

Proactive Contamination Control: The Only Way to Enable Yield of Advanced Semiconductor Manufacturing

S. Libman^a, G. Van Schooneveld^b and B. McIntosh^c

^a FTD Solutions LLC, Cupertino, California 95014, USA

^b CT Associates, Eden Prairie, Minnesota 55344, USA

^c Georg Fischer Piping Systems, Vancouver, Washington, 98661 USA

Advanced Semiconductor Manufacturing has reached the limits of metrology measuring some critical contamination parameters and defects on both the wafers and in the high purity materials. Ability of the advanced filters to control particles sub-10nm is limited, while high purity materials used in liquid delivery systems have never been qualified for such small particles.

To cope with the metrology gaps, the Yield Enhancement (YE) focus team of IRDS (International Roadmap for Devices and Systems) has defined a new process of proactive technology management in the space of contamination control.

This publication provides an overview of the process including the most recent experimental data generated by YE IRDS team and SEMI Liquid Chemical task forces in support to the proactive contamination control. It also offers examples of how SEMI standards help supply chain in developing new solutions to proactively address critical contamination issues.

Introduction: Why Proactive?

Current state of technology suggests significant gaps in contamination measurement capability. Complexity of the advanced semiconductor devices and continual geometrical scaling led to the situation when killer particle size became much smaller than capability of the most advanced metrology to detect those particles. This situation has been true in UPW (ultrapure water) for nearly a decade, with particle metrology providers investing into closing the gap, while the gap has been only increasing. Current definition of the killer size of the particles is based on half of critical dimension in logic devices and is as small as 3.5 nm for the most critical electrically active particles (EAP) (1).

Recently, the ability to monitor killer particles was also lost for the wafer surface as well. As the result, the risk is considered high to continue monitoring particles of the sizes much larger than the killer size in the attempt of killer particles control. What make the situation even more problematic are the following factors:

1. Most advanced particles filtration in UPW has reached its limits to control killer particles (the killer particles are significantly smaller than the largest filter pore sizes) (2).
2. There are indications that high purity materials shed significant number of particles at current killer sizes or larger (3).
3. There is a concern that high molecular weight polymers may form killer size particle when they attach to the wafer and the water dries out (3).

It should be noted that particles' control in UPW and liquid chemicals is not the only parameter that requires proactive approach. Particles are more critical than other types

of contaminants, leading to yield and reliability problems and therefore used in this paper to illustrate the approach.

Proactive in the IRDS target parameters

As the result of the above deficiencies, the YE chapter of IRDS has changed its direction from Reactive to Proactive Yield management (1). Figure 1 illustrates the idea comparing proactive with conventional reactive approach.

