Prof Farhang Shadman (Co-Chair), Prof Ara Philipossian, Avi Fuerst, Jim Jewett, Mansour Moinpour, Don Hooper, Carl Geisert, Dan Hodges, John Harland, David Harman

Acknowledgements

Executive Initiative:

Dr. Robert Shelton (President of UA)

Josh Walden (VP/GM of FSM)

Gabe Quenneville (Plant Manager, FSM)

Intel FSM Research Management Review Committee

PIs Team:

Intel : Avi Fuerst, Don Hooper, Mansour Moinpour, Carl Geisert, Dan Hodges

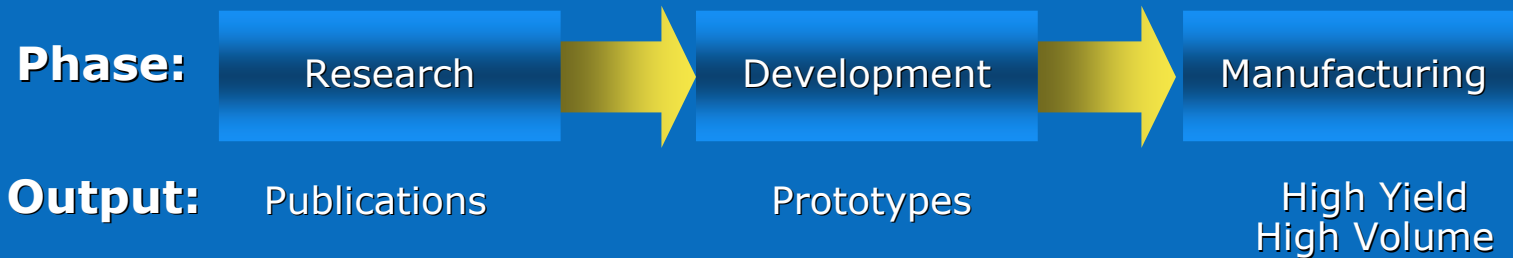
UA: Farhang Shadman, Ara Philipossian, Srini Raghavan, Jim Baygents, Jim Farrell

Current Intel/ERC Customized Research Projects

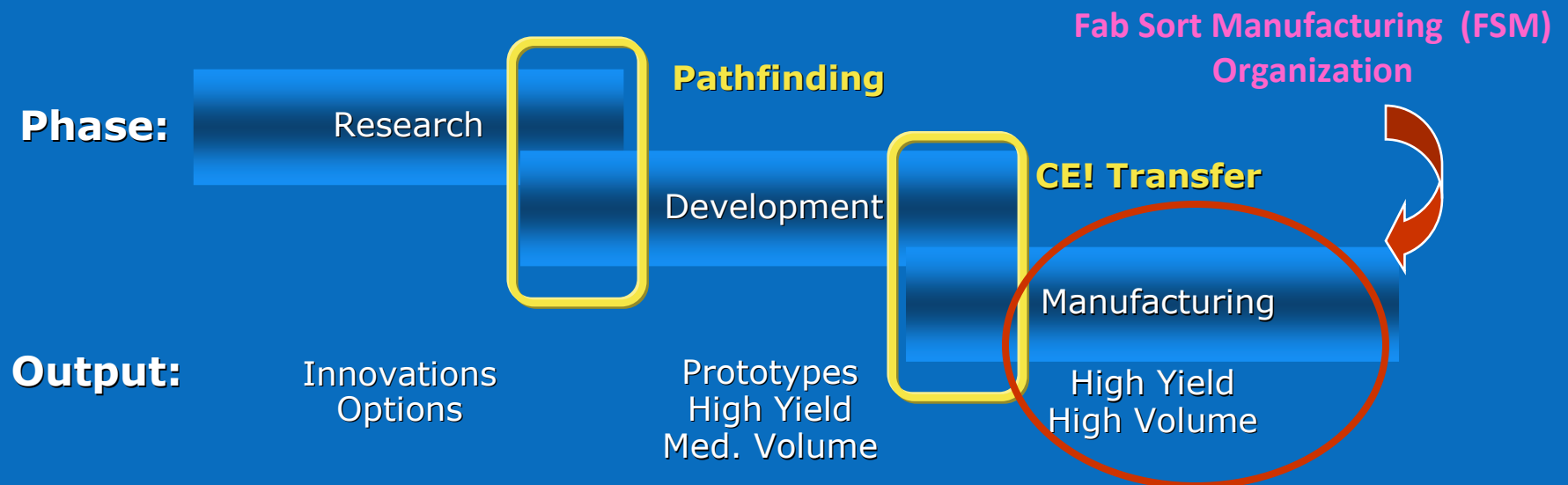
No	Project	Lead ERC PI	Lead Intel PI	Research Duration
1.	Investigation of the Relationship between Planarization & Pad Surface Micro-Topography	Ara Philipossian	Mansour Moinpour	3 yrs
2.	Retaining Ring and Conditioner Interactions	Ara Philipossian	Don Hooper	2 yrs
3.	Contamination Control in Gas Distribution Systems of Semiconductor Fabs	Farhang Shadman	Carl Geisert	1.5 yrs
4.	Develop an AFM based methodology to determine the optimal APM composition to remove particles from surfaces	Srini Raghavan	Avi Fuerst	2 yrs
5.	Integrated Electrochemical Treatment of CMP waste streams for water reclaim and conservation	James Baygents James Farrell	Don Hooper	2 yrs

Intel's Process Technology R-D-M Flow

Traditional R-D-M Method:



Intel's R-D-M Method:



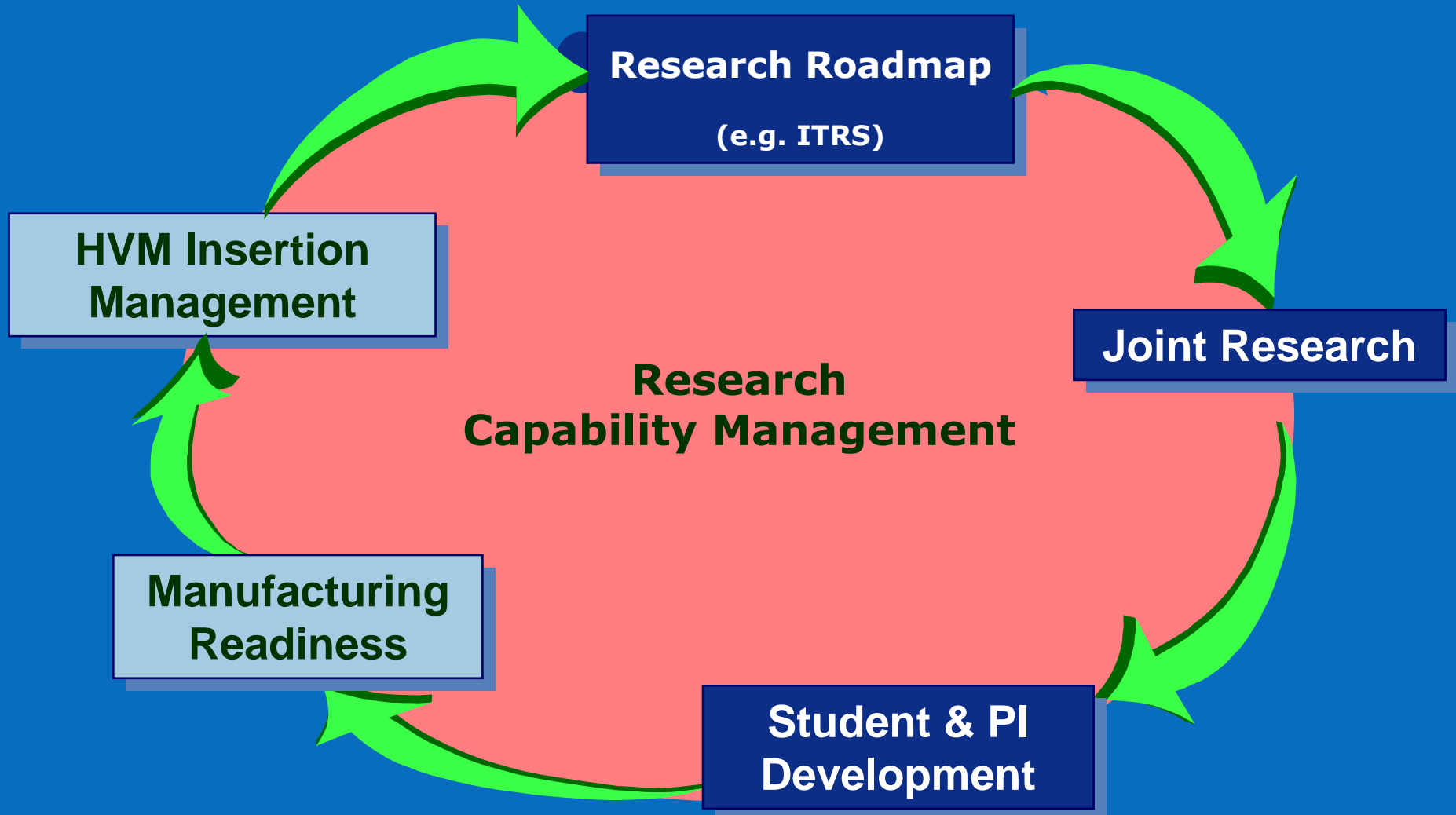
Justification for Research in Manufacturing

- Moore's Law enabling significant technology development
- Manufacturing methods, techniques and innovation must keep pace with our technology roadmap
- Leveraging universities for manufacturing research is a good option to explore fundamental and innovative concepts and theories
- FSM resources optimized for performing research in manufacturing

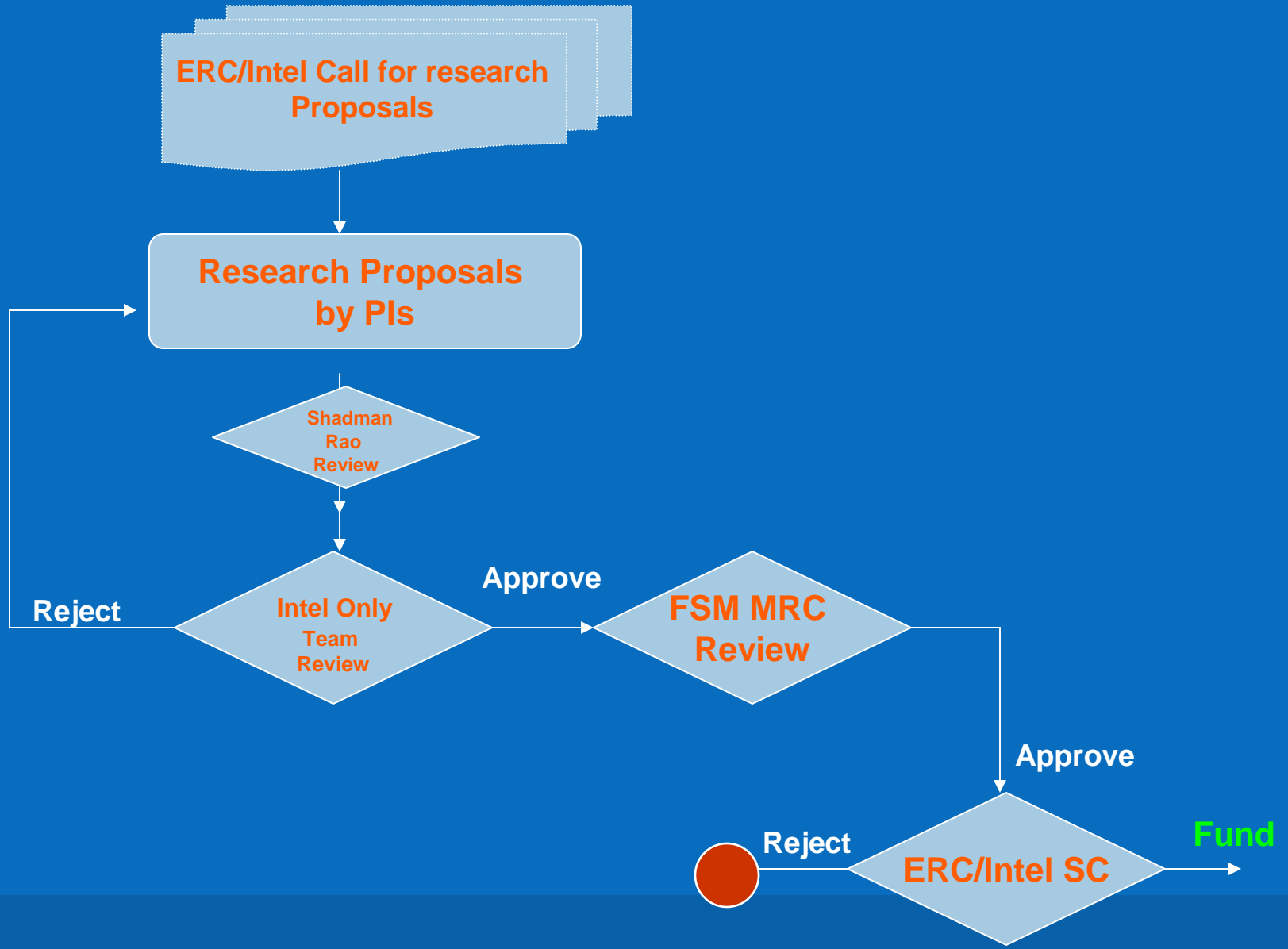
FSM Research Themes



Intel/ERC Initiative Scope

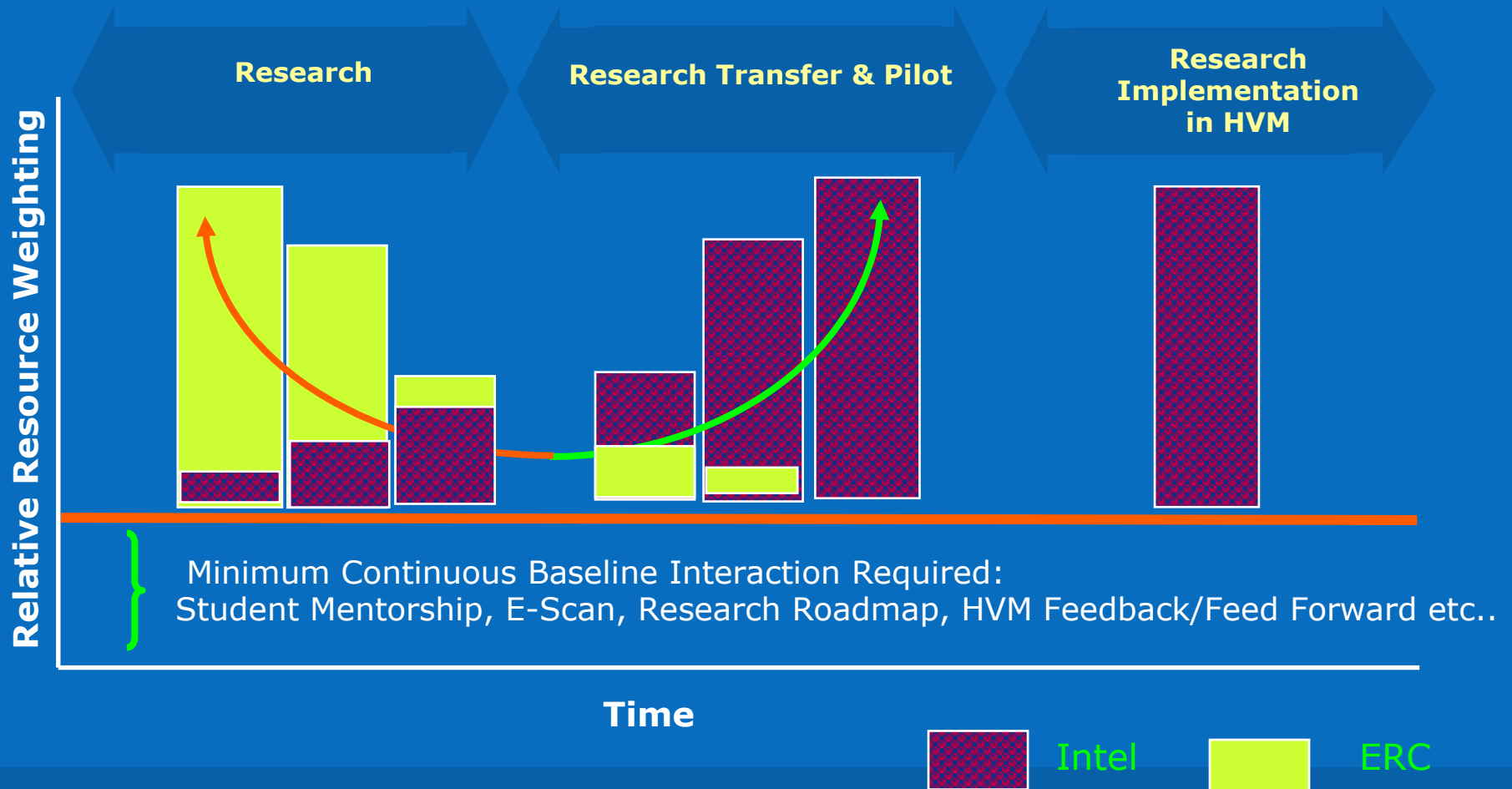


Research Proposal Ratification Process



Joint Intel/ERC Collaborative Research Model Resource Weighting

“Focusing on Research With HVM Implementation is Critical to Effectively Managing the Research/Student Supply Chain”



Conclusions

- Total Value of Intel/ERC Customized Research: ~\$1.3M
- Good collaboration between Intel and ERC PIs
- Research Program managed well by Intel/ERC Steering Committee
- Research Projects on track
- 'For FSM', 'By FSM', 'In FSM' Concept applied to harvesting research gaining traction

Thank You!

Retaining Ring Induced Frictional Pad Heating and its Effect on Pad Wear

PIs:

- Ara Philipossian, Chemical and Environmental Engineering, UA

Graduate Students:

- Zhenxing Han, Ph. D. student, Chemical and Environmental Engineering, UA
- Yubo Jiao, Ph. D. student, Chemical and Environmental Engineering, UA
- Xiaoyan Liao, Ph. D. student, Chemical and Environmental Engineering, UA

Other Researchers:

- Yasa Sampurno, Research Associate, Chemical and Environmental Engineering, UA
- Yun Zhuang, Research Associate, Chemical and Environmental Engineering, UA

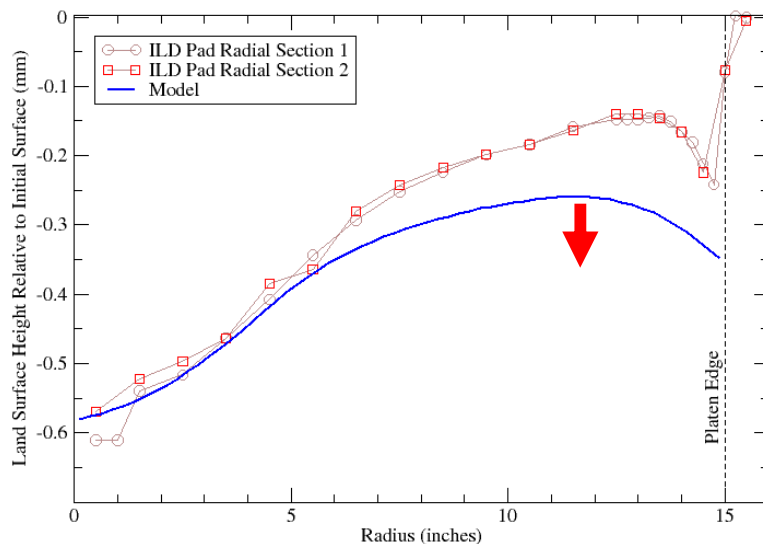
Retaining Ring Induced Frictional Pad Heating and its Effect on Pad Wear

Cost Share (other than core ERC funding):

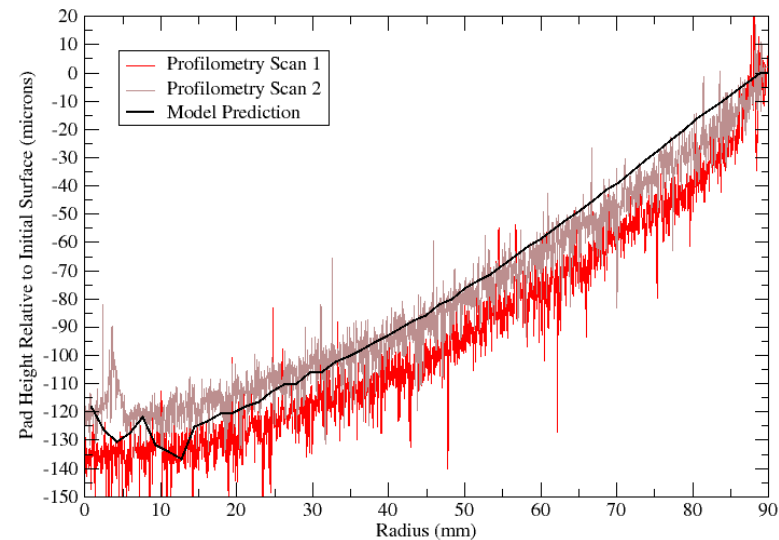
- In-kind donation (retaining rings) from Entegris
- In-kind donation (slurry) from Fujimi
- In-kind donation (conditioner disc) from Shinhan
- In-kind donation (conditioner disc) from 3M
- In-kind donation (polishing pad) from Dow
- In-kind donation (polishing pad) from Cabot Microelectronics
- In-kind donation (wafer) from Intel
- In-kind support from Araca

Project Essence

- Evidence suggests that retaining rings have a significant effect on macroscopic pad wear rates depending on polishing time.
- Pad cut rates in the presence of a ring are lower than expected from models that otherwise explain such data well.



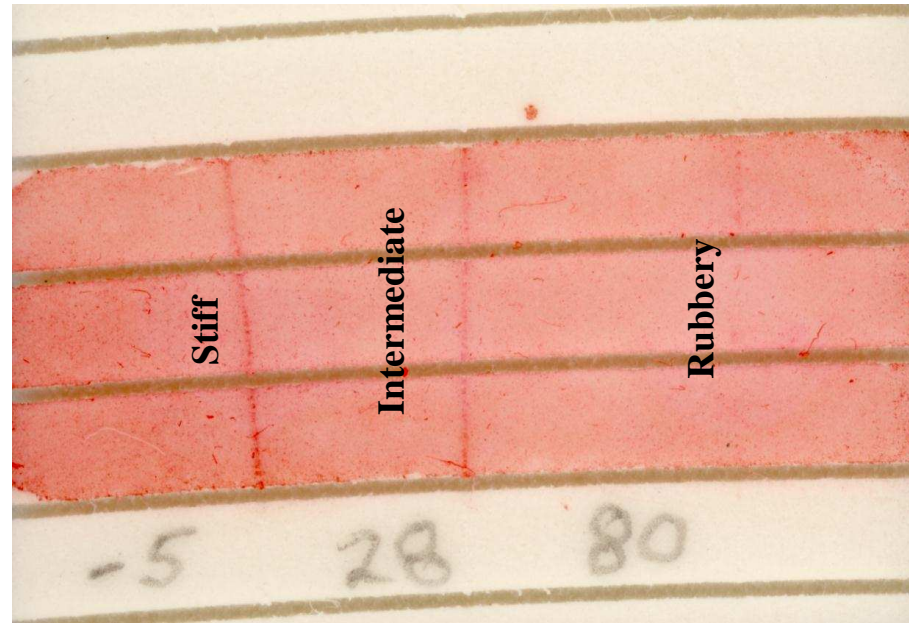
Model wear profile vs. data with a ring



Model wear profile vs. data without a ring

Project Essence

- Qualitative experiments suggest that pad cut rate reduction is thermal in origin and that it depends on how the material properties of the pad change with temperature.



Stylus scratches in IC pad at different temperatures at constant load.
Scratches **appear to heal behind the stylus at 80 C**, where the material is rubbery.

Specific Objectives and EHS Impact

- **Quantify the effect retaining ring materials, diamond disc conditioners, polishing pads and process conditions (i.e. pad temperature and polishing time) on pad cut rate and pad wear profile**
- **Determine whether or not temperature and/or time effects are the full explanation for pad wear profile differences in different processes**

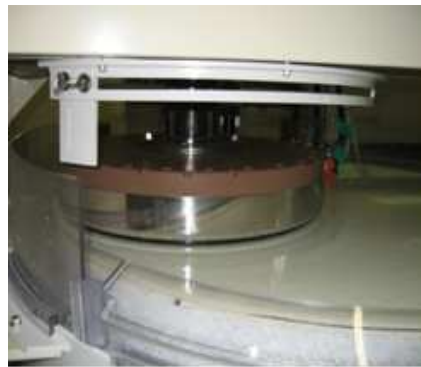
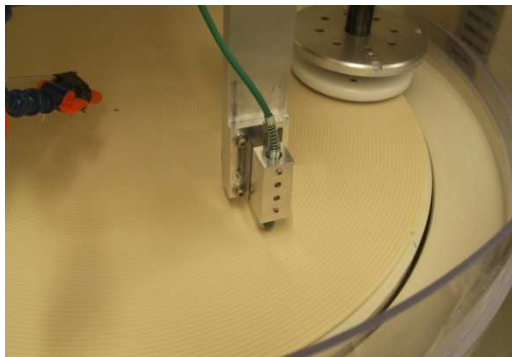
Reduce slurry, pad and energy consumption by 15 percent

Approach

Our approach for this 2 – year program (6/2008 to 5/2010) is as follows:

- **Modify and integrate a previously built in-situ offline pad cut rate measurement device for UA's 300 mm polisher**
- **Measure pad wear at three platen temperatures for two different pads, two retaining ring materials and two retaining ring design using a 300 mm polisher equipped with platen heating/cooling**
 - **CMC D100 and Dow IC1000 pads**
 - **PEEK1, PEEK2 and PPS2 retaining rings**
 - **Platen temperature maintained at 15, 25 and 50 °C (Note: pad temperature is in between platen and room temperature)**
- **Use micrometry to verify pad cut rate (PCR) measurements obtained from the in-situ device**

Equipment for 300-mm Polisher



Pad cut rate measurement device (top) ... Neslab heater & chiller (center) ... Araca APD – 800 platen heating and cooling interface hardware (bottom)

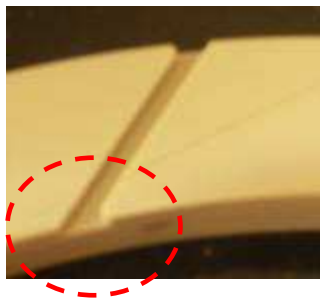
APD – 800 (300-mm polisher with automatic platen heating and cooling, 10-zone conditioner and in-situ frictional and pad temperature sensor)

Retaining Ring Materials and Slot Designs

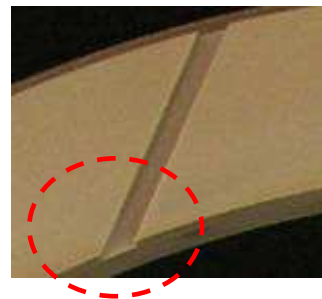
PEEK



Design 1



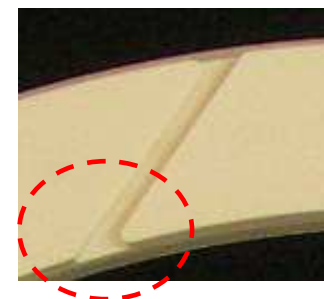
Design 2



PPS



Design 1



Retaining Ring Adaptor



Adaptor



Adaptor with the retaining ring installed



Overall setup on the APD – 800 carrier head

Retaining ring adaptor (courtesy of Araca and Entegris) has been successfully implemented. The adaptor enables the use of 300 mm industry standard retaining ring (i.e. for the AMAT Reflexion polisher) on APD – 800 300 mm polisher

Pad Cut Rate at Platen Temperature 25 °C

From Pad Cut Rate Measurement Device

Ring – Pad	Local Pad Cut Rate* (μ m/hr)				Average Local Pad Cut Rate (μ m/hr)
	0 – 2 hr	2 – 4 hr	4 – 6 hr	6 – 8 hr	
PPS1 – IC1000	14.7	12.1	10.0	-	12.2
PEEK1 – IC1000	36.6	19.4	20.5	-	25.5
PEEK2 – IC1000	N/A	N/A	19.9	25.6	22.8
PPS1 – D100	17.5	12.1	4.7	-	11.4
PEEK1 – D100	17.0	16.6	7.1	-	13.5
PEEK2 – D100	23.9	13.7	10.3	-	16.0

* Measured by the pad cut rate measurement device at R = 12.25” every 2 hours.

SRC/SEMATECH Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Pad Cut Rate at Platen Temperature 50 °C

From Pad Cut Rate Measurement Device

Ring – Pad	Local Pad Cut Rate* (μm/hr)			Average Local Pad Cut Rate (μm/hr)
	0 – 2 hr	2 – 4 hr	4 – 6 hr	
PEEK1 – IC1000	33.7	13.0	4.4	17.0
PPS1 – IC1000	32.9	27.9	21.3	27.4
PPS1 – IC1000 (Repeat Run)	22.3	27.9	10.8**	21.8
PEEK1 – D100	5.3	7.9	6.4	6.5
PPS1 – D100	< 4.0***	< 4.0***	< 4.0***	< 4.0

* Measured by the pad cut rate measurement device at R = 12.25” every 2 hours.

** Measured between 4 and 5.2 hours.

*** Within experimental error.

Pad Cut Rate Comparison

Platen Temperature at 25 and 50 °C

Ring – Pad	Local pad cut rate from pad cut rate measurement device (μm/hr)		Local pad cut rate from micrometry (μm/hr)		Global pad cut rate from micrometry (μm/hr)	
	25 C	50 C	25 C	50 C	25 C	50 C
PEEK1 – IC1000	25.5	17.0	29.6	23.8	27.5	20.3
PPS1 – IC1000	12.2	27.4	17.5	33.2	16.5	29.4
PPS1 – IC1000 (Repeat Run)	12.2	21.8	17.5	27.4	16.5	23.5
PEEK1 – D100	13.5	6.5	17.6	7.0	16.4	6.1
PPS1 – D100	11.4	< 4.0*	8.8	5.3	7.8	5.1

* Within experimental error.

The pad surface contained moisture when the pad cut rate device was used to measure local pad thickness during the wear tests. The pad was dry when a micrometer was used to measure local pad thickness after the wear tests. This contributed to different local pad cut rate measurements obtained by the pad cut rate measurement device and micrometer.

Summary of Year – 1 Results

Platen Temperature 25 °C

- For both IC1000 and D100, global PCRs of the PEEK rings were **significantly higher** (by appx. 62 – 110 percent) than PPS
- For both IC1000 and D100, global PCRs of the PEEK rings were **similar**, indicating that the slot curvature did not impact PCR
- For both PEEK1 and PPS1, global PCR for IC1000 was **significantly higher** (by appx. 68 – 112 percent) than D100 (**we would expect a much longer pad life when using D100**)
- PCR changed as a function of polishing time (possibly due to changes in pad properties in the ‘z’ direction)

Summary of Year – 1 Results

Platen Temperature 50 °C

- For IC1000, global PCR of PEEK1 was **lower** than that of PPS1 by 30 percent
- For D100, the trend was opposite: The global PCR of PEEK1 was **higher** than that of PPS1 by 16 percent (we don't yet understand the reason for this)
- For both PEEK1 and PPS1, global PCR for IC1000 was **significantly higher** than D100 (by appx. 233 to 476 percent) – **This indicates that the D100 will have a much longer pad life**
- PCR changed as a function of polishing time (possibly due to changes in pad properties in the 'z' direction)

Industrial Interactions and Technology Transfer

Industrial mentors and contacts:

- **Don Hooper (Intel)**
- **Mansour Moinpour (Intel)**
- **Jason Zanotti (Intel)**
- **Cliff Spiro (Cabot Microelectronics)**

Future Plans

Next Year Plan

- Complete PCR tests on Dow IC1000 and CMC D100 pads using PPS1 and PEEK1 retaining rings at a platen temperature of 15 °C

Long-Term Plan

- Develop new (or augment existing) theories to explain the results

Investigation of the Relationship between Planarization and Pad Surface Micro- Topography

PIs:

- Ara Philipossian, Chemical and Environmental Engineering, UA

Graduate Students:

- Yubo Jiao, Ph. D. candidate, Chemical and Environmental Engineering, UA
- Zhenxing Han, Ph. D. student, Chemical and Environmental Engineering, UA
- Anand Meled, Ph. D. student, Chemical and Environmental Engineering, UA
- Xiaoyan Liao, Ph. D. student, Chemical and Environmental Engineering, UA

Other Researchers:

- Yasa Sampurno, Research Associate, Chemical and Environmental Engineering, UA
- Yun Zhuang, Research Associate, Chemical and Environmental Engineering, UA

Investigation of the Relationship between Planarization and Pad Surface Micro-Topography

Cost Share (other than core ERC funding):

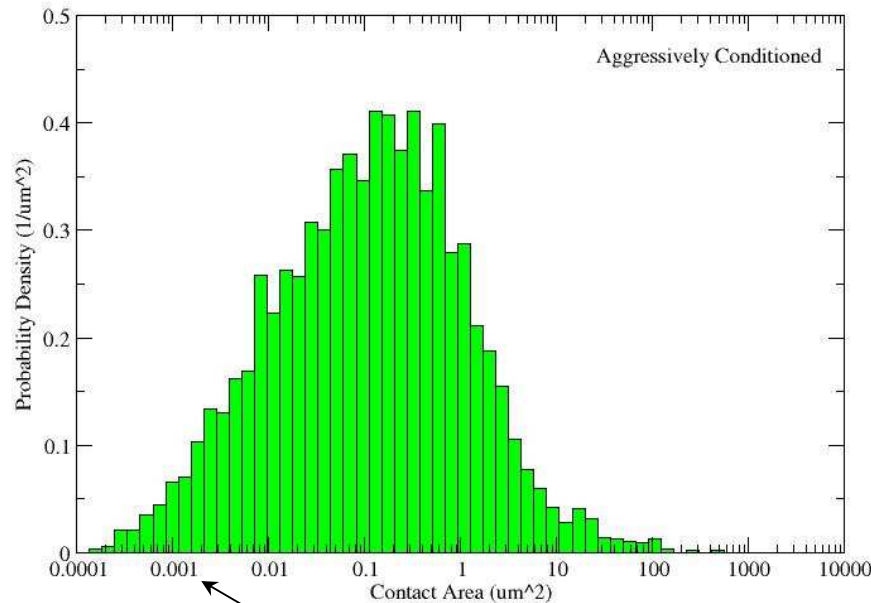
- **In-kind donation (conditioner disc) from Ehwa**
- **In-kind donation (conditioner disc) from Shinhan**
- **In-kind donation (conditioner disc) from 3M**
- **In-kind donation (polishing pad) from Dow**
- **In-kind donation (polishing pad and slurry) from Cabot Microelectronics**
- **In-kind donation (wafer) from Intel**
- **In-kind support from Araca**

Project Essence

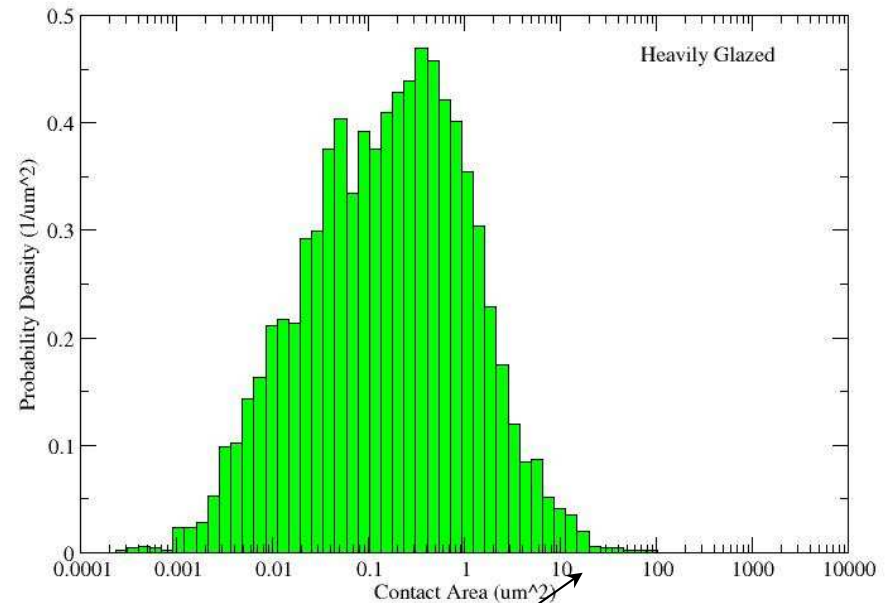
- **Planarization is a common thread across all CMP platforms.**
- **Planarization behavior depends on the slurry formulation and on the topography of the pad.**
- **A new method is available for investigating pad/wafer contact, creating an opportunity for deeper understanding and control of the factors that affect planarization.**
- **The new method, laser confocal contact area microscopy, shows directly which features of the pad topography are capable of reaching into a feature of a given size and reducing planarization efficiency.**
- **Microscopy data can be supplemented by rough surface contact removal rate modeling to confirm theories suggested by observations.**

Project Essence

Contact area histograms may be intimately related to polishing behavior



The aggressively conditioned pad has more contacts in the left-hand tail from tall “thin” asperities (i.e. 10 – 30 nm wide features). These presumably can reach into small features and reduce planarization efficiency. The heavily glazed pad by contrast has relatively few such contact areas.

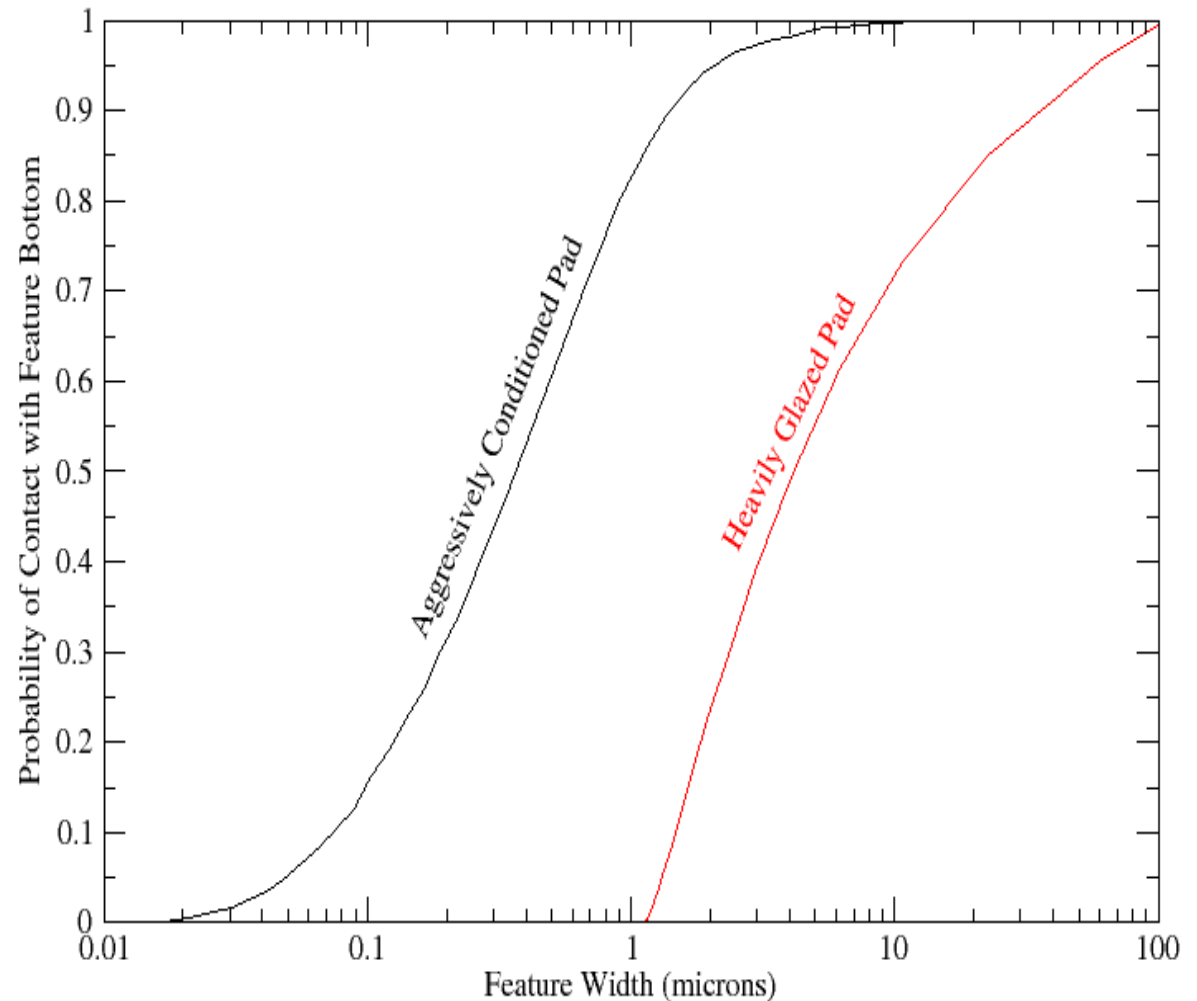
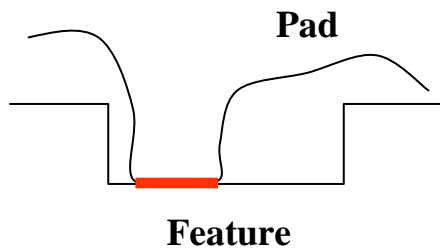


The heavily glazed pad also has a deficit of large area contacts from tall “fat” asperities (i.e. 3 – 10 micron wide) relative to the aggressively conditioned pad. This may be responsible for a lower removal rate.

Project Essence

CONNECTION WITH PLANARIZATION

In this graph, the histograms from the previous slide have been processed to estimate the probability that a randomly chosen contacting asperity will be capable of contacting the bottom of a feature of a given width. While illustrated here for trenches, similar estimates can be made for other kinds of features.



Objectives and EHS Impact

- **Gain a deeper understanding and control of factors related to pad topography that affect planarization**
- **Prove that contact area can predict planarization behavior**

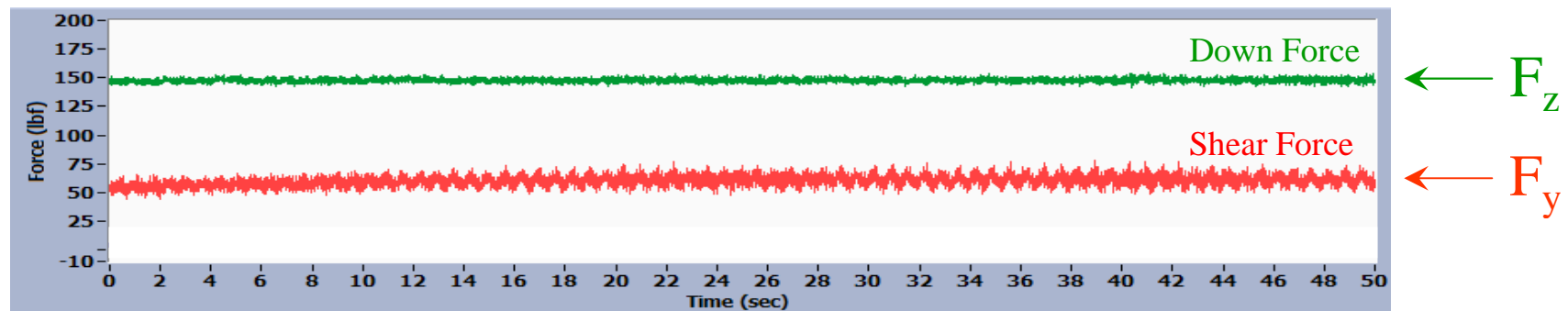
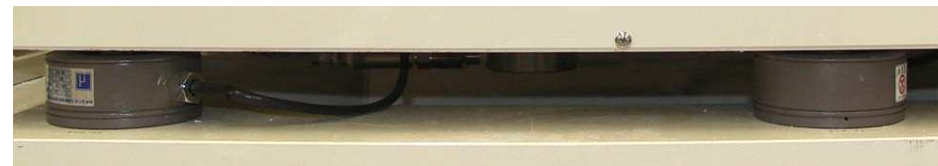
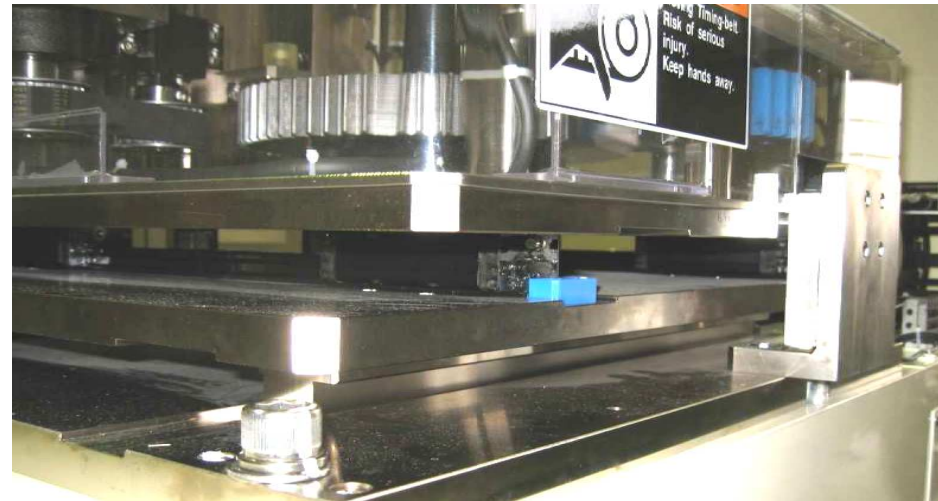
Reduce slurry, pad and energy consumption by 30 percent

Approach

Our approach for this 3 – year program (6/2008 to 5/2011) is as follows:

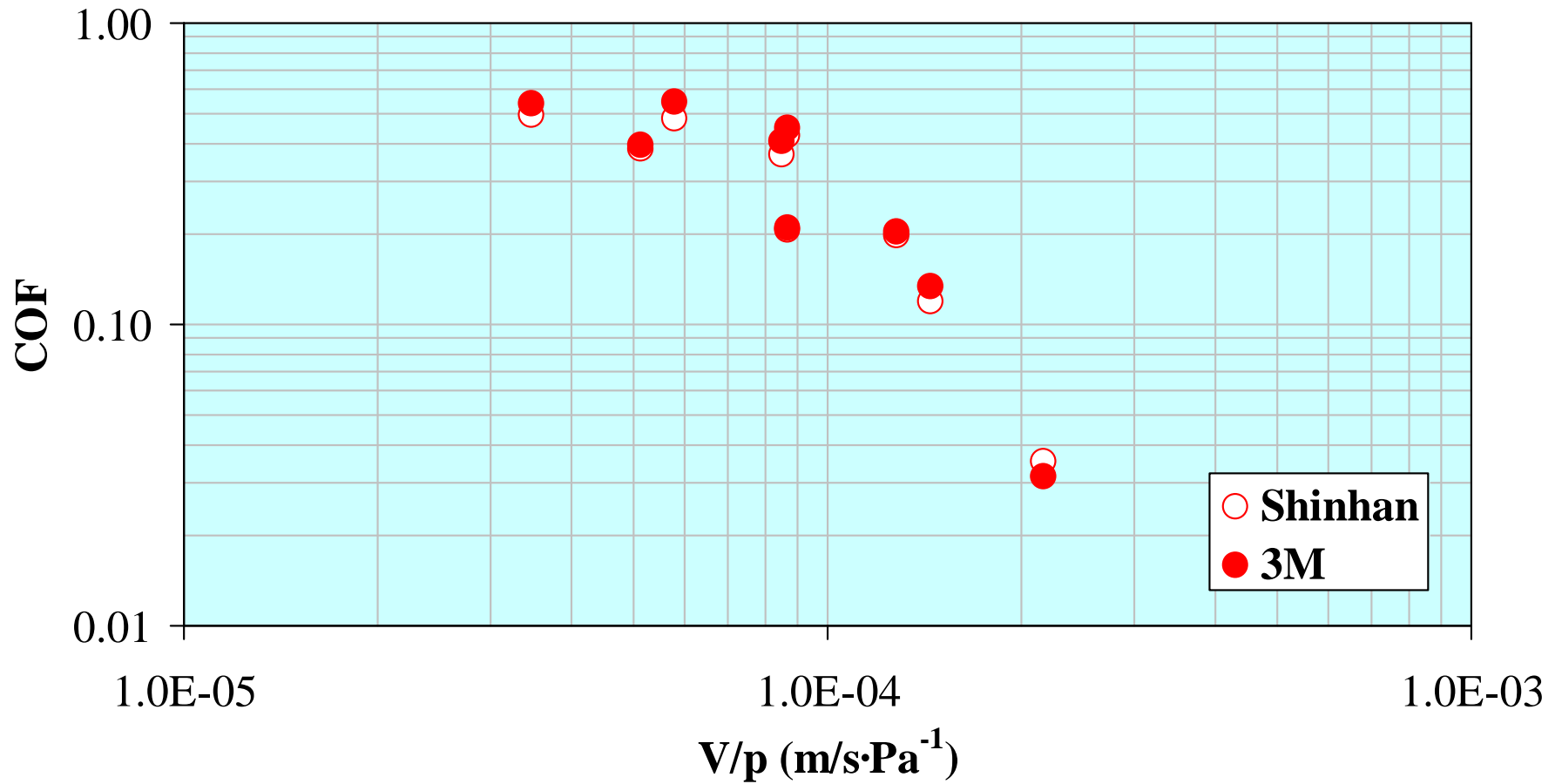
- **Polish 300-mm wafers using a variety of conditions and consumables (i.e. pads with different hardness and porosity and diamonds with different levels of aggressiveness) known or expected to improve or degrade planarization efficiency**
- **Examine pad samples under static loading (at CMP-relevant pressures) using flat, and possibly patterned, sapphire windows (patterned windows would replicate the contact conditions on patterned wafers and would make it feasible to actually see which pad features affect planarization)**
- **Correlate planarization behavior with contact area characteristics**
- **Tie observational data together with rough surface contact and removal rate models**