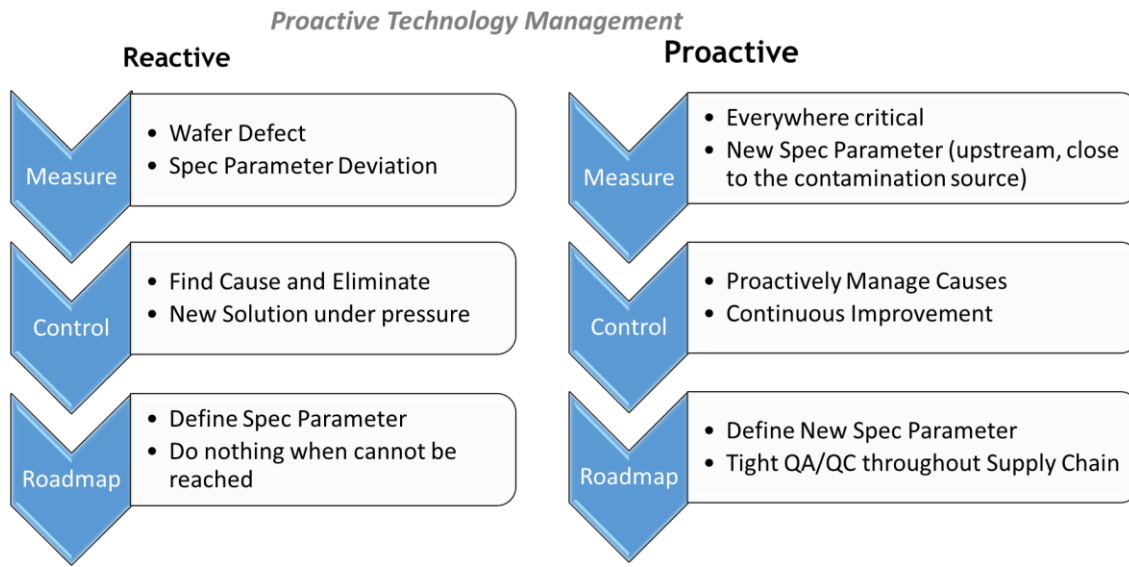


Figure 1. Proactive Technology Management

Therefore, new UPW IRDS parameters have been added to the roadmap targets. This approach assumes no ability to monitor killer particles in UPW, but instead focuses on particle occurrence prevention by both reducing particle challenge to the UPW final filters as well as ensuring adequate performance of the filters. While the roadmap targets for UPW quality define particles not to exceed 10 particles per milliliter at >3.5 nm size (at the point of entry, POE, to the tool chamber), the “proactive” parameter defines particle limit at 30 particles per liter with the size of 50-nm, the level measurable in UPW upstream to the final filters. At this location, the particle size distribution is not affected by filtration and power law correlation can be used to project the level of contamination to the target particle size. Knowing the efficiency of the final and point-of-use (POU) filters, relationship between the particle challenge to the filters and the level of particles at POE can be established. YE white paper (4) provides example of such calculation also explaining how the “proactive” particle target was defined.

Practical Implications of Proactive Particles Control

In contrast with reactive approach, dependent on the measurement at target point, proactive approach is focused on what is measurable and drives continuous improvement ignoring metrology deficiencies. Proactive approach also focuses on risk analysis as opposed to reported yield excursions. This is particularly important in the applications of the semiconductor devices where reliability is critical (i.e., automotive and medical).

The “proactive” particle target level defined by IRDS will help the end user operating team to control performance of their system minimizing the particle challenge to the filters. This includes controlling particle generation by pumps and other system components with high shear stress.

In recent years, YE IRDS team together with the SEMI Standards Liquid Chemical committee developed a series of the documents providing tools and methodologies to implement proactive particles control in advanced semiconductor facilities. Figure 2 indicates the numbers of the SEMI Standards supporting the effort. SEMI F104 (5) is the method for testing critical components for particles, thus helping material and components suppliers to improve particle performance. SEMI C79 (6) drives filter performance by measuring ability of the filters to remove particles down to 4-nm size and ensuring low particle shedding by the filters. Ion exchange resin was found to be a major potential contributor of small particles (7). SEMI C93 measures contamination shedding by ion exchange resin.

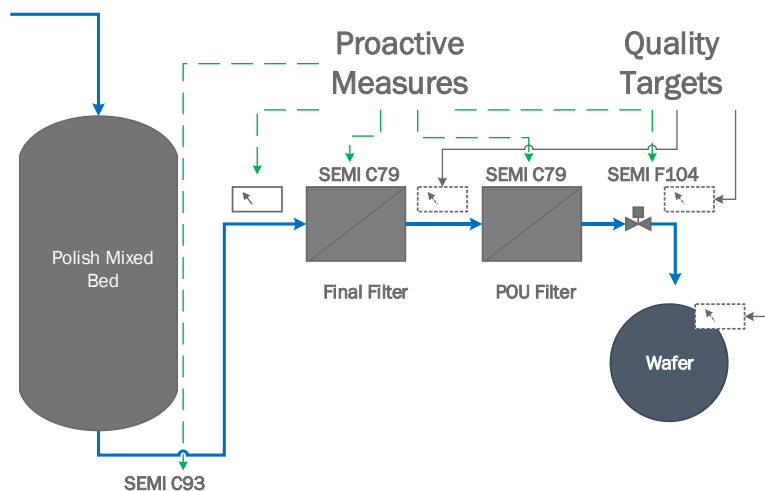


Figure 2. SEMI Standards Involved in Proactive Particle Management

Methodology of Proactive Particle Management

SEMI Standards

Use of the above standards is one of the ways to mitigate particle occurrence. The results section provides typical data of the application of those standards, which also serves examples for the industry end users when defining company specific targets for the quality specification. Combination of clean resin, high efficiency clean filters, and low shedding critical components of the UPW delivery system are key for reaching high manufacturing yield targets.