APD – 800 Polisher and Tribometer



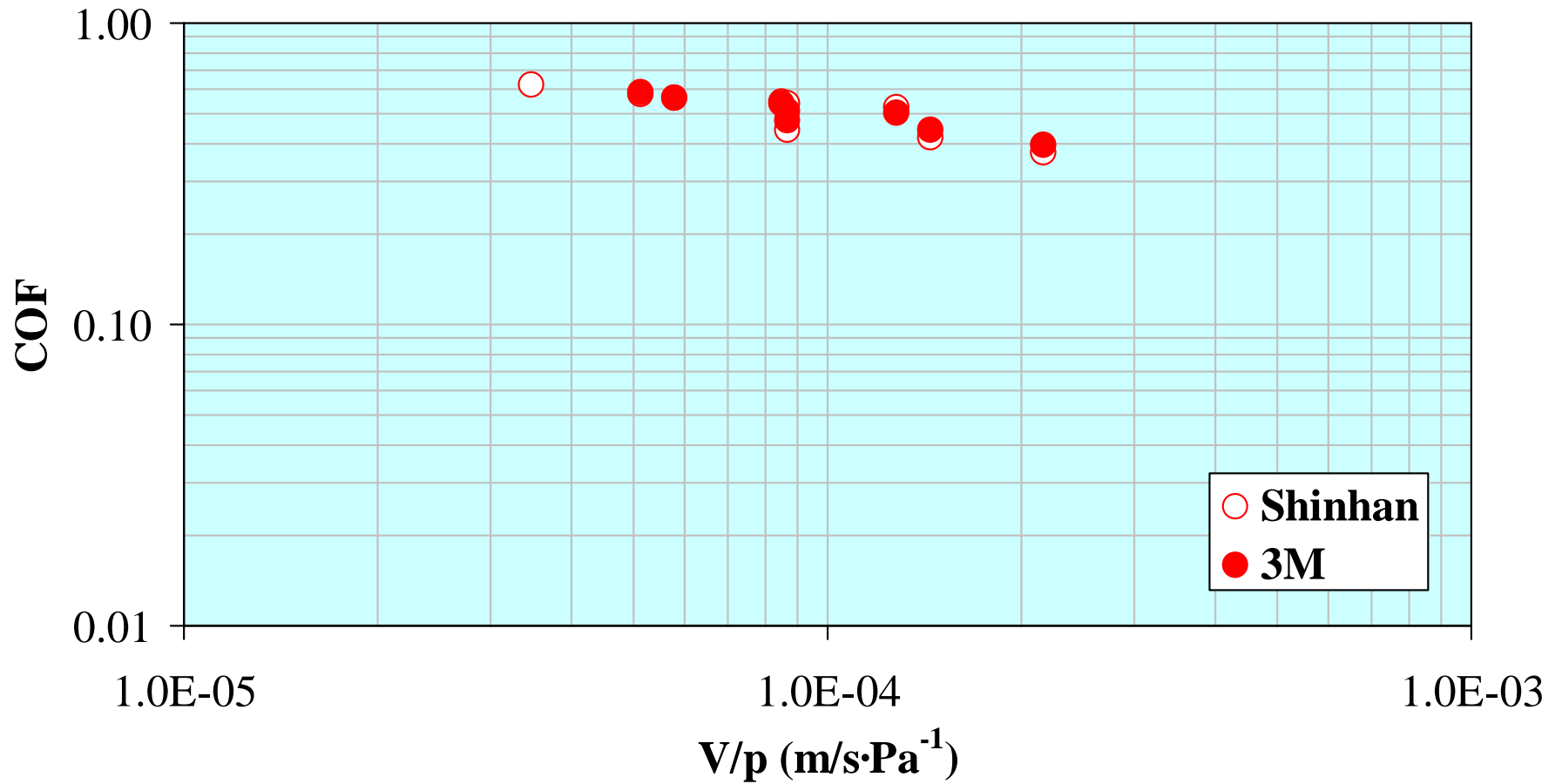
COF vs. V/p

IC1000



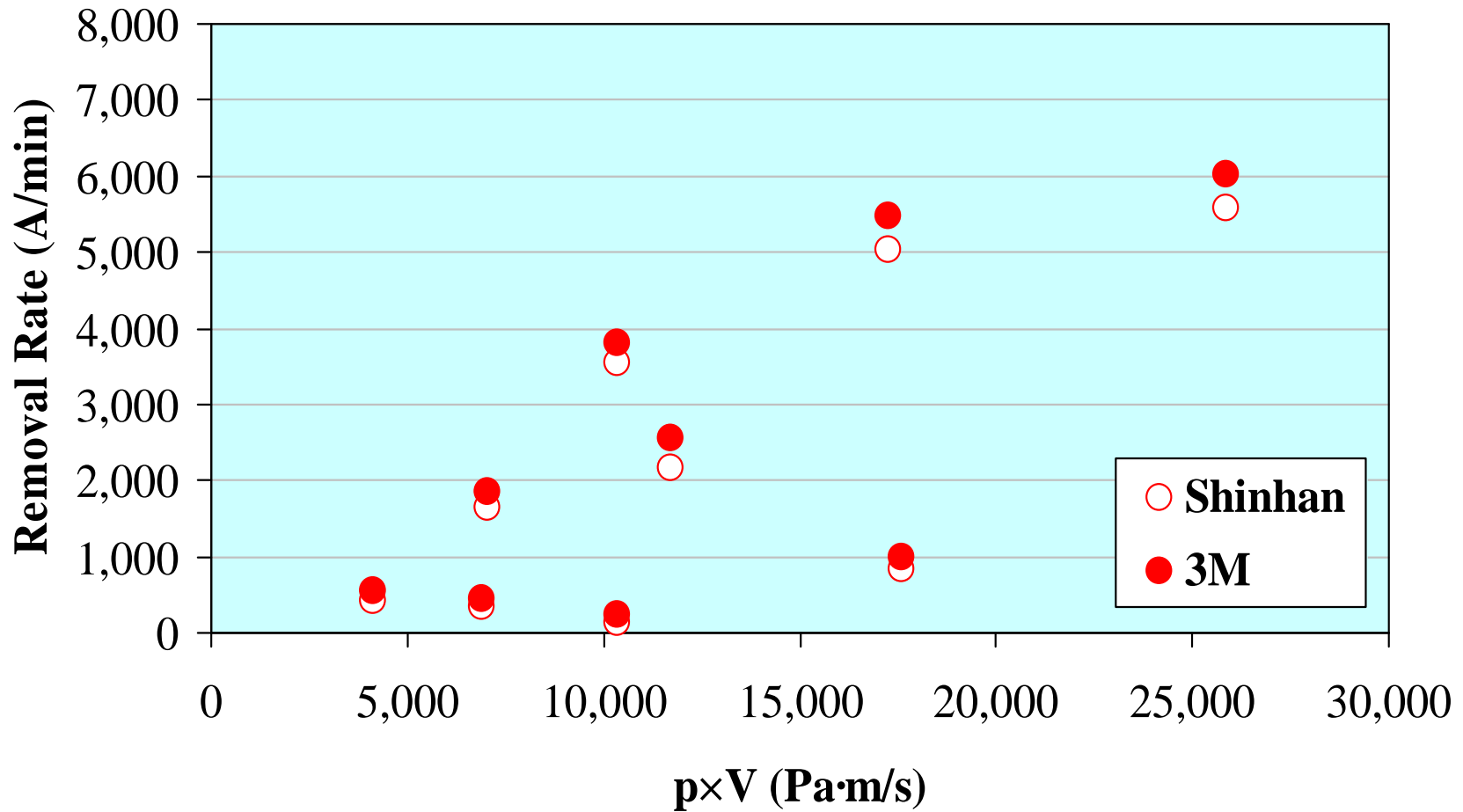
COF vs. V/p

D100



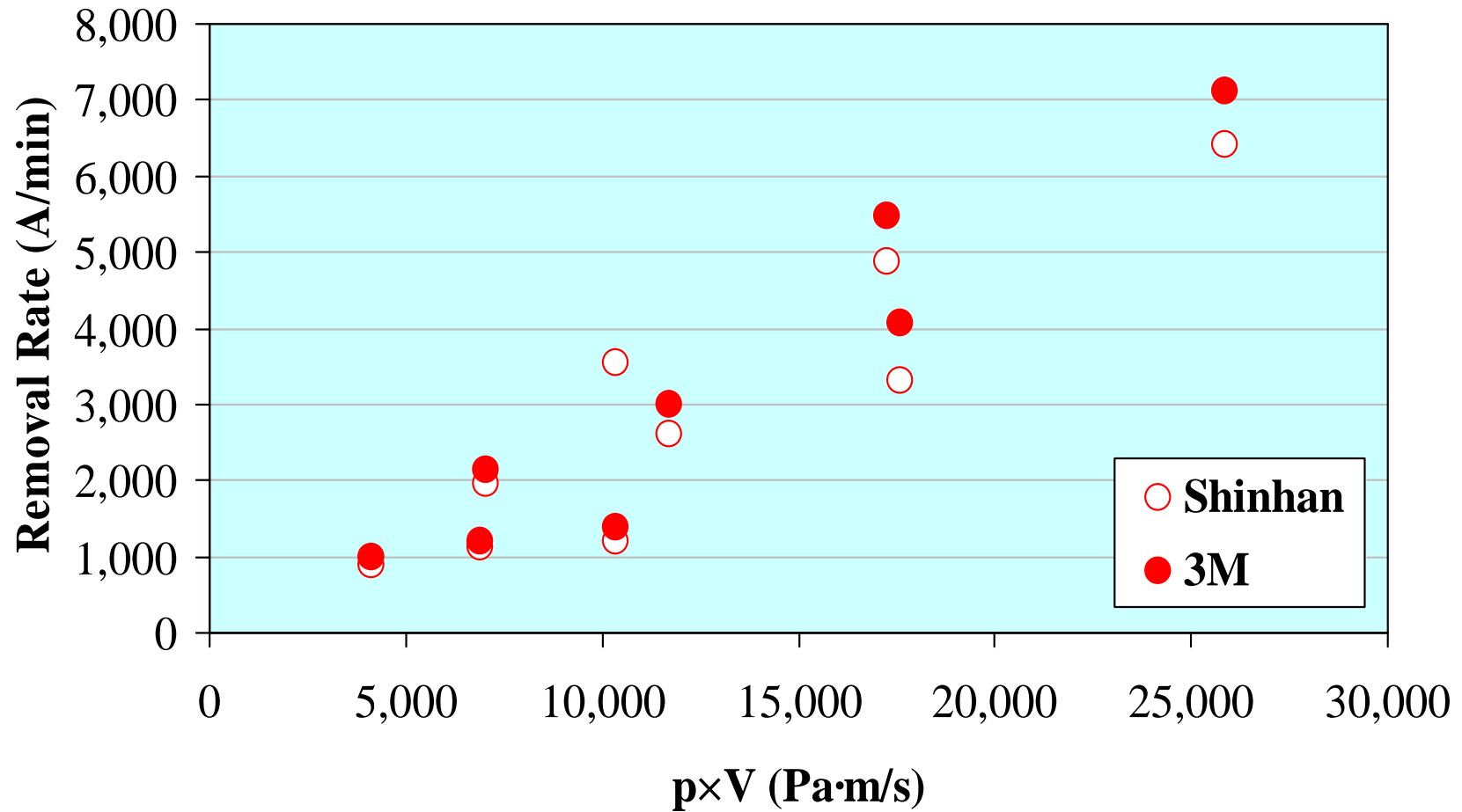
Removal Rate vs. $p \times V$

IC1000



Removal Rate vs. $p \times V$

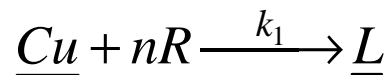
D100



2-Step Removal Rate Model

- Modified Langmuir-Hinshelwood model:**

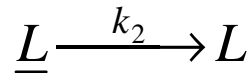
- n moles of reactant R in the slurry react at rate k_1 with copper film on the wafer to form a product layer L on the surface



$$k_1 = A \times \exp(-E_a / kT)$$

$$T = T_p + \frac{\beta}{V^{0.5+e}} \times COF \times p \times V$$

- Product layer \underline{L} is subsequently removed by mechanical abrasion with rate k_2



$$k_2 = C_p \times COF \times p \times V$$

- Abraded material L is carried away by the slurry

- The removal rate in this sequential mechanism therefore is a function of both thermal and mechanical attributes of the process**

$$R R = \frac{M_w}{\rho} \frac{k_1 k_2}{k_1 + k_2}$$

Fitting Parameters

E_a	Activation Energy (eV)
A	Pre-exponential factor of chemical rate constant (mole \times m⁻² s⁻¹)
C_p	Proportionality constant of mechanical rate constant (mole/J)
e	Exponential factor of sliding velocity derived from pad heat partition fraction
β	A constant that depend on wafer size, tool geometry and properties of pad surface and bulk material (10⁻³ K/Pa(m/s)^{0.5-e})

Summary of Optimized Parameters

Based on Two Level Optimization Process

Fitting Parameters	IC1000 Shinhan	IC1000 3M	D100 Shinhan	D100 3M
E_a (eV)	1.267	1.267	1.267	1.267
A (10^{17} mole \times m ⁻² s ⁻¹)	7.734	7.734	7.734	7.734
C_p (10^{-7} mole/J)	1.640	1.536	1.550	1.667
e	1.562	1.901	1.006	0.740
β (10^{-3} K/Pa(m/s) ^{0.5-e})	1.967	2.133	1.143	1.176
RMS (A/min)	145	176	84	74

Summary of k_1/k_2

Pressure (PSI)	Velocity (m/s)	k_1/k_2			
		IC1000 Shinhan	IC1000 3M	D100 Shinhan	D100 3M
1.0	0.6	3.6	5.2	2.2	2.1
1.7	0.6	8.0	17.6	3.0	2.8
2.5	0.6	41.3	206.1	5.7	NA
1.0	1.0	2.3	2.5	1.3	1.5
1.7	1.0	2.4	3.4	1.7	2.1
2.5	1.0	7.0	14.5	2.9	3.7
1.0	1.5	3.6	4.7	1.0	1.1
1.7	1.5	1.1	1.2	1.3	1.8
2.5	1.5	2.7	3.6	2.3	3.3

Summary of Year – 1 Results

- **RR results for all combinations of 2 pads and 2 discs showed gross non-Prestonian behavior**
- **Compared to D100, the IC1000 pad exhibited a much faster transition from boundary lubrication to partial lubrication mode**
- **The 2-step Langmuir-Hinshelwood model successfully simulated the non-Prestonian RR results thus indicating that the observed scatter could be fully explained theoretically**
- **Wilcoxon Signed Rank test of simulation results showed that:**
 - **D100 exhibited a more chemically-controlled process (CCP) than IC1000**
 - **Shinhan exhibited a more CCP than 3M**

Industrial Interactions and Technology Transfer

Industrial mentors and contacts:

- **Don Hooper (Intel)**
- **Mansour Moinpour (Intel)**
- **Cliff Spiro (Cabot Microelectronics)**
- **Peter Ojerholm (Ehwa)**

Future Plans

Next Year Plans

- Repeat all of the tests (i.e. effect of pad and process conditions) and simulations performed in Year – 1 with 2 different Ehwa disc (**already completed**)
- Identify **consumables and optimum polishing conditions** and run 300 mm patterned wafers for pad analysis
- Examine pad samples under static loading using a flat sapphire window to determine wafer-level topography, pad-wafer contact and near-contact area, pad asperity shape and density and number of large pad debris

Future Plans

Long-Term Plans

- **Examine limited number of pad samples under static loading using a patterned sapphire window to better mimic the contact phenomena that the pad actually sees during patterned wafer polishing**
- **Determine contact area, pad asperity shape and density and number of large pad debris**
- **Correlate planarization behavior with contact area data**
- **Tie observational data together with rough surface contact and RR models**
- **Propose methodology for using confocal microscopy as part of a screening process or diagnostic technique for new consumables and processes**

Contamination Control in Gas Distribution Systems

Customized Project; Sponsored by Intel

PI:

- **Farhang Shadman, Ph.D., Professor of Chemical and Environmental Engineering, UA**

Co-PI:

- **Carl Geisert, Sr. Principal Engineer, Intel**

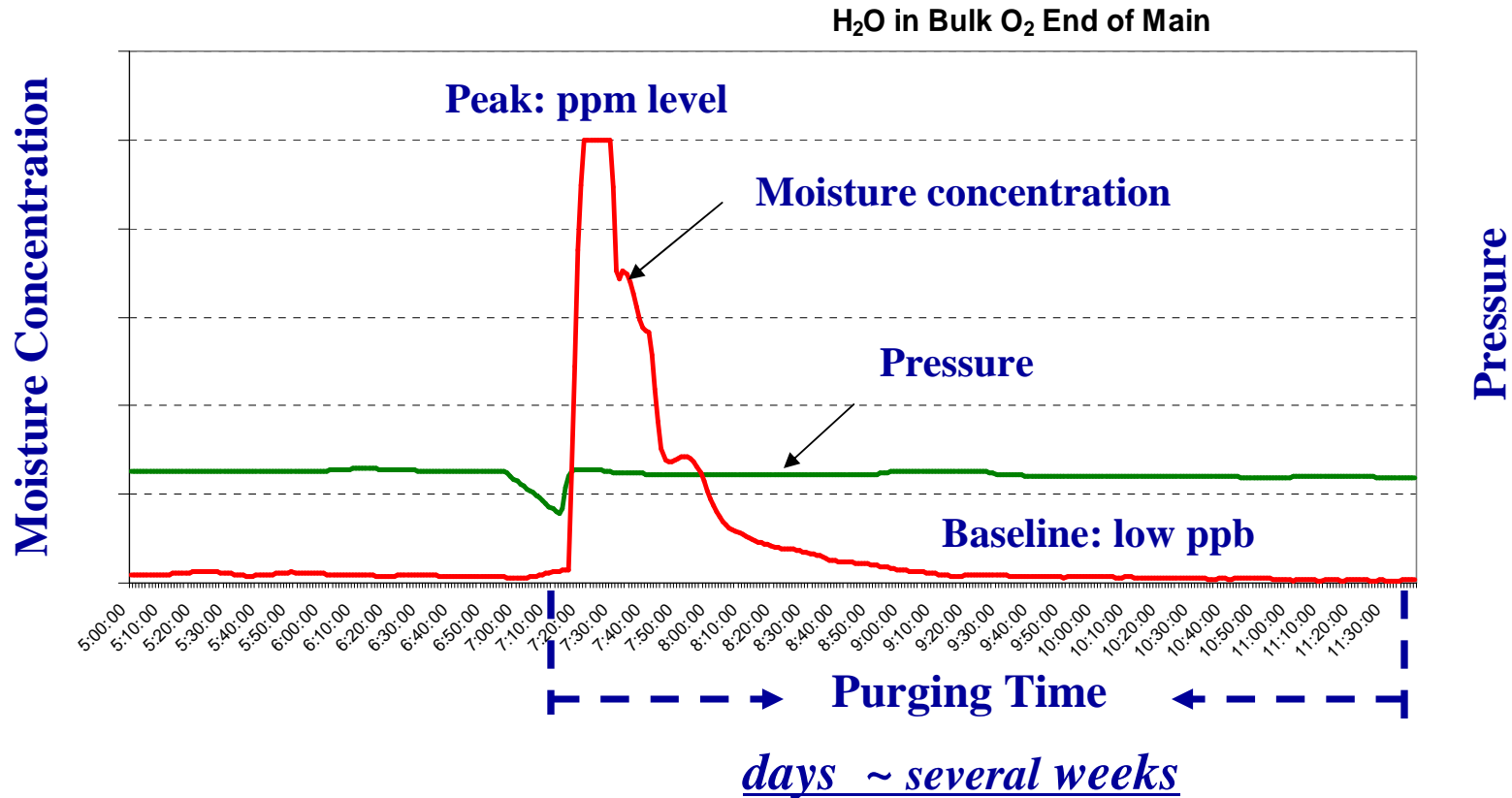
Research Engineer:

- **Junpin Yao: Ph.D., Postdoctoral, Chemical and Environ Eng, UA**

Graduate Students:

- **Hao Wang: Ph.D. student, Chemical and Environmental Engineering, UA**
- **Roy Dittler: Ph.D. student, Chemical and Environmental Engineering, UA**

Moisture Contamination & Dry-down



Fab closure due to contamination of the gas distribution system could result in revenue loss between \$5M and \$15 M/day

** Source: Intel*

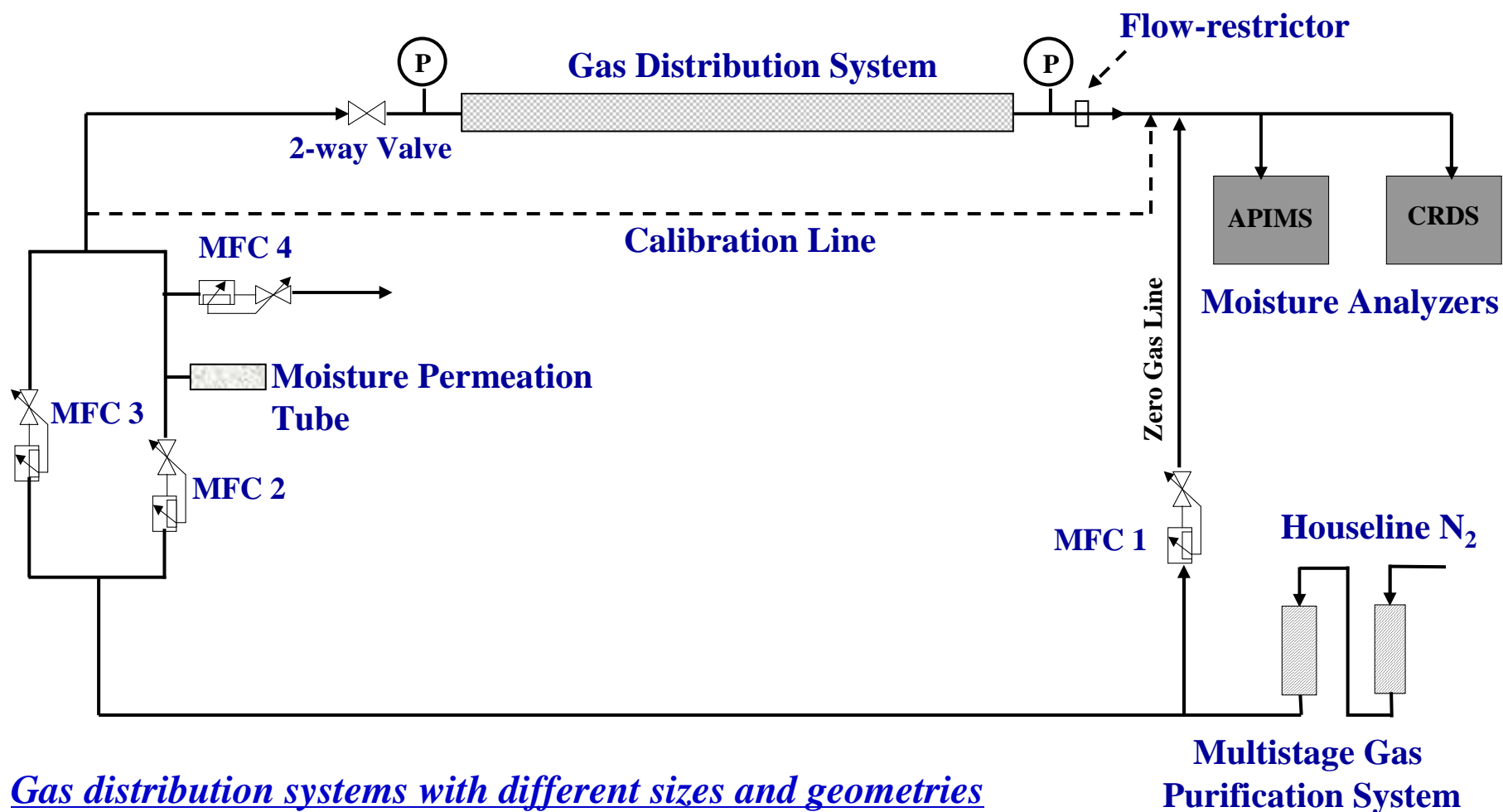
Objectives

- **Develop tools and techniques for analysis of contamination distribution and removal in ultra-pure gas distribution systems.**
- **Develop and validate a user-friendly process simulator suitable for field application to minimize purge time and gas usage during system start up, system recovery, or during the operation of gas distribution systems.**

ESH Impact

- **Significant reduction in the usage of resources (ultra-pure gas and energy) as well as reduction of dry-down time.**

Experimental Set-up and Testbed

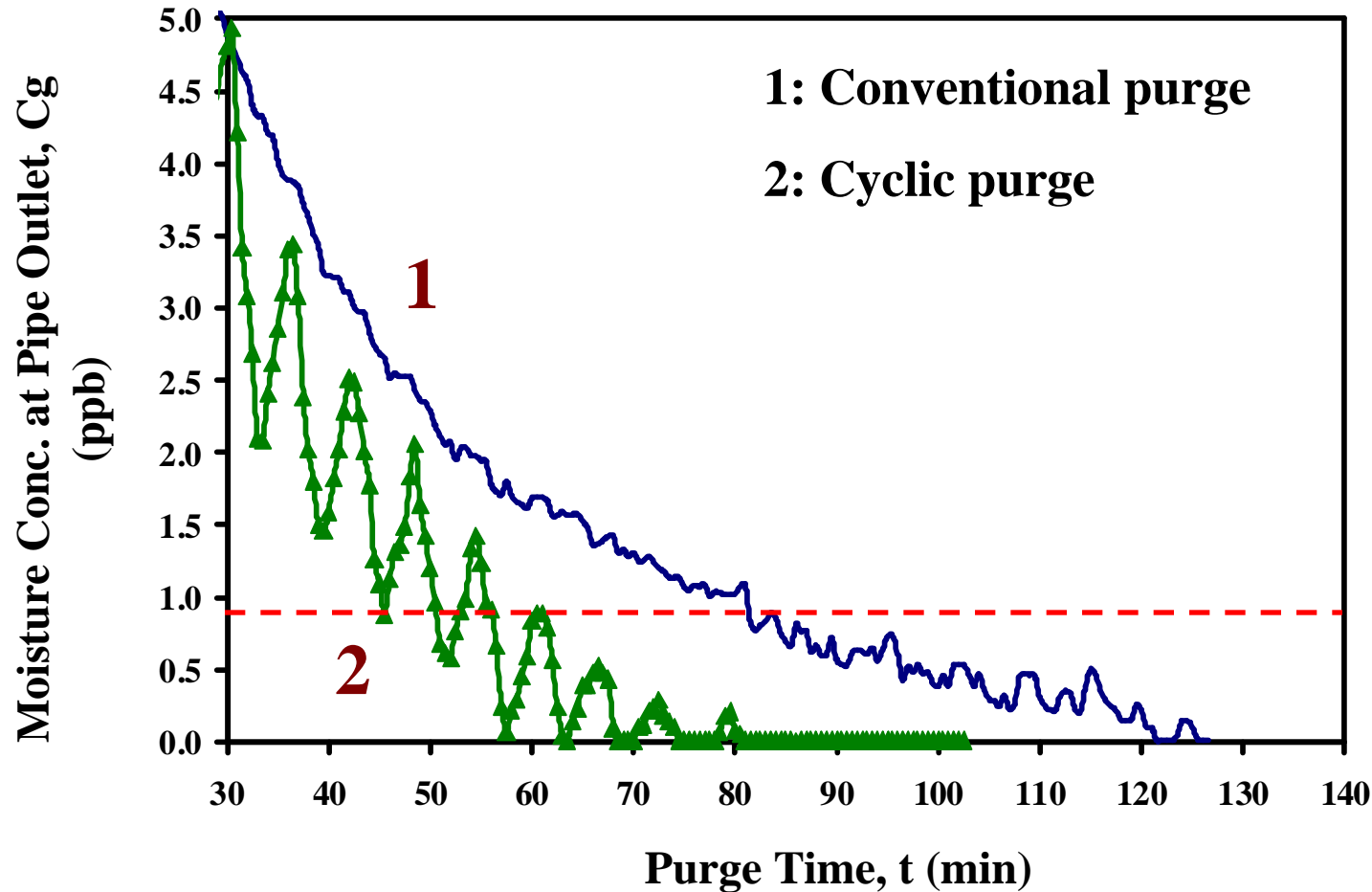


Gas distribution systems with different sizes and geometries were fabricated and provided by Intel; CRDS (Tiger Optics): middle ppb – low ppm; APIMS: sub ppb – low ppb

Cyclic Purge vs. Conventional Purge

Lab-scale system

EP SS pipe with 1.5 inch OD and 76 inch length. Initial conc. 90 ppb



To reach 1 ppb baseline: conventional purge: 80 minutes; Cyclic purge: 45 minutes ; cyclic purge saves 40% of purge gas.

Purge Process Simulator

System Pressure:

$$\frac{\partial P}{\partial t} = -P \frac{\partial u}{\partial x} - u \frac{\partial P}{\partial x}$$

Velocity:

$$\frac{\partial u}{\partial t} = -\frac{RT}{PM} \frac{\partial P}{\partial x} - u \frac{\partial u}{\partial x}$$

Absorbed Moisture:

$$\frac{\partial C_s}{\partial t} = k_a C_g (S_0 - C_s) - k_d C_s$$

Gas Phase Moisture:

$$\frac{\partial C_g}{\partial t} = D_L \frac{\partial^2 C_g}{\partial x^2} + \frac{\partial D_L}{\partial x} \frac{\partial C_g}{\partial x} - u \frac{\partial C_g}{\partial x} - C_g \frac{\partial u}{\partial x} + \frac{4}{d} [k_d C_s - k_a C_g (S_0 - C_s)]$$

C_s : moisture concentration on pipe wall, mol/cm²; C_g : moisture concentration in gas, mol/cm³;

k_{ads} : adsorption rate constant, cm³/mol/s; k_{des} : desorption rate constant, 1/s

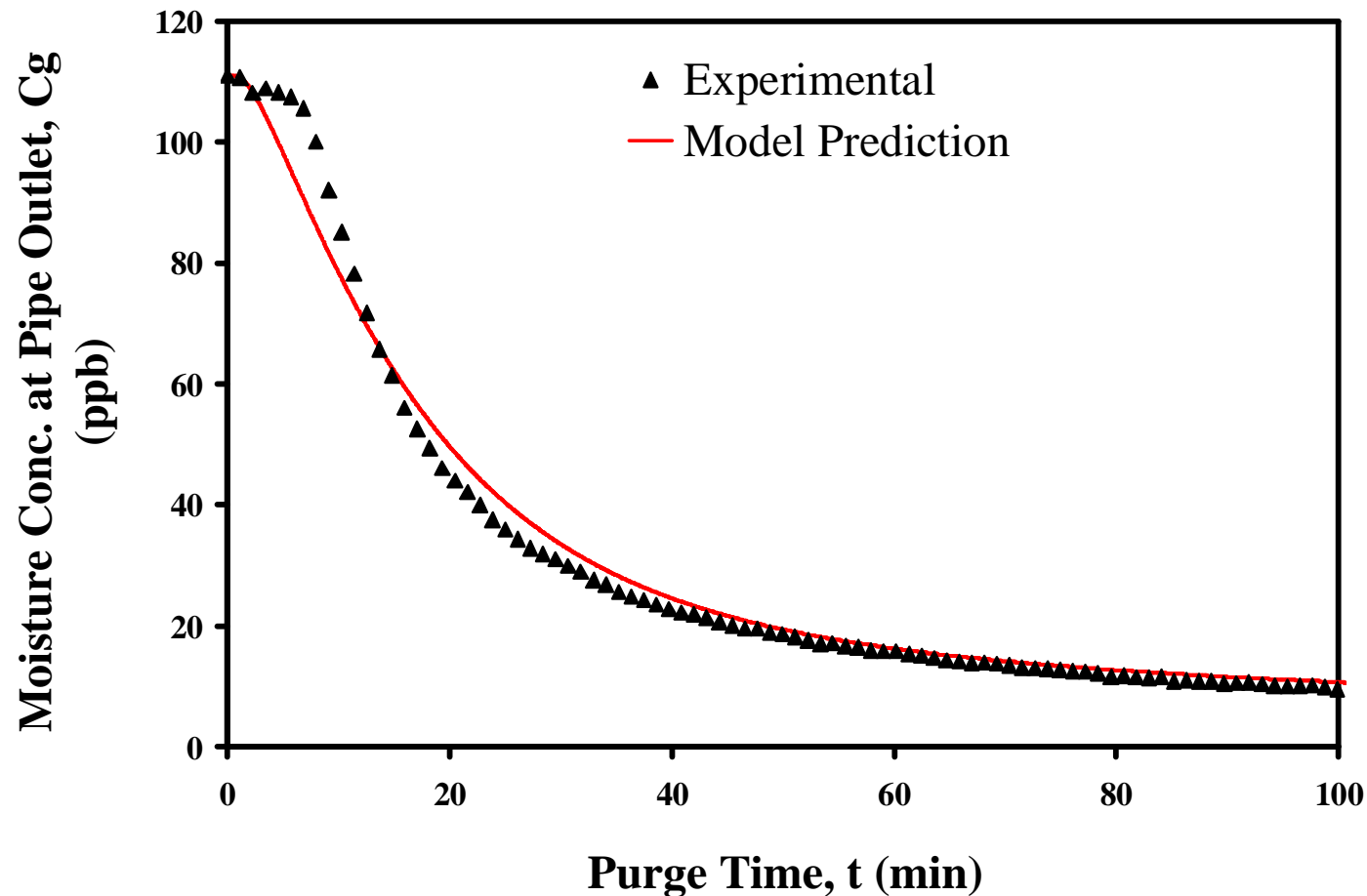
S_0 : site density of surface adsorption, mol/cm²; D_L : dispersion coefficient, cm²/s;

u : velocity, cm/s; d : diameter; P : pressure

The simulator is scalable and applicable to various size systems

Simulator Verification- Predicting Conventional Purge

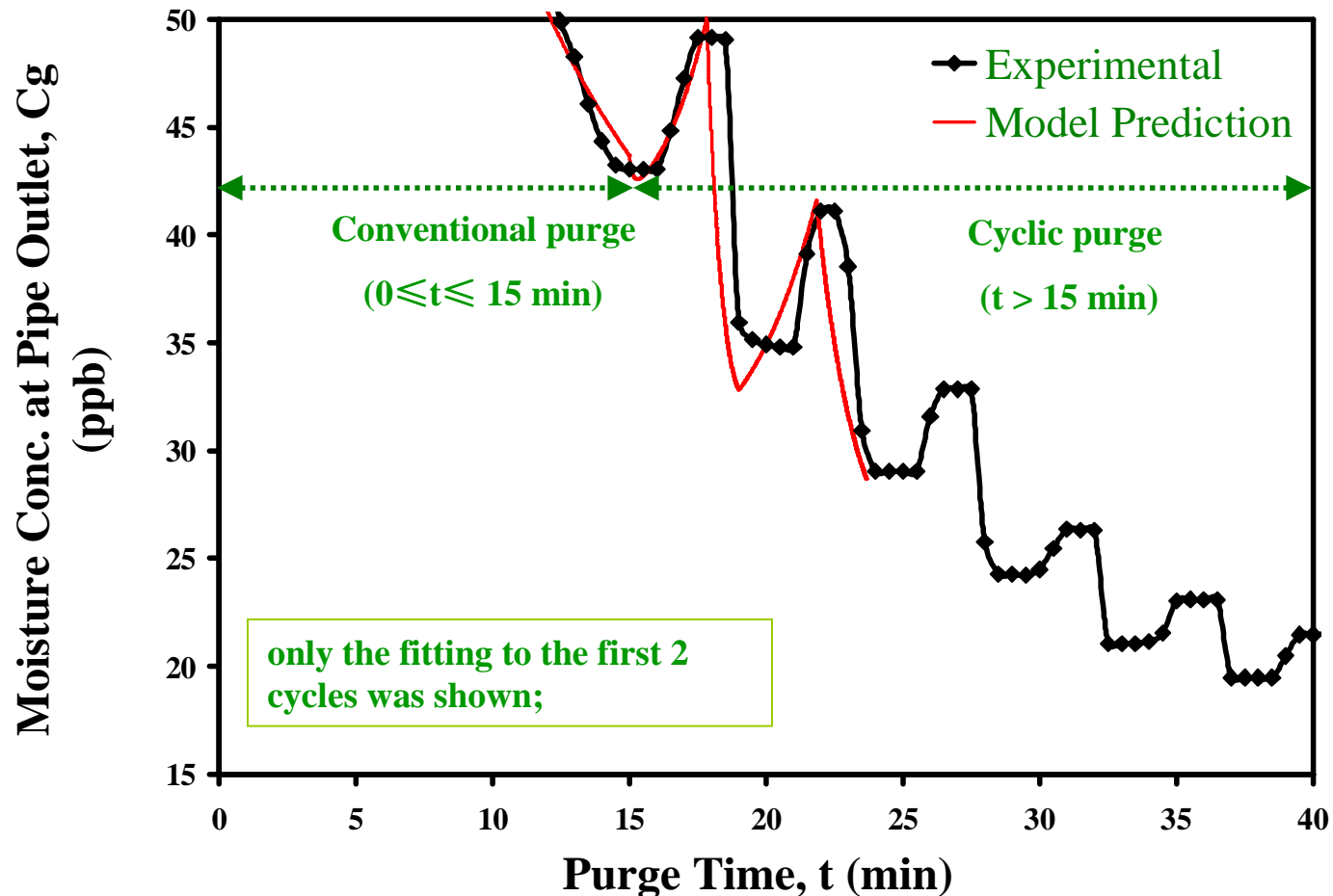
EP SS pipe with 1.5 inch OD and 76 inch length. Initial conc. 115 ppb



The process simulator well predicts conventional purge process

Simulator Verification- Predicting Conventional and Pressure-cyclic Purge

EP SS pipe with 1.5 inch OD and 76 inch length. Initial conc. 350 ppb

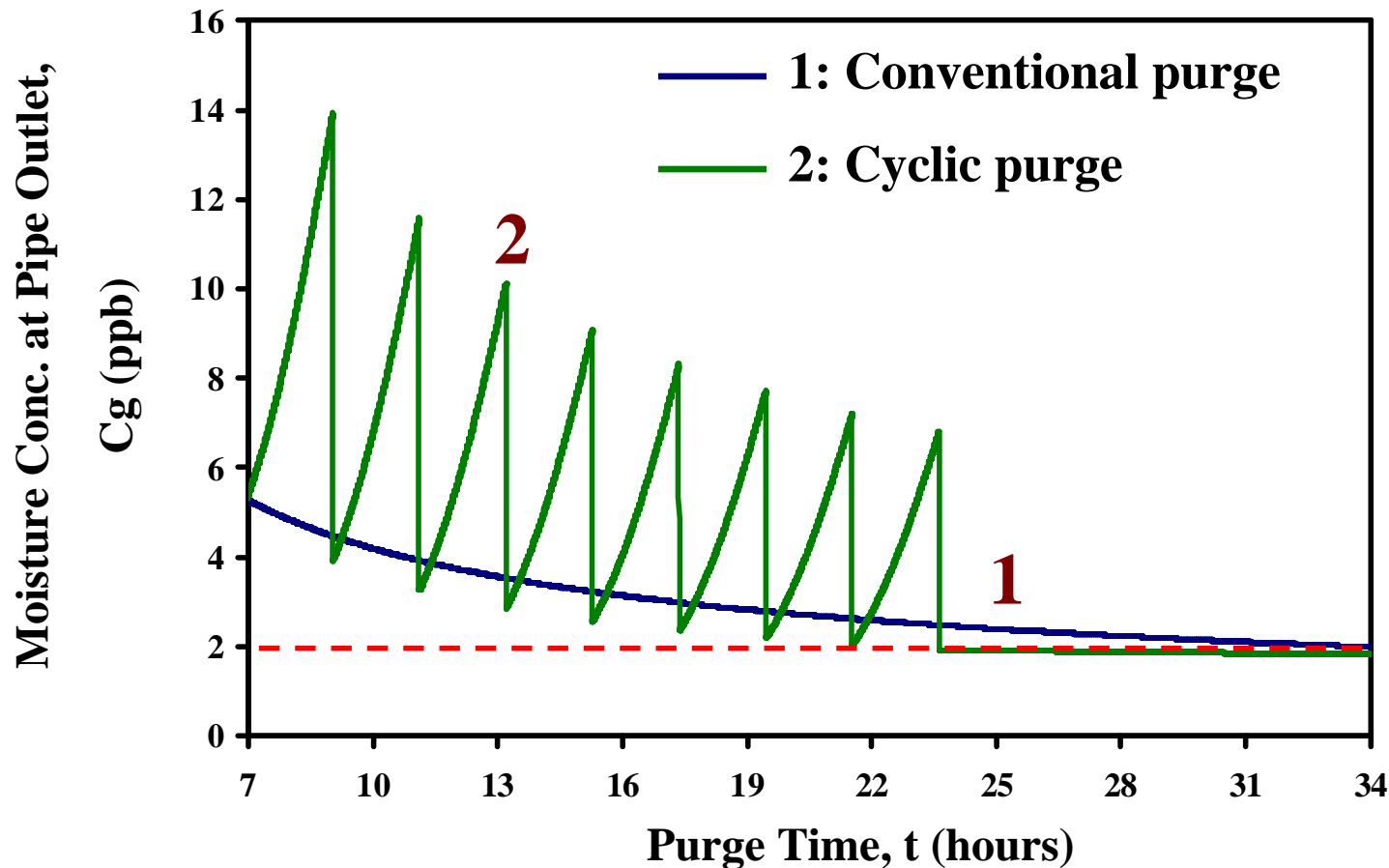


The process simulator well predicts combination of conventional and cyclic purge processes

Cyclic Purge vs. Conventional Purge

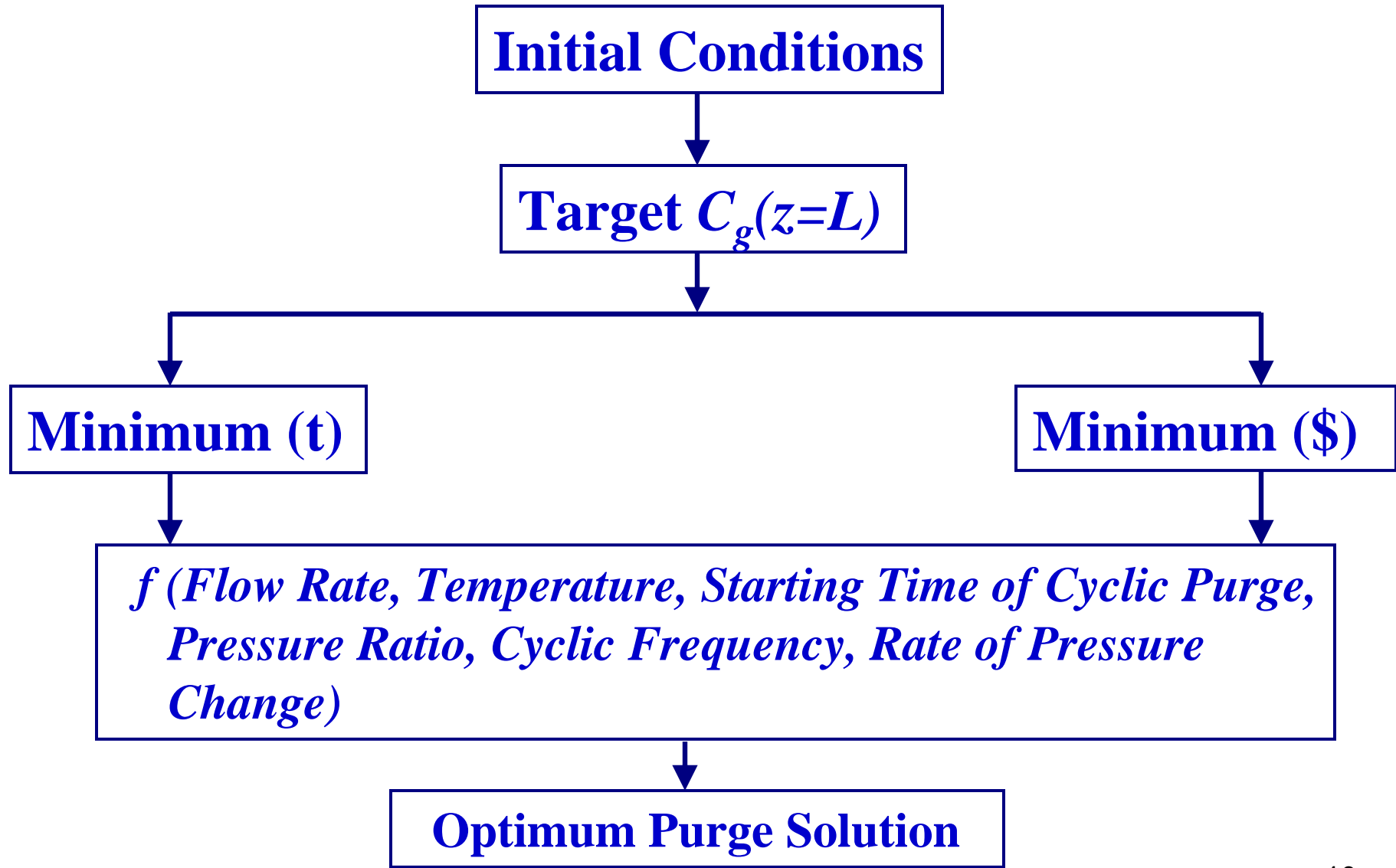
EP SS pipe with 1.5 inch OD and 1640 feet length. Initial conc. 200 ppb;
cyclic purge starts when the moisture concentration reaches 5 ppb.