IRDS Studies – Round Robin Test for Advanced Particles Metrology

In addition to the above risks, YE IRDS team conducted additional studies to help to further contamination control. There are several instruments for particle measurement in UPW that have been recently developed but have not been fully commercialized. To assist those technology providers in qualification of their devices and methods YE IRDS team initiated round robin study together with seven leading semiconductor manufacturing

facilities in the USA. The facilities included Intel, Samsung, Micron, GlobalFoundries, and IMFT.

The following advanced particle metrology was used in the study:

- sTPC by Kanomax FMT
- nano-Spotlight (Focused Aerosol Deposition – FAD) by Kanomax FMT
- 10nm SEM with Centrifuge preconcentration by Kurita
- APC (acoustic particle counter) by Uncopiers
- ICPMS by Air Liquide Balazs Labs

Each method has different mechanisms for analysis and different limitation. Ability to interpret results and validate accuracy of the readings is difficult at the size of the particles of concern. The purpose of the study was to compare all the data and identify opportunities for application of the different methods.

Due to logistical reasons, only two sites were tested by APC and 10nm SEM. APC data is presented, while 10nm SEM requires more sites to be visited before data is available for publication. The data is presented anonymously to preserve information security.

IRDS Studies – Particle Precursors

In addition to the focus on traditional particles (suspended solid matter), there is a concern that a single nanometer size particle can be formed from dissolved compounds (particle precursors) with relatively high molecular weight. This concern was triggered by the earlier reports (see Figure 3) from Kanomax FMT, experimenting with their sTPC instrument.

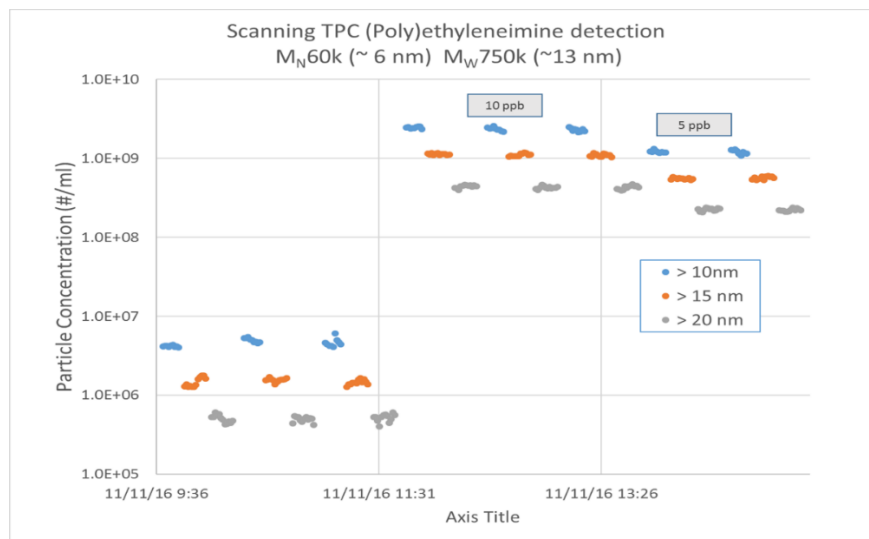


Figure 3. Effect of High Molecular Weight (HMW) Organics to Particle Formation (2)

The risk of particle precursors is particularly high for the following reasons:

1. High probability of occurrence. There is data (7) collected as part of the SEMI C93 analysis suggesting particle precursor leach from the ion exchange resin.
2. Silica is known to form polymeric structure and even colloidal particles by ion exchange resin (2). Those contaminants are also difficult to remove in the UPW polish system (2).
3. Filters are not designed to remove dissolved compounds.
4. Colloidal silica is known to be particularly difficult for filtration.

5. There is no online organic speciation or sensitive enough silica analyzer to address detectability problem of the particle precursors.

What was not clear was the severity of the impact of the precursors. YE IRDS team has conducted deposition study dispensing HMW Organics on bare silicon wafer. The material selected for this study was poly(sodium 4-styrenesulfonate), a polymer widely used in the manufacturing of ion-exchange resin.