Industrial-scale system



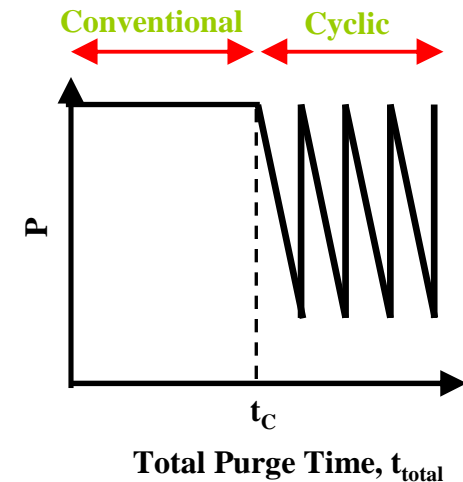
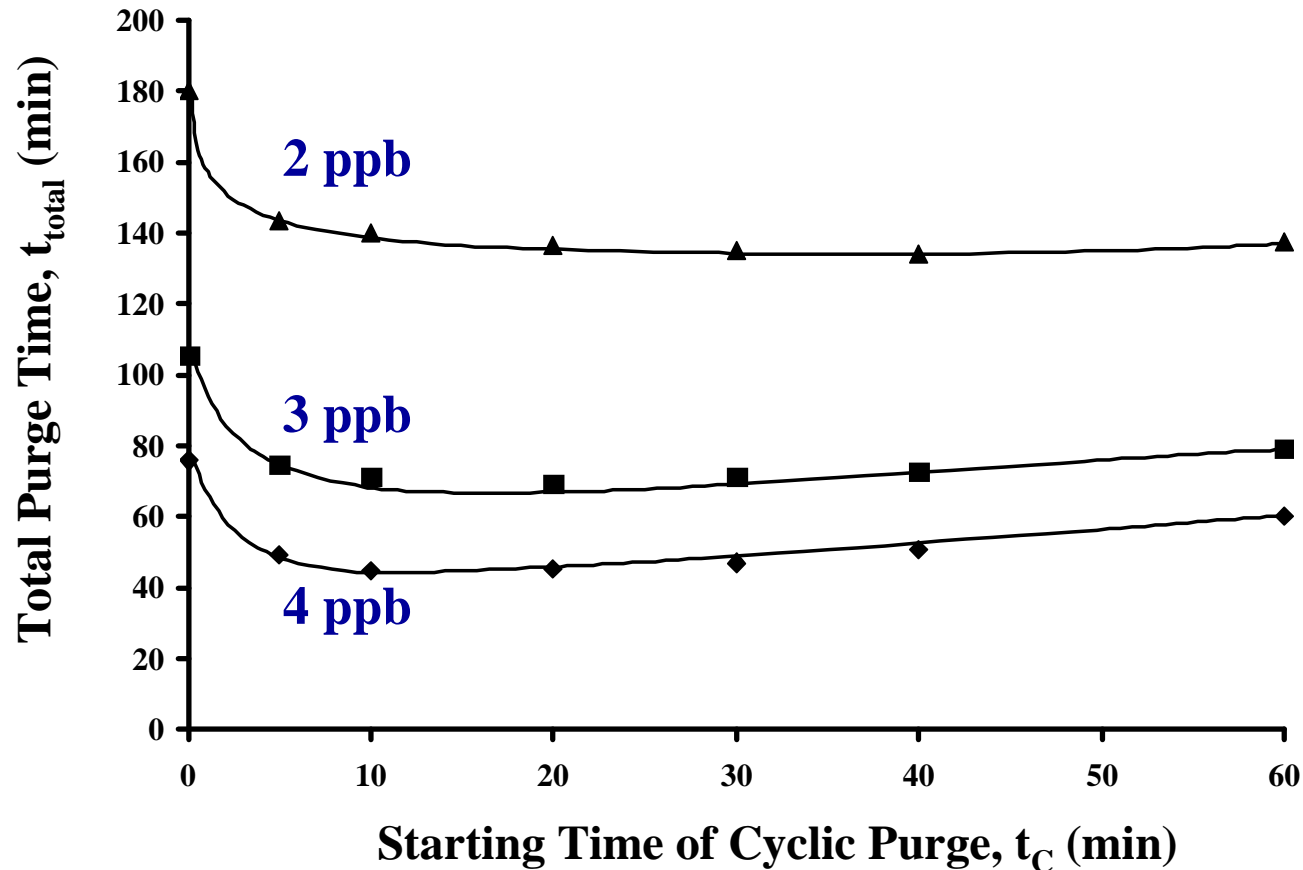
To reach 2 ppb baseline: conventional purge: 34 hours; Cyclic purge: 23 hours; cyclic purge saves 76% of purge gas

Optimization of Purging Processes



Optimum Starting Time of Cyclic Purge

Pipe Length: 10 m, O.D.: 1.5 inch; initial conc.: 200 ppb



The starting time of cyclic purge needs to be optimized in order to minimize the total purge time

SRC/Sematech Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Highlights

- **Compared with conventional purge with high system pressure and high purging gas flow rate, pressure cyclic purge takes less purge time and consumes less purge gas usage.**
- **The purge process simulator developed in this work well predicts experimental results and can be applicable to various size systems.**
- **Purge process must be optimized in order to minimize the total purge time and gas usage.**

Interactions and Future Plans

- Continue working with Intel on this customized joint project; initiate similar applications and studies for other members
- Prepare a process simulator for field applications

Presentations and Papers

- Lowering Material and Energy Usage during Purging Ultra-High-Purity Gas Distribution Systems (presenter), AIChE 2009 Annual Meeting, Nov. 2009, Nashville, Tennessee.
- Application of Pressure Cyclic Purge (PCP) in Dry-down of Ultra-High-Purity Gas Distribution Systems, under preparation and will be submitted to *Chemical Engineering Sciences*

Acknowledgements

- Intel: Val Strazds
- Tiger Optics LLC

Integrated Electrochemical Treatment of CMP Waste Streams for Water Reclaim and Conservation (Customized Project)

PIs:

- James Farrell, Chemical and Environmental Engineering, UA
- James C. Baygents, Chemical and Environmental Engineering, UA

Graduate Students:

- Francis Dakubo, PhD candidate, Chemical and Environmental Engineering, UA
- David Hubler, PhD candidate, Chemical and Environmental Engineering, UA
- Mark Brown, MS candidate, Chemical and Environmental Engineering, UA
- Pui Foon Lai, MS candidate, Chemical and Environmental Engineering, UA

Undergraduate Students:

- Jake Davis, Ritika Mohan, Kyle Kryger, Chemical Engineering, UA

Cost Share (other than core ERC funding):

- GEP Smith Fellowship (D. Hubler), Triffet Prize (D. Hubler), Mining Engineering Fellowship (F. Dakubo), NASA Space Grant Fellowship (K. Kryger), Water Sustainability Program Fellowship (R. Mohan)
- Water Resources Research Center (\$17K)

SRC/SEMATECH Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Objectives

- **Develop an electrochemical method for removing Cu^{2+} , H_2O_2 , colloidal abrasives, chelating agents and corrosion inhibitors from wastewater generated during CMP.**
- **Compare contaminant removal with industry benchmarks for use of reclaimed water.**
- **Build a prototype reactor and pilot test on real CMP wastewater.**
- **Compare the economic impact per gallon of water used in semiconductor industry to impact for other water uses.**

ESH Metrics and Impact

1. *Reduction in emission of ESH-problematic material to the environment*

- Eliminate the disposal problems associated with membrane concentrates.
- Eliminate the disposal of Cu-laden nanoparticles into hazardous waste landfills.

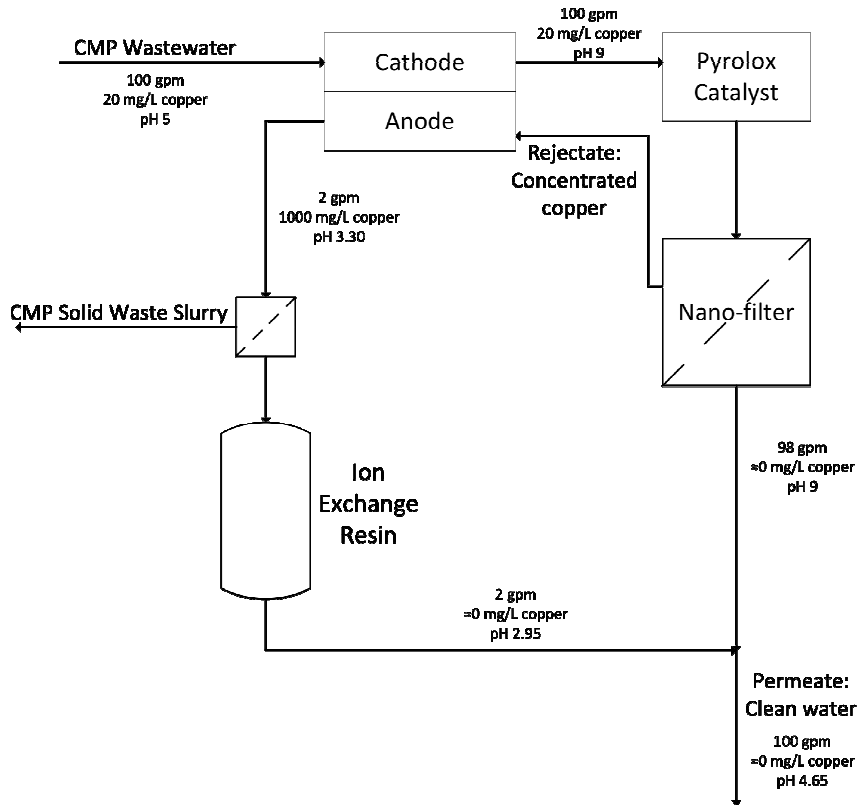
2. *Reduction in the use of natural resources (water and energy)*

- Reclaim CMP wastewater for use in mechanical systems.
- CMP wastewater accounts for up to 30% of fab water use.

3. *Reduction in the use of chemicals*

- Eliminate the need for pH adjusting chemicals and reducing agents that add to TDS load.
- Eliminate the need for activated carbon regeneration.

Electrochemical Treatment System



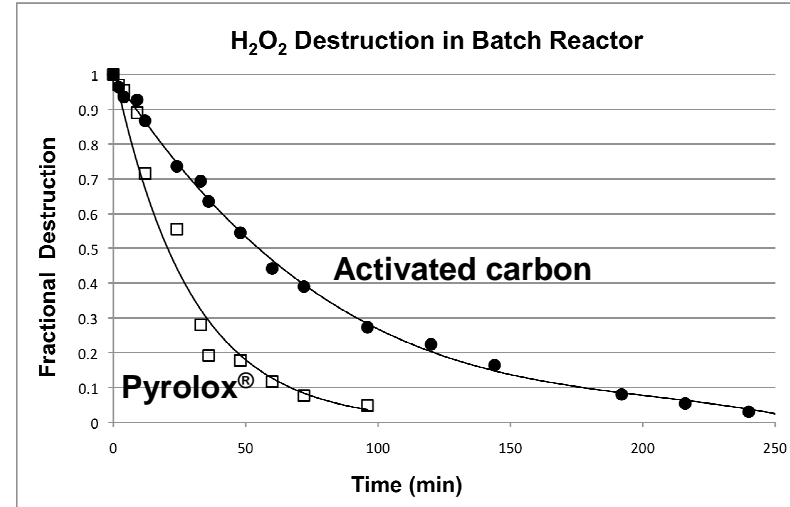
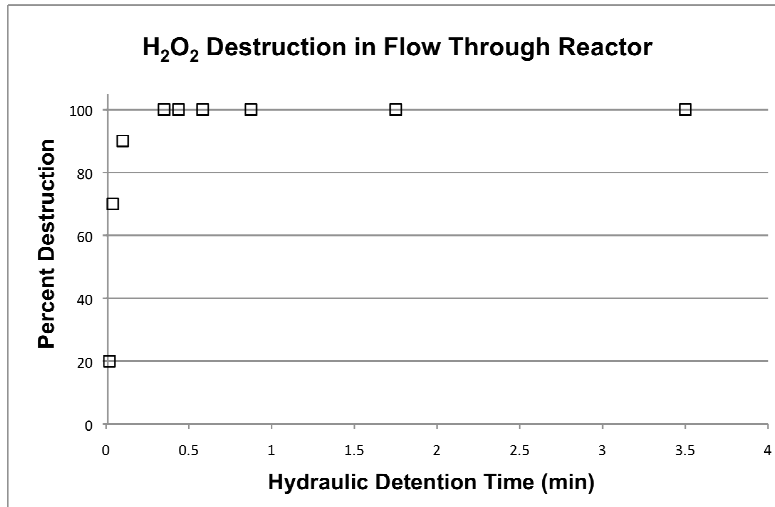
System combines standard filtration unit operations with:

- 1) novel H_2O_2 destruction catalyst
- 2) electrochemical pH manipulation
- 3) electrochemical ion exchange regeneration

Advantages of electrochemical treatment:

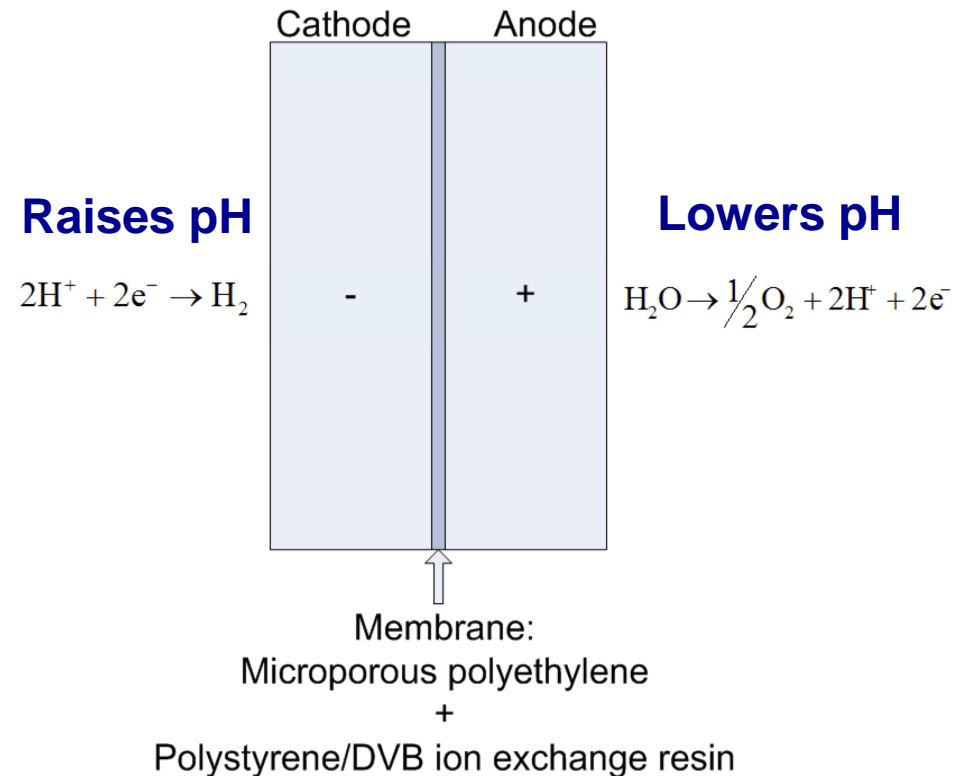
- 1) elimination of chemical additives (e.g., pH adjusting chemicals)
- 2) elimination of secondary waste stream production requiring further treatment or disposal (e.g., ion exchange brines)
- 3) small footprint, low capital and operating costs

Peroxide Destruction Catalyst



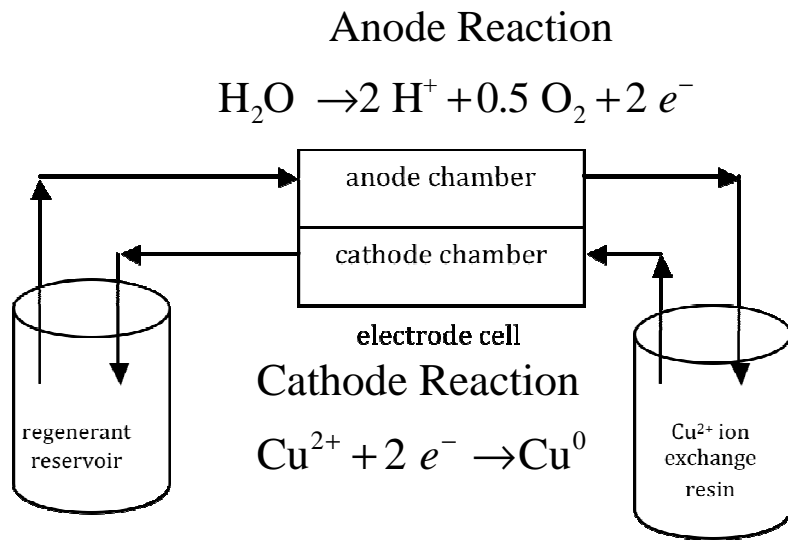
- Conducted tests on six possible peroxide destruction catalysts
- Conducted tests on ultrasonic, O₃ and UV-light peroxide destruction
- Pyrolox® (pyrolusite= β -MnO₂) catalyst determined to be the most effective
- Pyrolox® is commercially available and used for removing Fe²⁺ from drinking water

Membrane for Electrochemical pH Manipulation



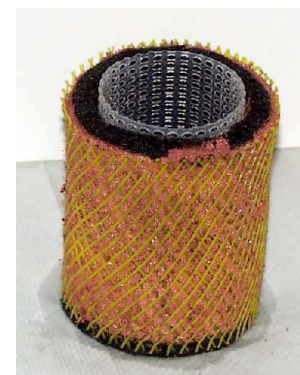
- Developed a novel membrane that is resistant to pinhole leaks common with conventional membranes
- Novel membrane will be useful in other electrochemical applications

Electrochemical Ion Exchange Regeneration



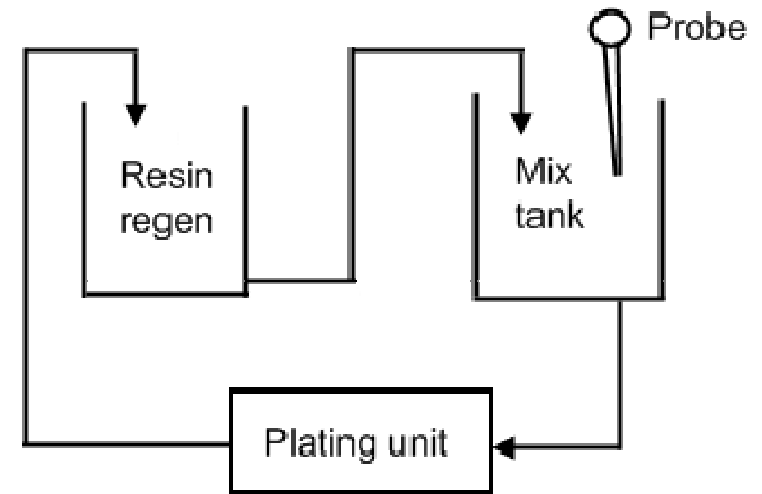
Power cost = \$8.40/m³-resin

Cu value = \$240/m³-resin



Mathematical Model for Ion Exchange Regeneration

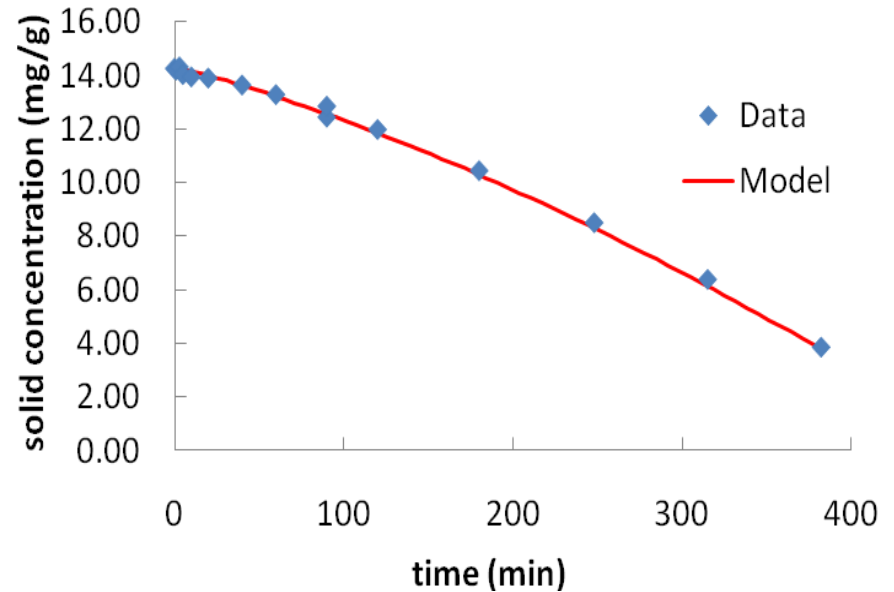
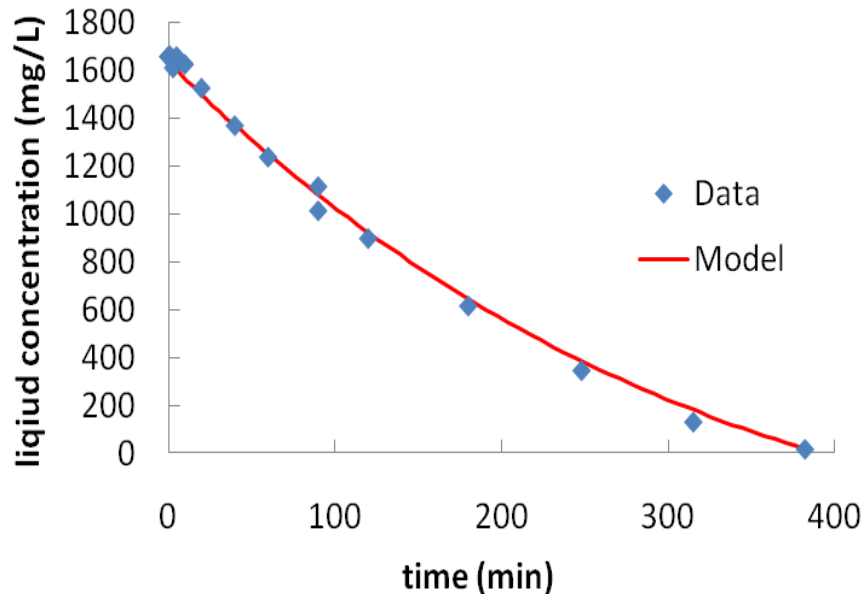
- Cu plating rate can be limited by mass transfer or applied current
- Trade-off between Cu plating rate and Faradaic efficiency
- Mathematical modeling used to optimize the plating rate for cost or time considerations



Liquid mass balance:
$$\left(V_{MIX} + V_P + \varepsilon V_{RR} \right) \frac{dc}{dt} = -k_0 A + V_{RR} K_m \left[c_{eq}^l (c_s) - c \right]$$

Solid mass balance:
$$V_{RR} \frac{dc_s}{dt} = -V_{RR} K_m \left[c_{eq}^l (c_s) - c \right]$$

Resin Regeneration Modeling



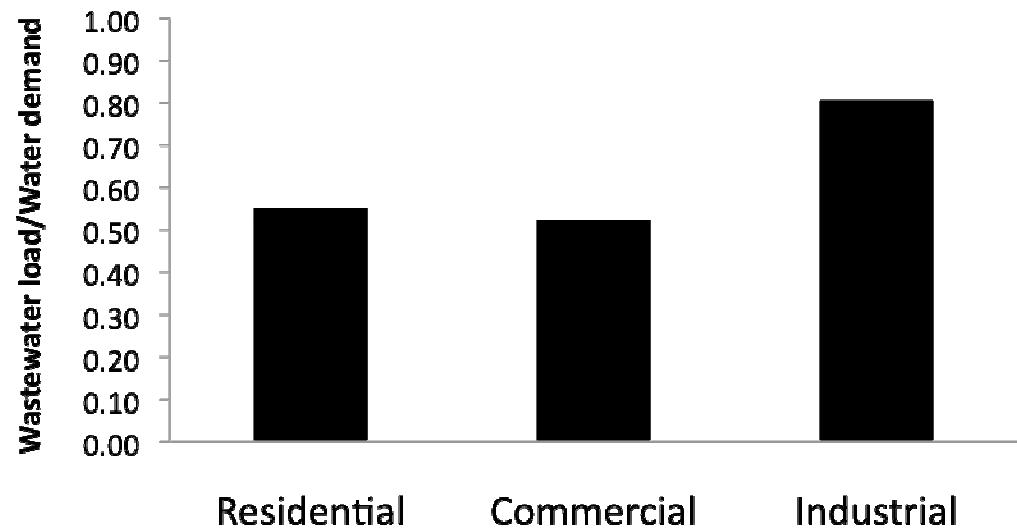
- Modeling can predict both liquid and solid phase Cu²⁺ concentrations
- Modeling will be used to scale-up from prototype to full scale system

Parameters: $V_{MIX} = 6.0$ L; $V_{RR} = 3.4$ L; $V_P = 3.0$ L; $\varepsilon = 0.3$; $K_m = 0.162$ min⁻¹; $k_0A = 110.7$ mg/min
Isotherm: $c_{liq} = (1/(1.97 \times 10^{-3})) \times (c_{sol}/(18.6 - c_{sol}))$, c_{liq} in mg/L, c_{sol} in mg/g

Economic Impact of Semiconductor Water Use

- Evaluate the economic benefits of selected uses of water
- Question: Is high volume semiconductor manufacturing a rational use of water, especially in a semi-arid community?

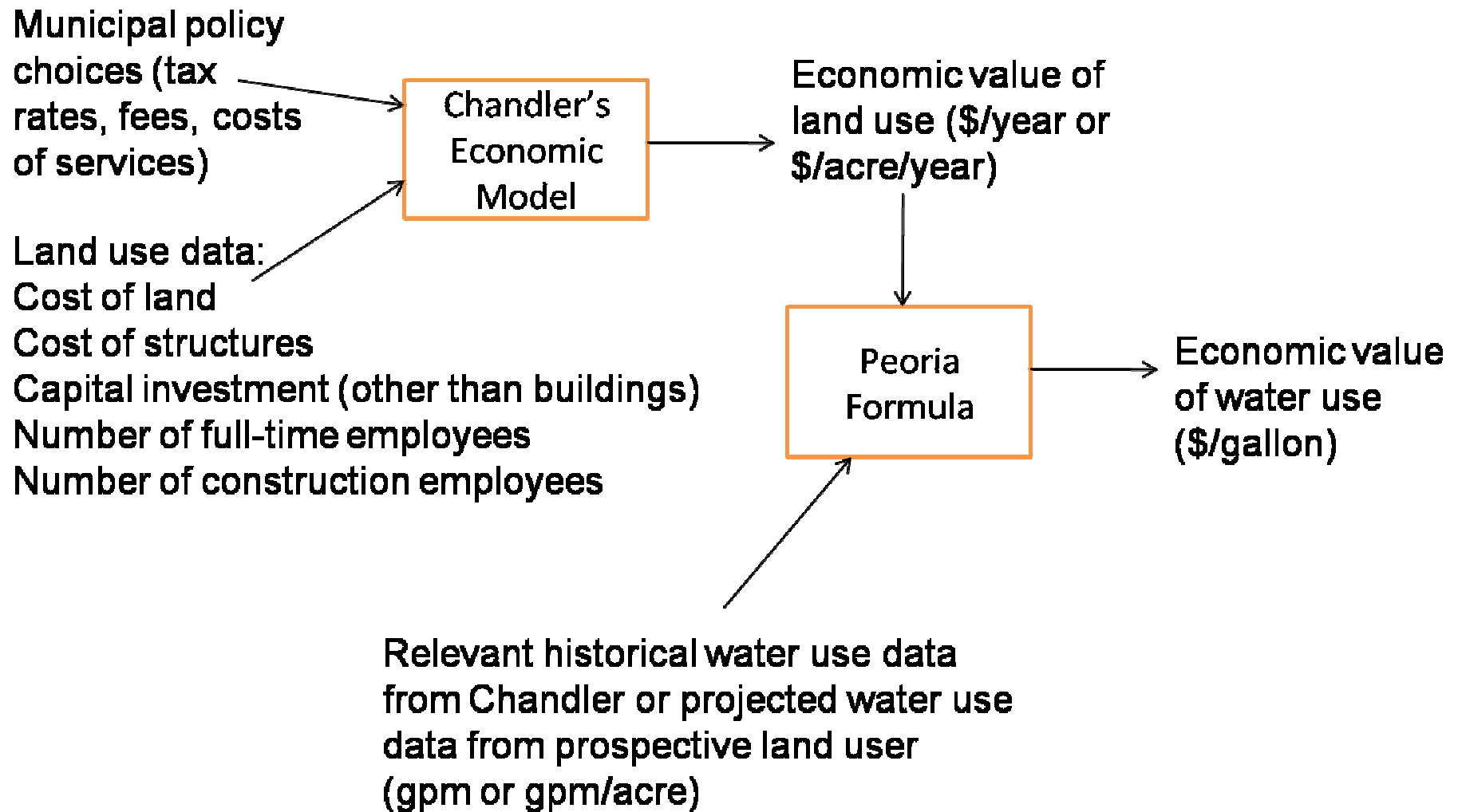
Fraction of water returned by different land users:



- Not just a matter of total water use, as industrial users can return more water to the city, and semiconductor HVM can go beyond other industrial users

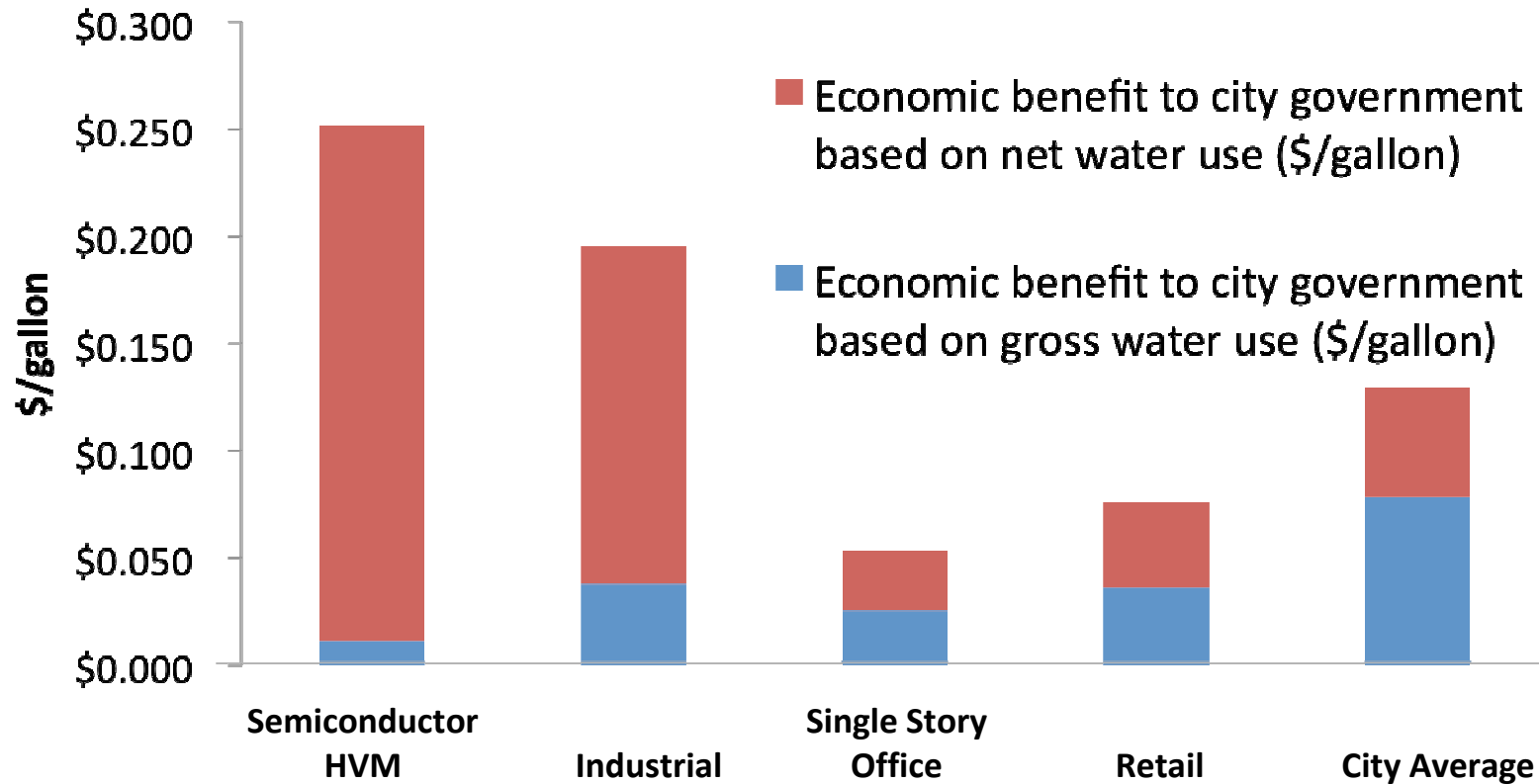
Economic Impact of Semiconductor Water Use

Method for evaluating economic impact per gallon:



Economic Impact of Semiconductor Water Use

Economic benefit to city government:



- Semiconductor HVM is a reasonable use of water in semi-arid or arid communities, as long as infrastructure exists to recapture value from wastewater streams.

Industrial Interactions and Technology Transfer

- **Don Hooper, Intel Corporation**
- **Dan Hodges, Intel Corporation**
- **Allen Boyce, Intel Corporation**
- **Avi Fuerst, Intel Corporation**

- **Sharon Megdal, Water Resources Research Center, UA**
- **Christine Mackay, Economic Development Director, Chandler, AZ**

Future Plans

Next Year Plans

- Pilot test of complete system

Long-Term Plans

- Apply electrochemical treatment methods to other wastewater streams

Publications, Presentations, and Recognitions/Awards

- **David Hubler: Triffet Prize and GEP Smith Fellowship**
- **Francis Dakubo: Department of Mining Engineering Fellowship**
- **Kyle Kryger: Ella Philipossian Memorial Scholarship & Pillars of Excellence Award**
- **David Hubler: First Place Award, University of Arizona 2009 Student Showcase, B.P.A. Division, November 6-7, 2009, Tucson, AZ**
- **“Economic Benefit of Commercial and Industrial Water Uses in a Semi-arid Municipality,” presented at the Arizona Hydrological Society/American Institute of Hydrology 2009 Hydrological Symposium, August 30-September 2, 2009, Scottsdale, AZ**
- **“Electrochemical Methods for Water Reclaim in Semiconductor Manufacturing,” presented at the International Conference on Microelectronics Pure Water, November 11-12, 2008, Mesa, AZ**
- **“Electrochemical Water Treatment using Diamond Film Electrodes,” presented at the University of Illinois at Urbana-Champaign, November 7, 2008**

Optimization of Dilute Ammonia-Peroxide Mixture (APM) for High Volume Manufacturing Through Surface Chemical Investigations (Intel Customized Project)

PIs:

- Srini Raghavan, Material Science and Engineering, UA
- Jinhong Zhang, Mining and Geological Engineering, UA

Graduate Students:

- Shariq Siddiqui, PhD candidate, Material Science and Engineering, UA

Cost Share (other than core ERC funding):

- Horiba SC-1 monitor on loan

Objectives

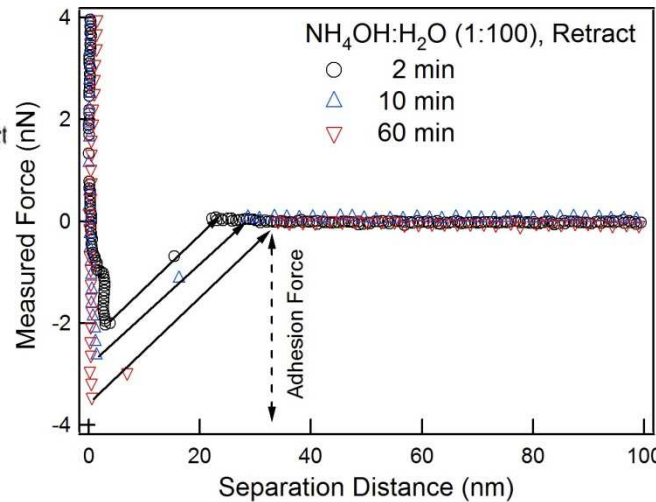
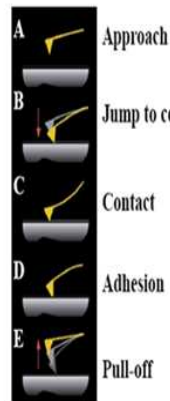
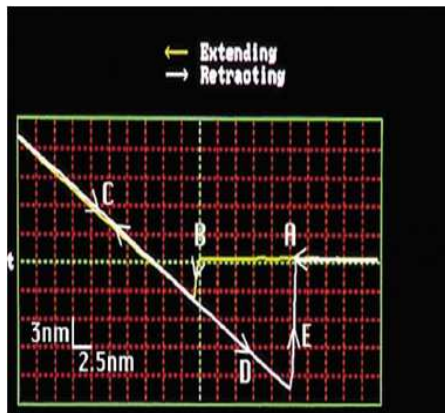
- 1) Optimization of ammonia-peroxide mixtures (APM) for particle removal through interaction force measurements using atomic force microscope (AFM).
- 2) Investigate the stability of ammonium hydroxide and hydrogen peroxide in APM solutions as a function of temperature, dilution ratio and trace metal ion.

RESULTS –Part I

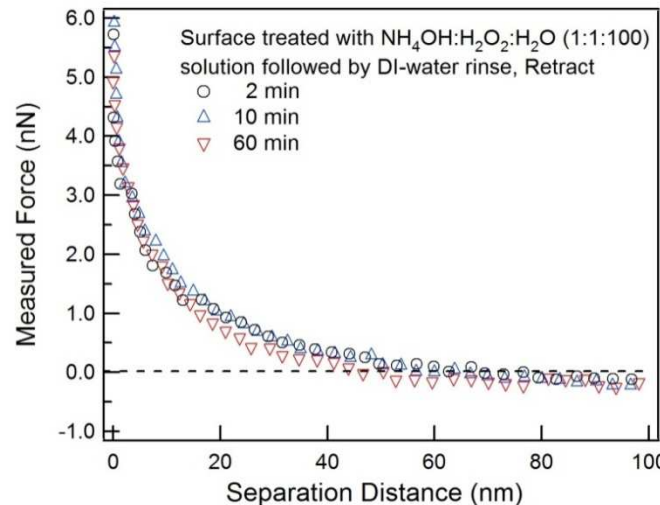
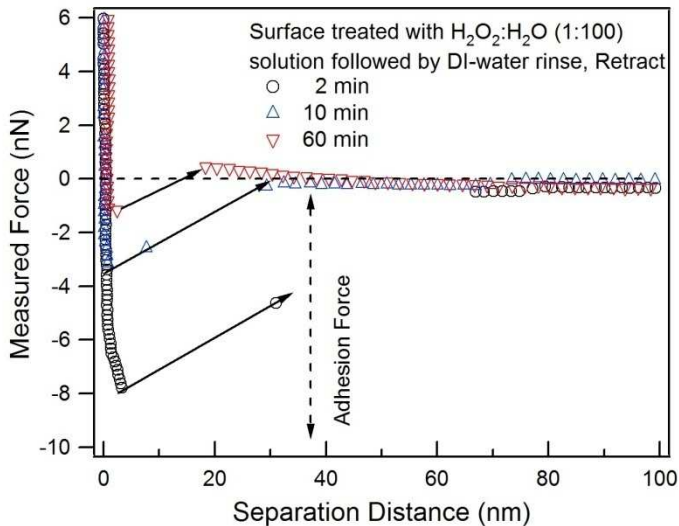
Force measurements between *H-terminated Si surface* and a *H-terminated Si tip* in dilute APM (1:1:100) solution and its components.

Adhesion Force Measurements between a H-terminated Si surfaces in a dilute APM Solution & Components

• p-type Si (100) substrate and Si AFM tip.

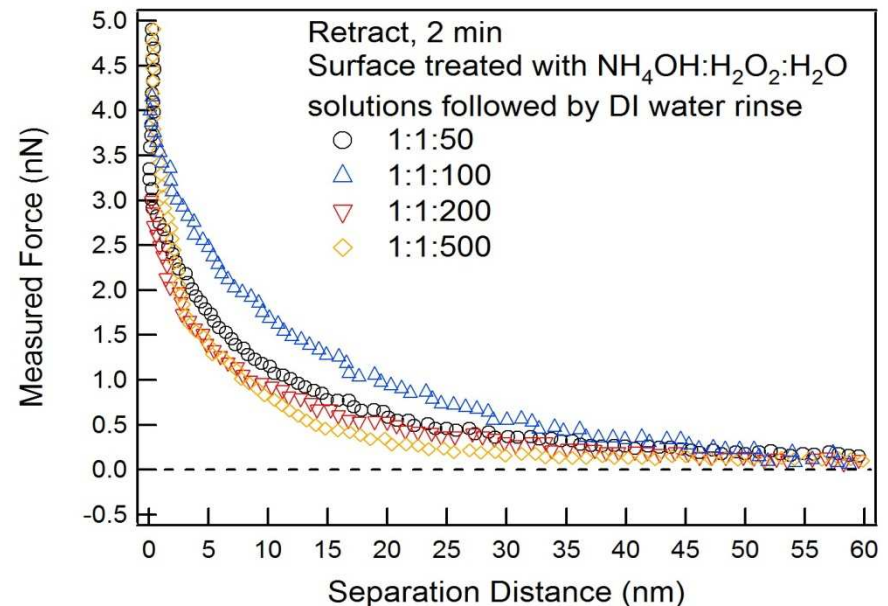
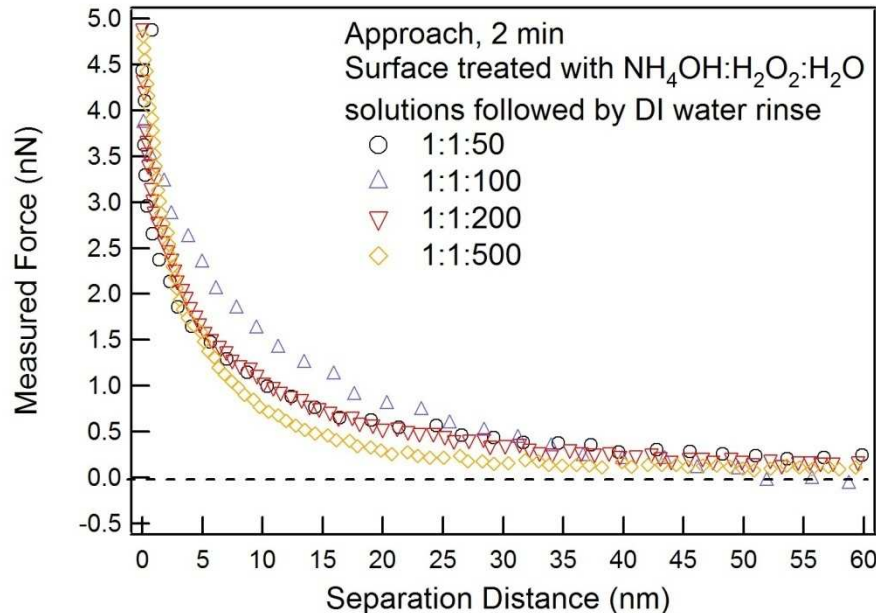


• Adhesion force exists between Si surface and Si tip $\text{NH}_4\text{OH}:\text{H}_2\text{O}$ (1:100) and $\text{H}_2\text{O}_2:\text{H}_2\text{O}$ (1:100) solutions.



• Only repulsive force was measured between Si surface and Si tip in a dilute APM 1:1:100 solution.

Effect of dilution on Interaction forces between H-terminated Si surfaces

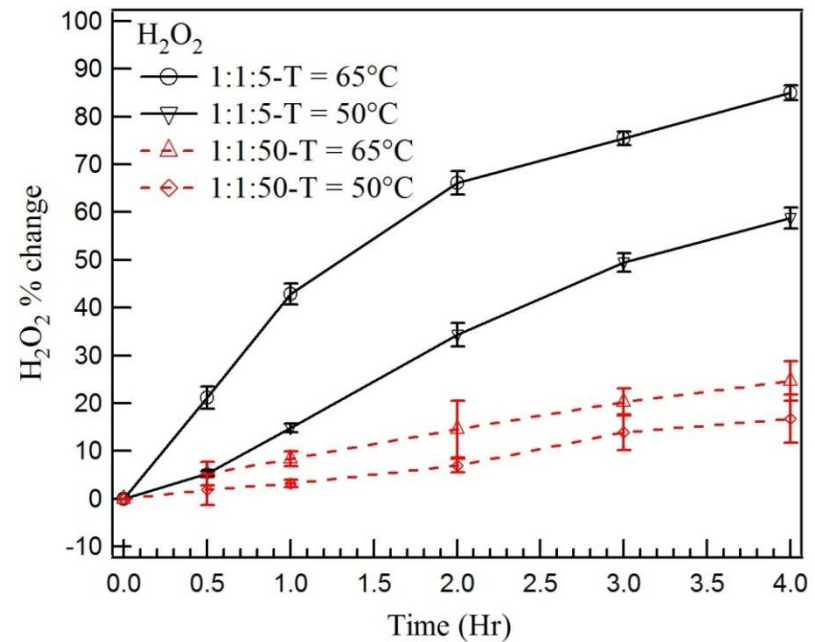
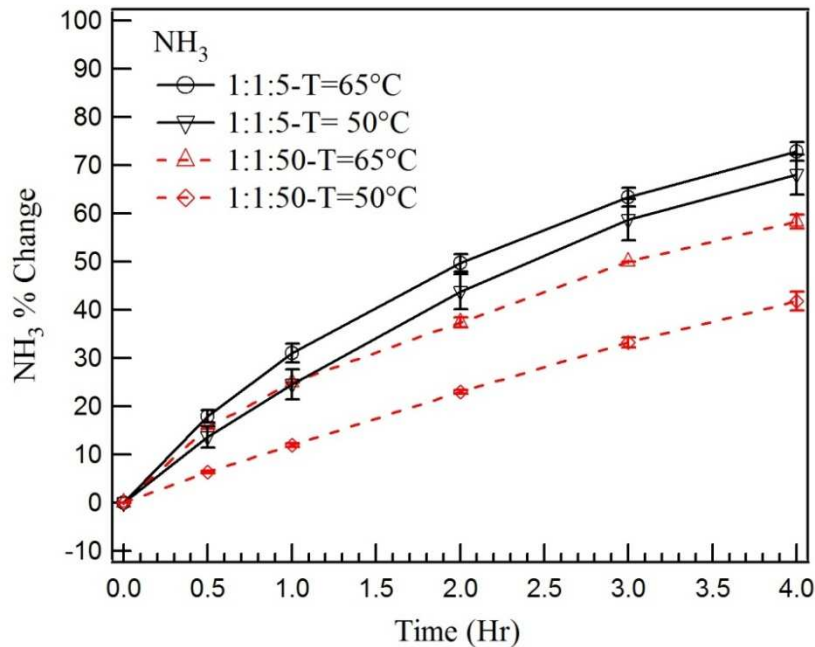


- Both approach and retract curves between Si surface and Si tip show a net repulsive force in APM solutions ranging from 1:1:50 to 1:1:500 within 2 min of immersion.
- *These results indicate that particle re-deposition can be prevented in even very dilute APM solutions.*

RESULTS –Part II

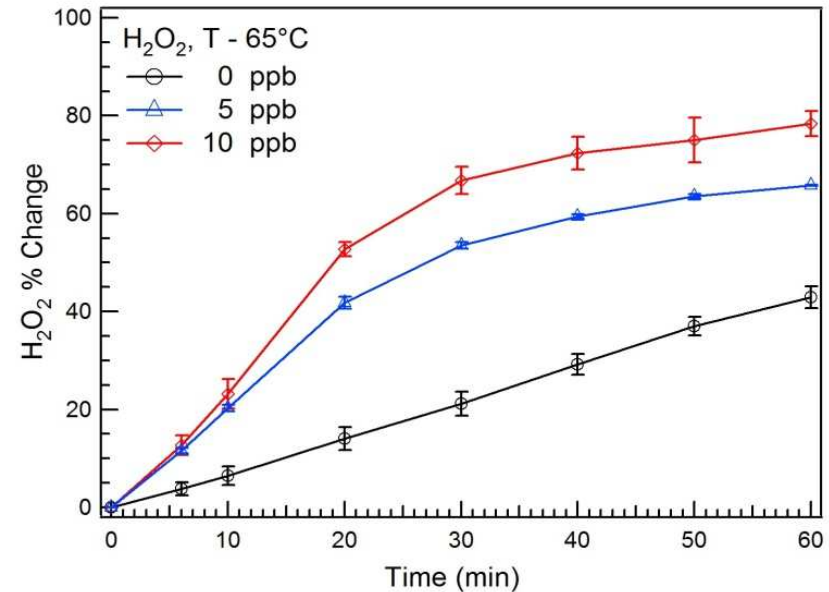
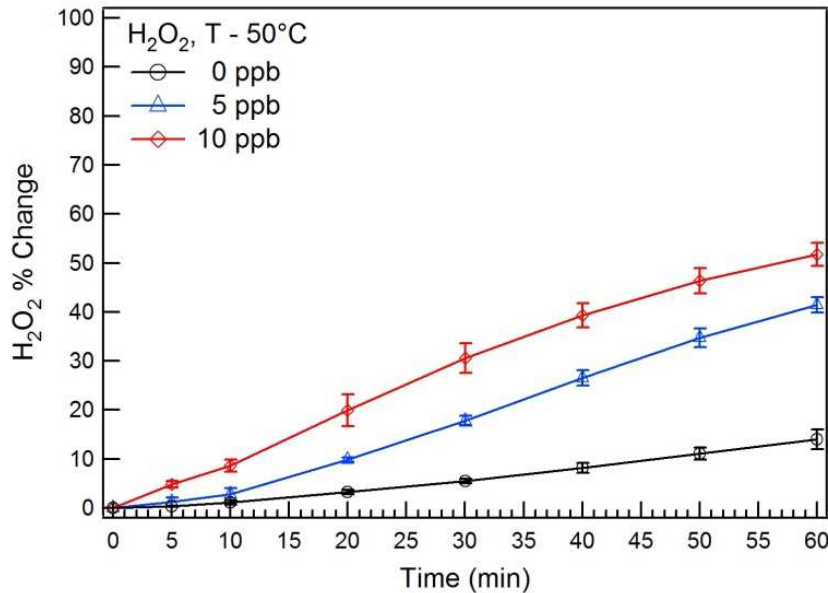
Measurement of the stability of *ammonium hydroxide* and *hydrogen peroxide* in APM solutions using Horiba SC-1 composition monitor.

Effect of Dilution on NH_4OH and H_2O_2 Decomposition



- Extent of decomposition of both ammonium hydroxide and hydrogen peroxide decreases with dilution of APM solution.
- In four hours, ammonia concentration decreased by 30% in a dilute (1:1:50) APM solution vs. 65% decrease in a conventional (1:1:5) APM solution at 50°C.
- Hydrogen peroxide decomposition measured to be less than 15% in four hours at 50°C and 65°C for a dilute APM (1:1:50) solution.

Decomposition of H_2O_2 in the presence of Fe^{2+} ions in 1:1:5 APM Solution



- Hydrogen peroxide decomposition increases with Fe^{2+} concentration and temperature.
- Highest peroxide decomposition was measured in the presence of 10 ppb Fe^{2+} ion at $65^\circ C$.
 - Time for 50 % decomposition is roughly 30 and 20 min at 5 and 10 ppb of Fe^{2+} .

Highlights

- Adhesion forces prevailed between H-terminated Si surface and a H-terminated Si tip in DI water, ammonium hydroxide and hydrogen peroxide solutions up to 60 min.
- Interaction force measurements in dilute APM solutions showed repulsive forces between Si surface and Si tip within 2 min of immersion.
- Ammonium hydroxide and hydrogen peroxide decomposition increases with temperature for a conventional APM (1:1:5) solution.
- Dilute APM solution (1:1:50) showed less decomposition at elevated temperatures when compared to 1:1:5 solution
- Hydrogen peroxide decomposition increases with Fe^{2+} concentration and temperature.

Future Plans

- Interaction force measurements at elevated temperatures (40, 50 and 65°C)
- Measurements of stability of ammonium hydroxide and hydrogen peroxide in dilute APM in the presence of trace metal ions.
 - Proposed metal ions are: Fe²⁺ and Fe³⁺

Industrial Interactions

Industrial Mentor

- **Avi Fuerst, Intel Corporation**
- **Barry Brooks, Intel Corporation**
- **Eric Hebert, Horiba Inc.**

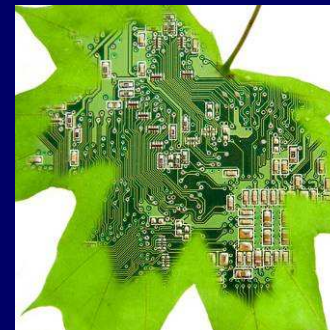


Accelerating Sustainable Manufacturing

Challenges in Semiconductor Energy Reduction and the Progression Towards Idle Mode

Thomas Huang, P.E.

Project Manager,
ESH Technology Center





Agenda/Topics

- Challenges in Energy Reduction for Fabs and Equipment
- SEMATECH/ISMI Previous Highlights
- 2008 Case Study: Idle Mode Feasibility
- 2009 Pump Idle Demonstration
- Next Steps



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Fab Challenges in Energy Reduction

- Rapid changes in fab loading make it difficult to stay optimized for energy efficiency
- Operating well below design capacity usually results in poor efficiency

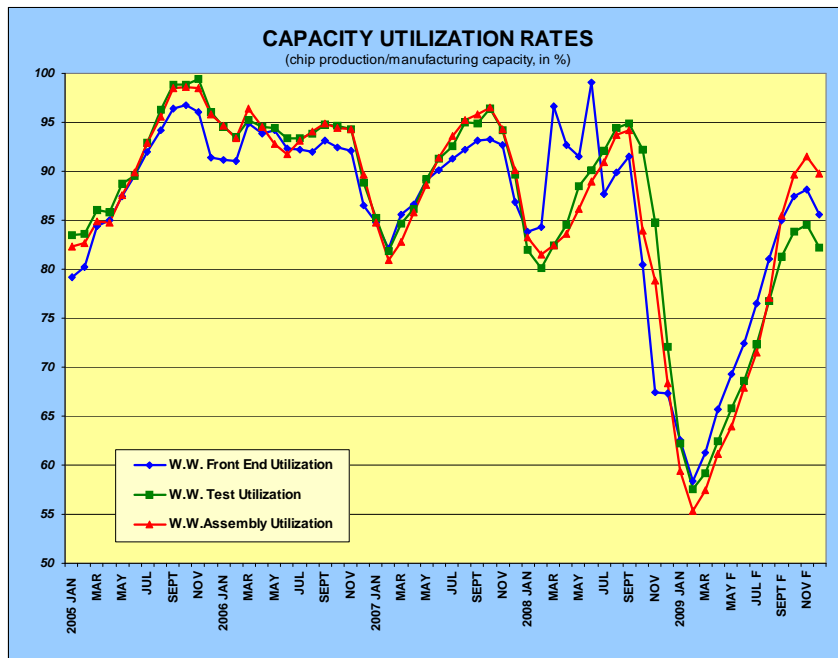


Chart Source: *Industry Pulse*, VLSI Research

Unloading Efficiency

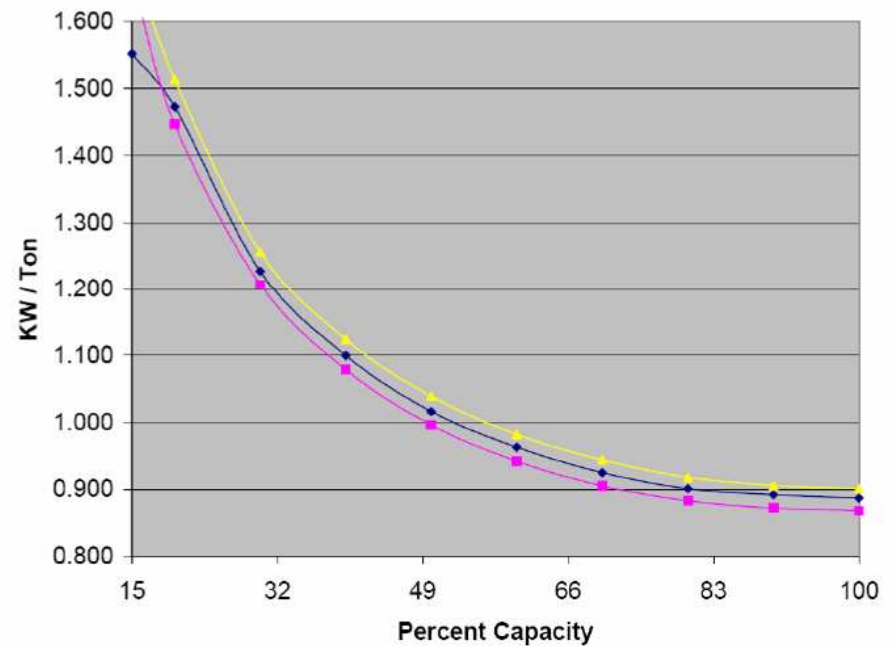


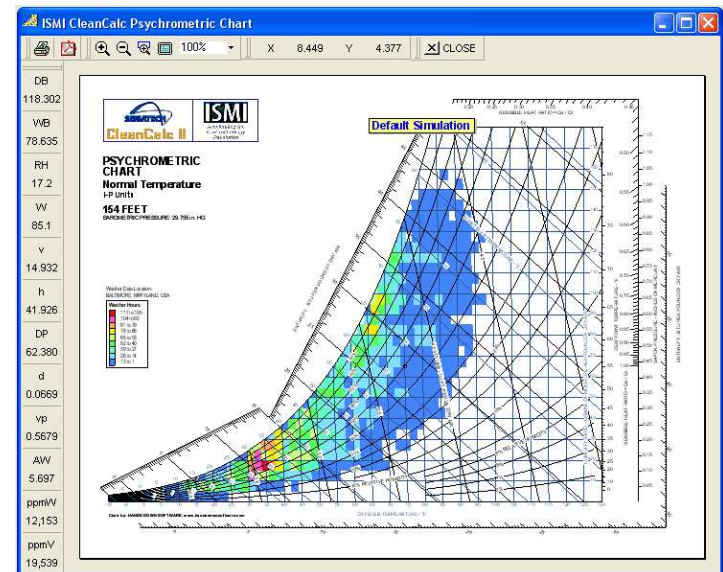
Chart Source: Source: Cascadia Region Green Building Council, "Heat Recovery Chillers" R. Scott Rushing and Eric Vander Mey,

Facilities E-Reduction Challenges

- Process and production liability far outweighs potential energy savings

Example:

- Approach: increase temperature and humidity tolerances to reduce HVAC energy requirement for tight process control
- Potential annual cost savings \$100-250k USD per fab
- Process impact: stepper and scanner focus and alignment issues, critical dimension control

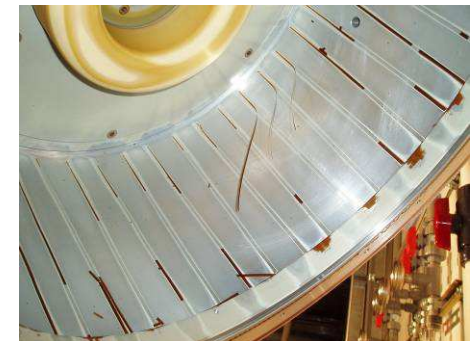


Equipment E-Reduction Challenges

- Process stability and uptime conflicts with energy reduction

Example:

- Approach: reduce chamber heating during tool idle periods
- Potential annual cost savings \$5k USD savings per tool
- Process impact: chamber deposition peeling and flaking requiring wet cleans and significant down time



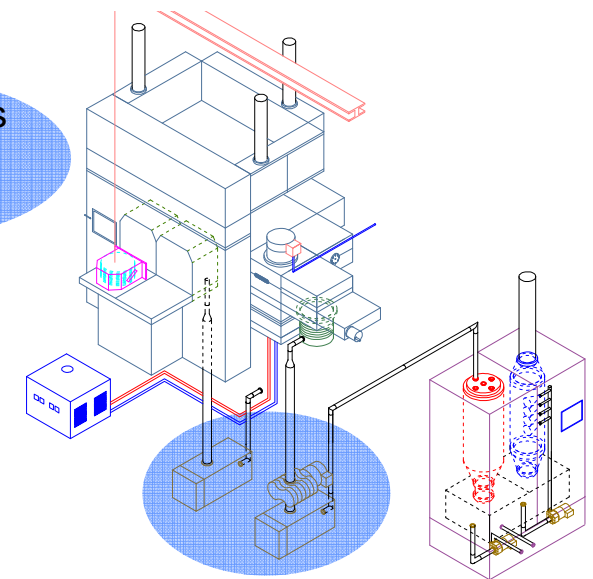
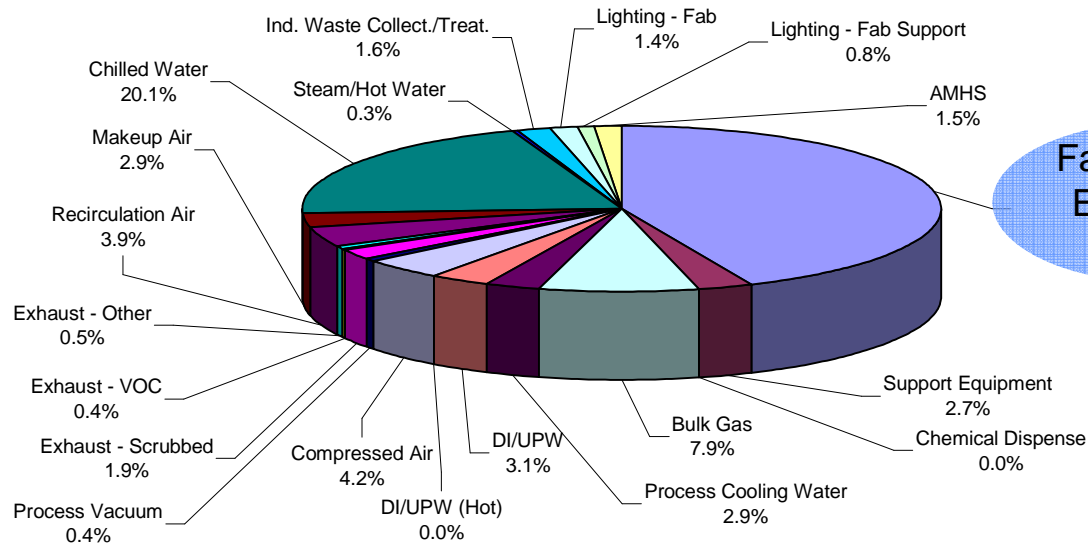


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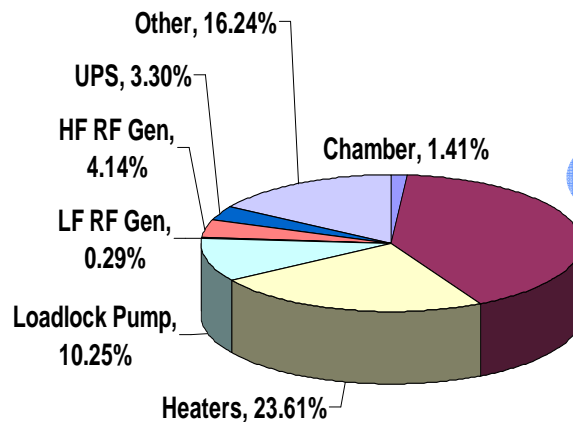
Previous Fab and Equipment Energy Studies

300 mm Fab energy



Focus on Pumps

300mm CVD tool energy



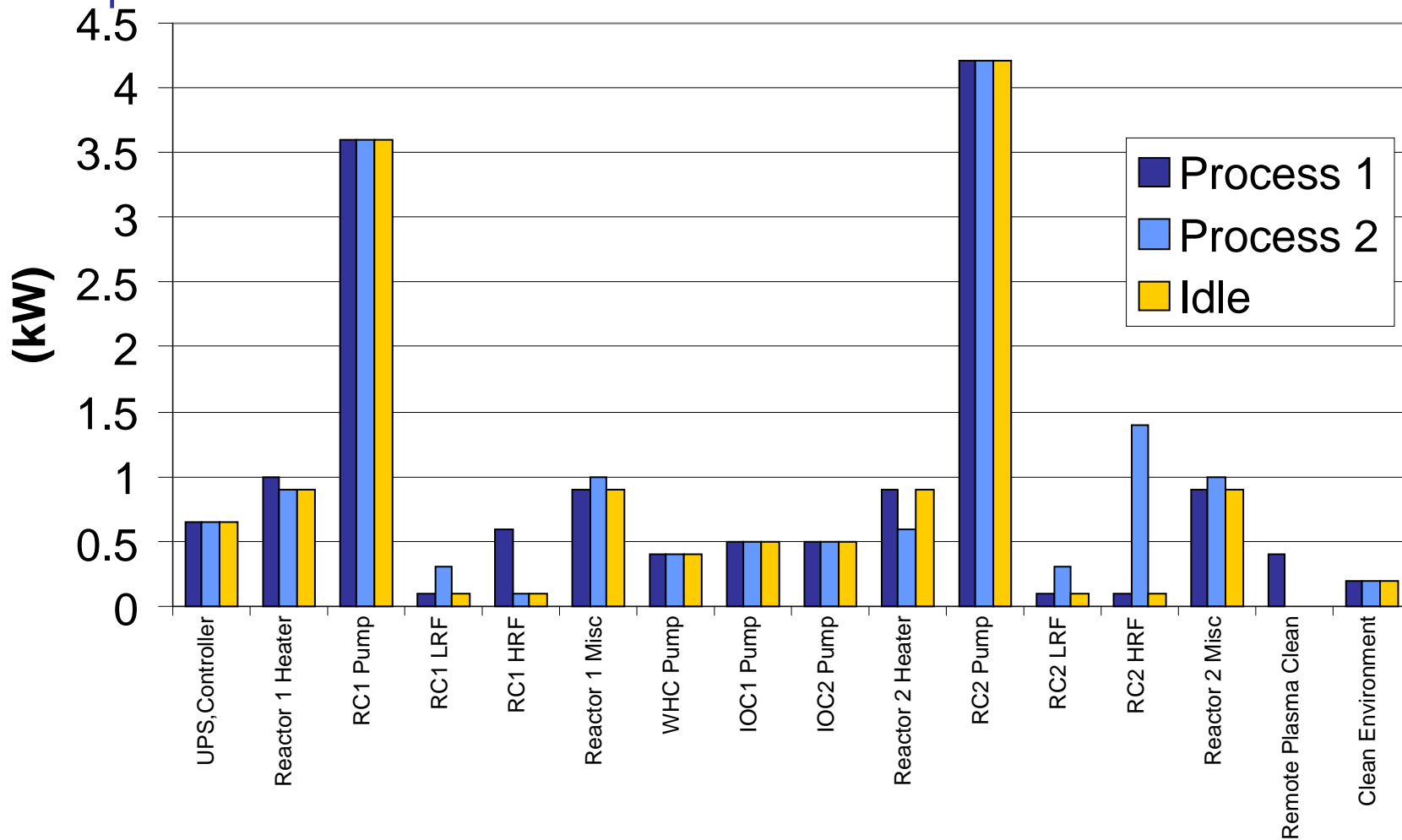
Chamber Pump
40.77%

Chamber Pump	40.77%
Loadlock Pump	10.25%
Heaters	23.61%
Total	74.63%

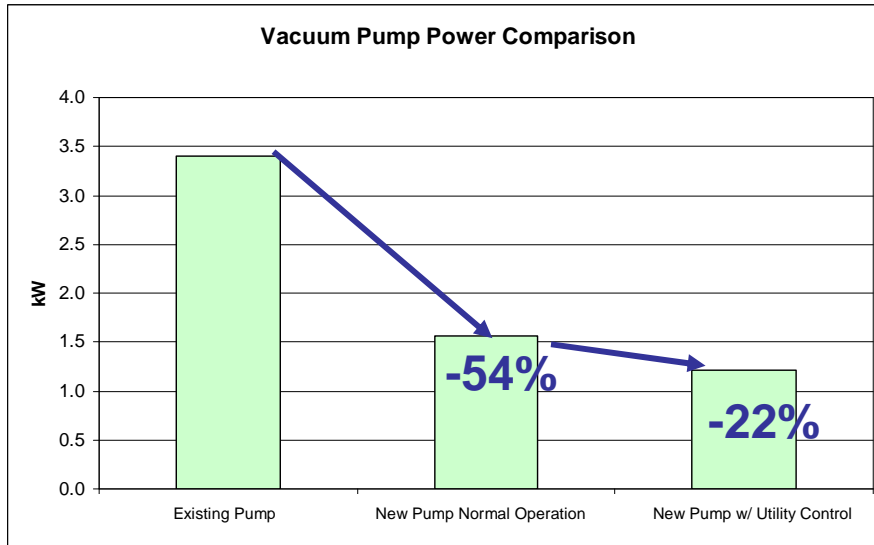
Energy Pareto Chart for CVD Processes



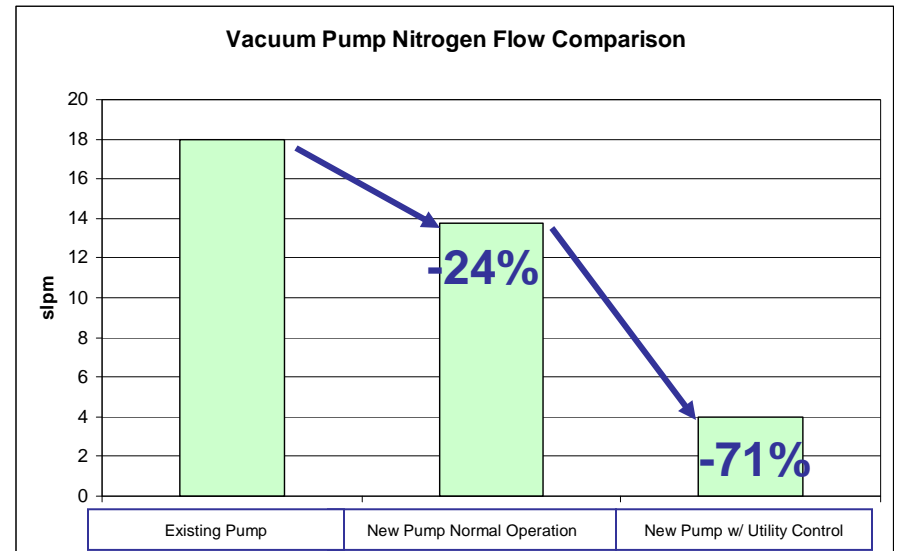
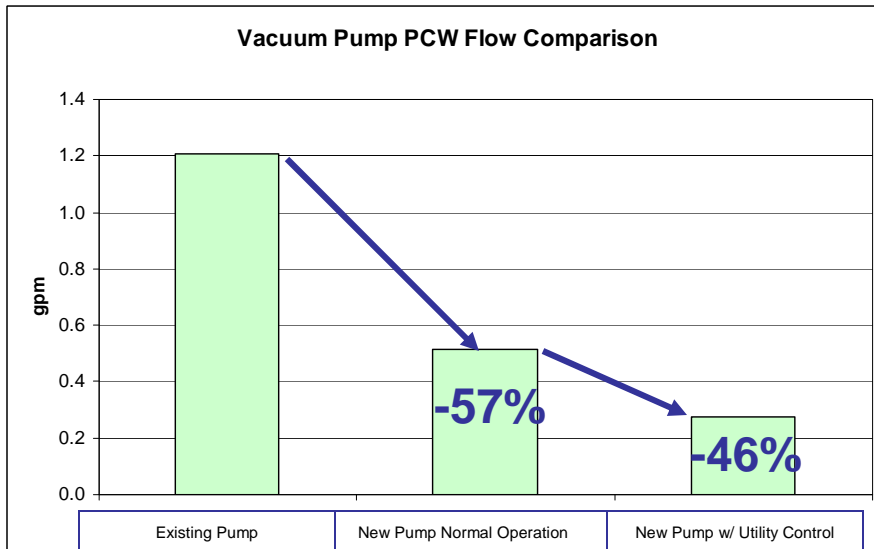
Back in 2000, there was no change from idle to process except in RF



Vacuum Pump Speed Control Results



- Idle mode power could be further reduced depending on process.
- Nitrogen purge reduction can be reduced an additional 71% if the process allows.





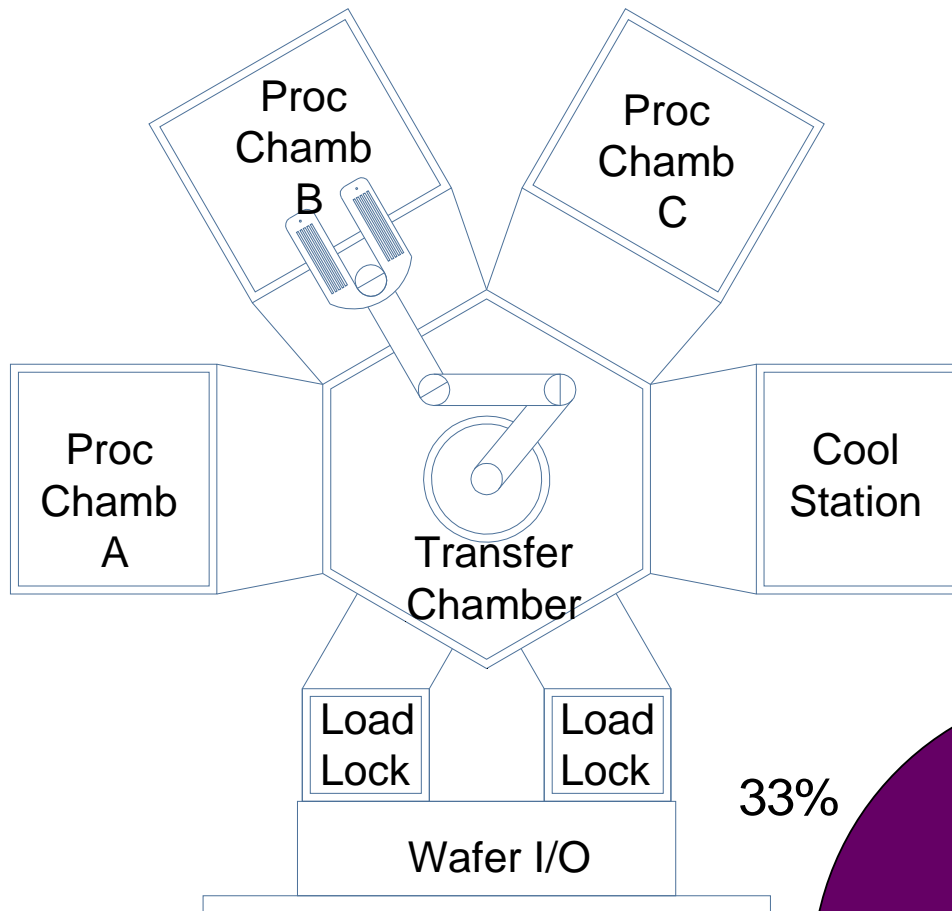
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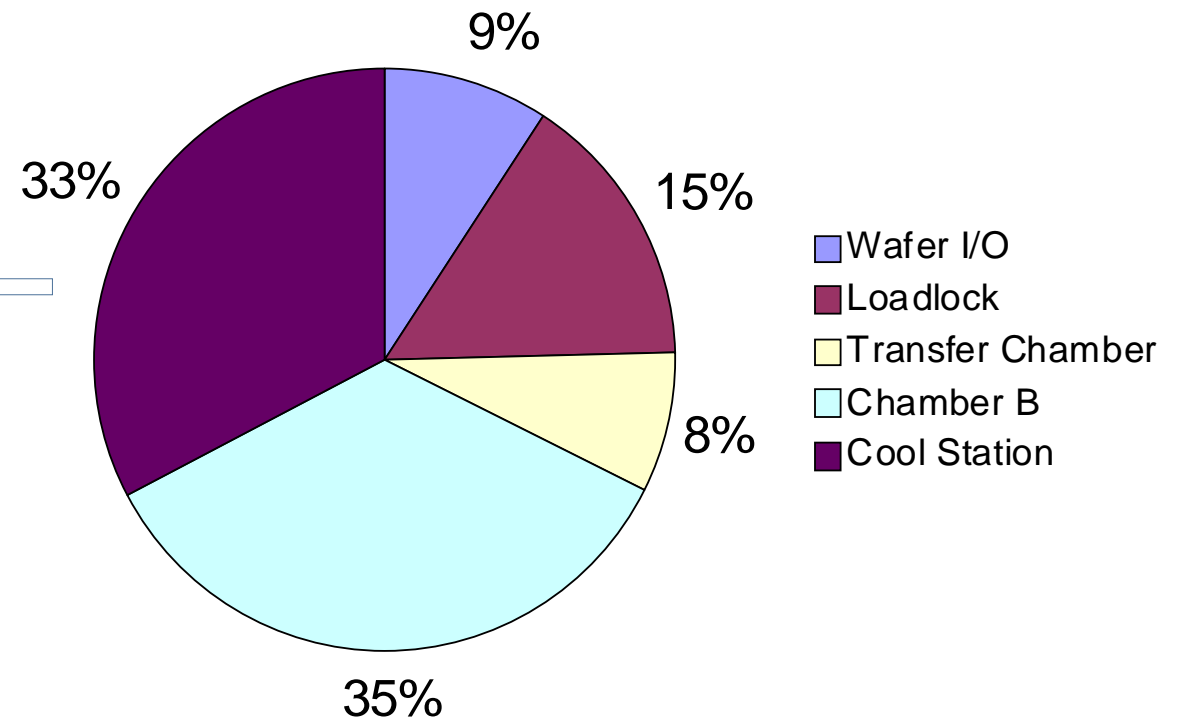
Wafer Event Analysis – Raw Tool Log Data



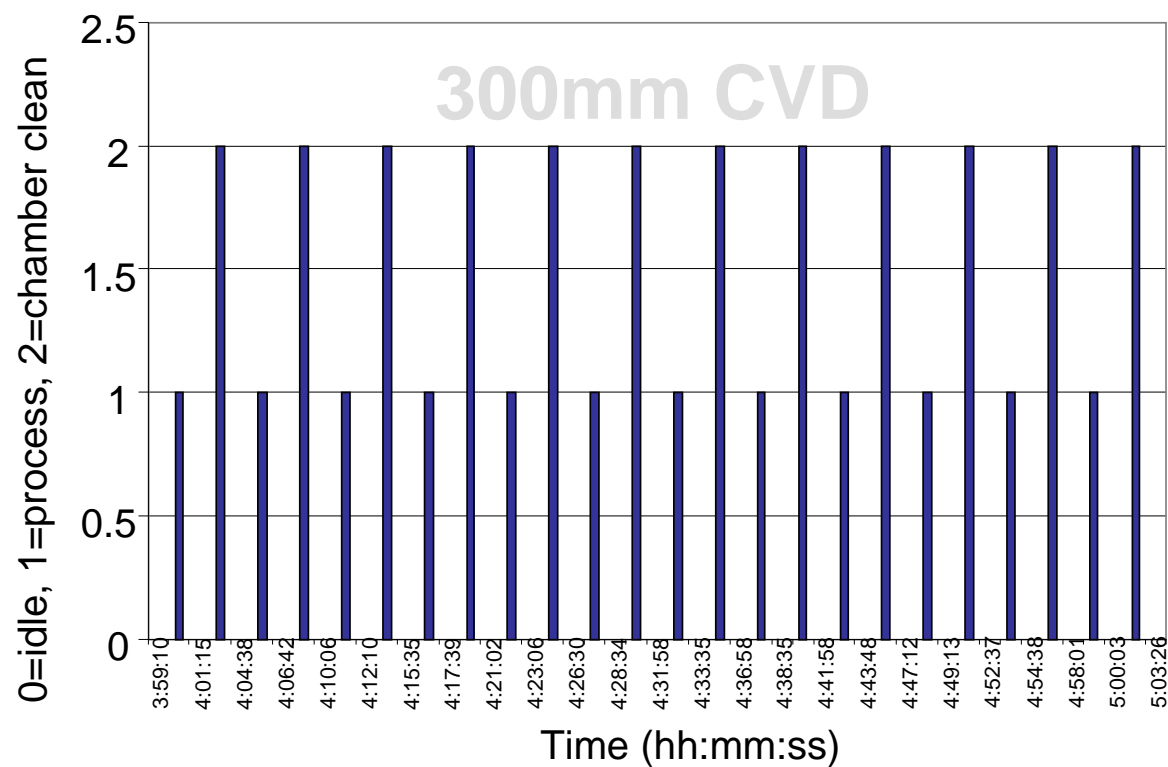
#EventID	Severity	Source	CreationTir	Text
3420	147464	AT/LL	56:18.6	Wafer transfer info - LL Vent finished
3362	147464	AT/LL/Leftl	56:20.5	Wafer transfer info - Door Opened
3362	147464	AT/LL/Righ	56:20.6	Wafer transfer info - Door Opened
204	147464	AT/System	56:25.2	Wafer CB:15 moved from FIC_1_1 to LL_1_1
204	147464	AT/System	56:25.2	Wafer CB:16 moved from FIC_2_1 to LL_1_2
37	147464	AT/System	56:25.2	System/Sequencer.LL_Slot 1_XferCount changed from 244855 to 244856
204	147464	AT/System	56:31.0	Wafer CB:13 moved from LL_2_1 to FIC_1_1
204	147464	AT/System	56:31.0	Wafer CB:14 moved from LL_2_2 to FIC_2_1
3362	147464	AT/LL/Leftl	56:31.9	Wafer transfer info - Door Closed
3361	147464	AT/Buffer/κ	56:33.3	Wafer transfer info - Slit Opened
3362	147464	AT/LL/Righ	56:33.3	Wafer transfer info - Door Closed
3417	147464	AT/LL	56:33.4	Wafer transfer info - Start pump down LL
204	147464	AT/System	56:35.0	Wafer CB:13 moved from FIC_1_1 to COOL_STN_1_1
37	147464	AT/System	56:35.0	System/Sequencer.COOL_STN_Slot 1_XferCount changed from 158143 to 158144
37	147464	AT/System	56:35.0	System/Sequencer.COOL_STN_Slot 1_SoFarCount changed from 159210 to 159211
37	147464	AT/System	56:35.0	System/Sequencer.COOL_STN_Slot 1_CleanOnLoad_SoFarCount changed from 158067 to 158068
58	147464	AT/COOL_	56:35.0	Slot1 Process recipe cool-22 started.
229	147464	AT/COOL_	56:35.0	Recipe step 1 started
204	147464	AT/System	56:35.3	Wafer CB:14 moved from FIC_2_1 to COOL_STN_4_1
37	147464	AT/System	56:35.4	System/Sequencer.COOL_STN_Slot 4_XferCount changed from 148089 to 148090
37	147464	AT/System	56:35.4	System/Sequencer.COOL_STN_Slot 4_SoFarCount changed from 149154 to 149155
37	147464	AT/System	56:35.4	System/Sequencer.COOL_STN_Slot 4_CleanOnLoad_SoFarCount changed from 148011 to 148012
58	147464	AT/COOL_	56:35.4	Slot4 Process recipe cool-22 started.
229	147464	AT/COOL_	56:35.4	Recipe step 1 started
3446	147464	AT/Buffer/\	56:42.5	Chamber B vacuum robot wafer detect data updated
204	147464	AT/System	56:42.6	Wafer CA:21 moved from CHB_1_1 to Buffer_1_1
204	147464	AT/System	56:42.6	Wafer CA:22 moved from CHB_1_2 to Buffer_1_2
37	147464	AT/System	56:42.6	System/Sequencer.Buffer_Slot 1_XferCount changed from 489224 to 489225
3446	147464	AT/Buffer/\	56:42.9	Chamber B vacuum robot wafer detect data updated
3361	147464	AT/Buffer/κ	56:43.2	Wafer transfer info - Slit Closed
37	147464	AT/System	56:43.3	CHB/Side1/HighFreqRF.cFwdPwrDeviationTmo changed from 5 s to 10 s
37	147464	AT/System	56:43.3	CHB/Side2/HighFreqRF.cFwdPwrDeviationTmo changed from 5 s to 10 s
229	147464	AT/CHB	56:44.2	Recipe step 1 started
3418	147464	AT/LL	56:46.2	Wafer transfer info - LL pump down finished
230	147464	AT/CHB	56:47.2	Recipe step 1 completed
229	147464	AT/CHB	56:47.5	Recipe step 2 started
230	147464	AT/CHB	56:47.5	Recipe step 2 completed
229	147464	AT/CHB	56:47.5	Recipe step 3 started
3361	147464	AT/Buffer/κ	56:47.9	Wafer transfer info - Slit Opened
289	147464	AT/System	56:50.0	Cassette A Wafer 24 Started
204	147464	AT/System	56:50.0	Wafer CA:24 moved from CASS_A_24_1 to COOL_STN_7_1
37	147464	AT/System	56:50.0	System/Sequencer.COOL_STN_Slot 7_XferCount changed from 233368 to 233369
230	147464	AT/CHB	56:50.5	Recipe step 3 completed
3380	147463	AT/CHB/Si	56:55.8	RF is ON
3380	147463	AT/CHB/Si	56:55.8	RF is ON



During a wafer process cycle, where does the wafer spend most of its time?



	Summed Duration	Percent	Event Duration
Clean	12:03:58	38%	1-6 min
Idle	15:57:08	50%	12s-10min
Process	3:39:52	12%	1.5-2 min
Total	31:40:58	100%	





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2009 Projects Relating to Energy Reduction

- Resource Conservation
 - Fab Energy and Utility Calculator
 - Energy Star Compatible LEED EB Database
 - Alternative/Renewable Energy for Fabs Feasibility Study
 - Heat Exhaust Recycling Benchmark and Cost Benefit Analysis
 - On-board Total Equivalent Energy (TEE) for Process Equipment
 - SEMI S23 Application Guide and TEE tool Revisions
 - **Vacuum Pump Speed Control Demonstration**
- Technology Development
 - Feasibility of Recycle and Reclaim of Fab and Assembly/Test Process Wastewater
 - Feasibility of Treating and Abatement Solvent-corrosive Mixtures
 - Supplier Leadership Workshops on Energy Reduction

Phase 1 Test Fixture

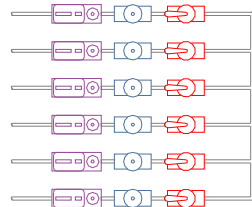


Chamber

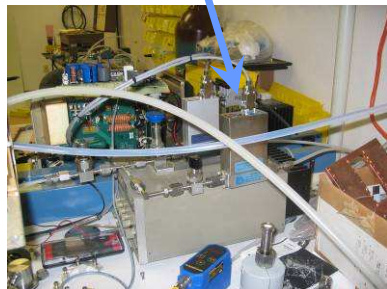
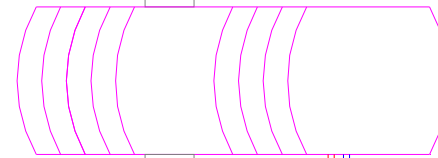
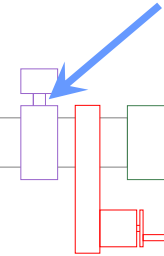
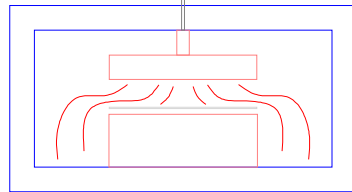
Throttling/Isolation/
Pres Meas



Pump



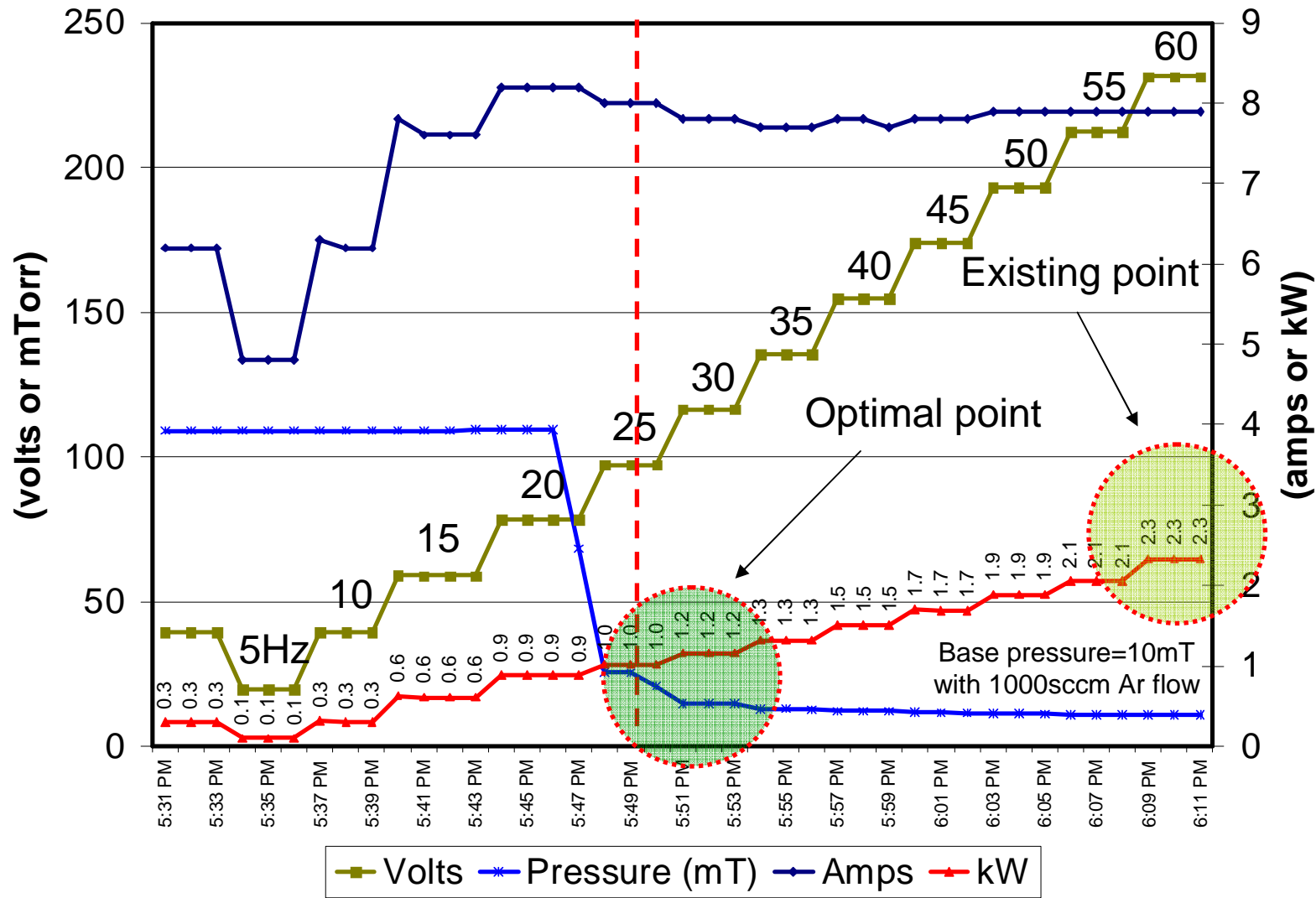
Gas Flow Control



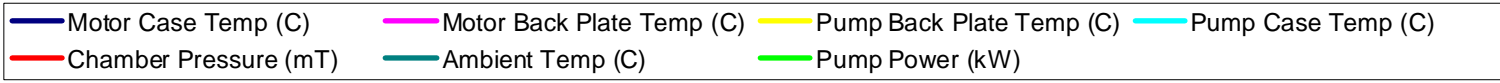
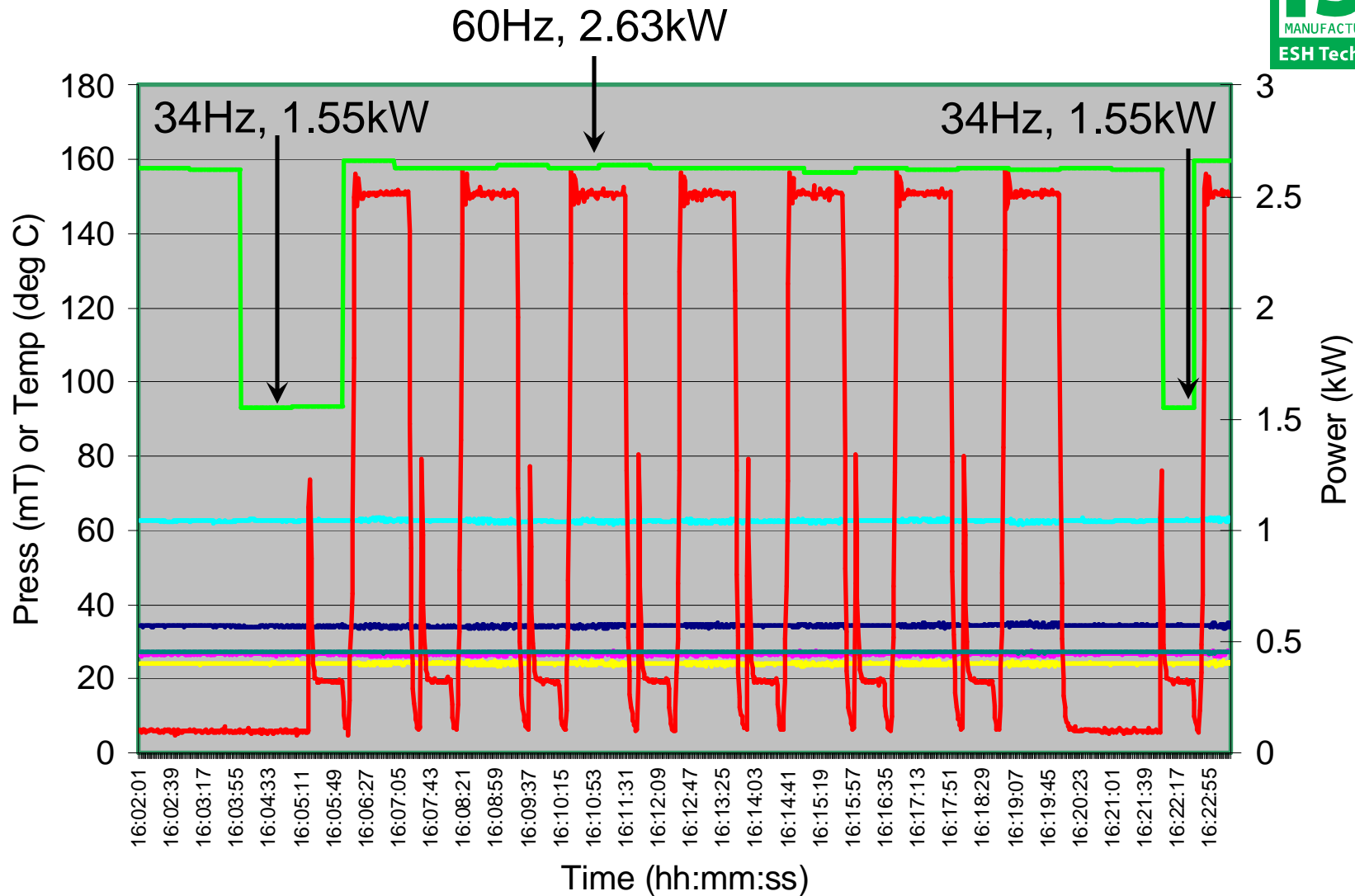
Test Data



For this pump and chamber configuration 25Hz is min



Roughing Pump Power Consumption





Summary

- Data taken on the test stand showed the pump held base pressure while reducing pump frequency from 60 to 25Hz while pumping against a 1000 sccm Ar gas load. Resulted in pump power reduction from 2.3kW to 1.0 kW.
- Pump VFD and Tool Interface Retrofit was repeated on an oxide etch tool
 - Installed on a process chamber, not loadlock or transfer
- On an oxide etch tool, reduced the roughing pump frequency (speed) from 60Hz to 36Hz with no impact to the turbopump load or speed. Resulting power reduction was from 2.66kW to 1.55kW
- 144 wafers were run making comparisons with the tool in baseline and idle modes. ER, PC and Uniformity were monitored via pre/post thickness data and particle counts. The statistical analysis showed no effects on the process data from running the tool in idle mode.



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2010 Projects in Energy Reduction

- Revisit/refresh Facilities Energy BKM's
- Standardize communication interface for support equipment (i.e., pumps, abatement)
- Address over specification of utility specifications for process tools
- ESH assessment on EUV with focus on utility consumption
- Continue efforts to enable reporting and improving process tool energy consumption with Total Equivalent Energy (TEE) software

Some Suggestions

- Reduce redundancy between facilities and process equipment
 - mini-environment vs room air
 - POU chiller vs facility cooling water
- Improve heat transfer and control to eliminate overcool then trim heat strategy
 - POU chiller with immersion heater in reservoir
- Reduce extreme temperature and vacuum requirements
 - idle temperature for furnace = 600-800°C
 - many vacuum levels are at 10^{-9} Torr requiring cryo pumping
 - even a reduction helps
- Increase utilization or recycle percentages
 - single wafer wet tools vs immersion bath tools
 - gas diffusion and plasma utilization
 - source power for EUV, 90kW produces 4W of usable EUV power at intermediate focus