First, preliminary experiment, was conducted at CT Associates laboratory, using 1” wafer. UPW containing 1E8 particles per milliliter of the 70,000 and 1,000,000 dalton poly(sodium 4-styrenesulfonate) material solution was dispensed on the wafer, which was subsequently dried under clean hood. Dry wafer was carefully protected from environmental contamination. The equivalent mass concentrations for these solutions were 0.166 and 0.012 ppp respectively. 100 nm silica tracer particles were included in the mixture at a concentration of 1E5 per mL to aid in surface locating during the SEM analysis. It was tested using a Hitachi SU8230 field emission SEM. SEM image of the tested wafer was compared to that of the clean one.

Second experiment was conducted at Screen-SPE facility in Japan, mimicking conditions of the semiconductor manufacturing process, using an advanced single wafer processing tool. The wafers exposed to the 1,000,000 dalton poly(sodium 4-styrenesulfonate) material solution was dried and tested under SEM.

Results of the Development Work for Proactive Particle Management

SEMI Standards – SEMI F104

While new nanoparticle counting metrologies are becoming available, the optical particle counter (OPC) that use laser light scattering continue to be the dominate and most widely-deployed particle counting instruments. For this reason, the OPC continues to be the instrument of choice for SEMI F104. It is known that filtration efficacy can be influenced by particle concentration in the liquid however the primary risk of particle shedding comes from components that are downstream of filtration. High surface area materials such as tubing and components that are mechanically active during their operation are of particular concern.

Historically, in the absence of filtration, the particle concentration “Power Law” as presented in Equation 1 has held. By taking advantage of the power law and establishing particle budgets, particle concentration requirements established within the IRDS roadmap can extrapolated to larger particle sizes and concentrations that are measurable by OPC’s.

(9) A power law slope of 3 is used in this methodology.

$$f(x)=k*(1/d^n) \text{ where:} \quad [1]$$

k = Cumulative number concentration (#/mL) \geq d (nm)

d = channel size (nm)

n = slope exponent (typically ranges from 2.5 to 3.5)

One significant finding from the study was that OPCs from different manufacturers provided high-corelated rinse data over the tests. Figure 4 shows the rinse results from two valve manufactures using three different particle counters.

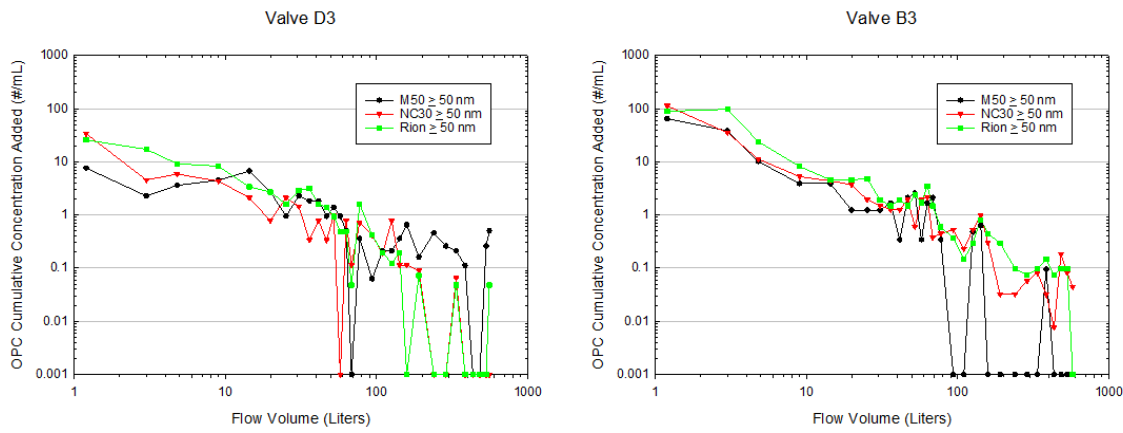


Figure 4. OPC Comparison of UPW Rinse Data for two Valves at > 50 nm (9)

Another significant finding was that under static rinse conditions, all the components tested (tubing, valves and regulators) would reach the budgeted level of particle shedding well within typical process start-up times (Figure 5).

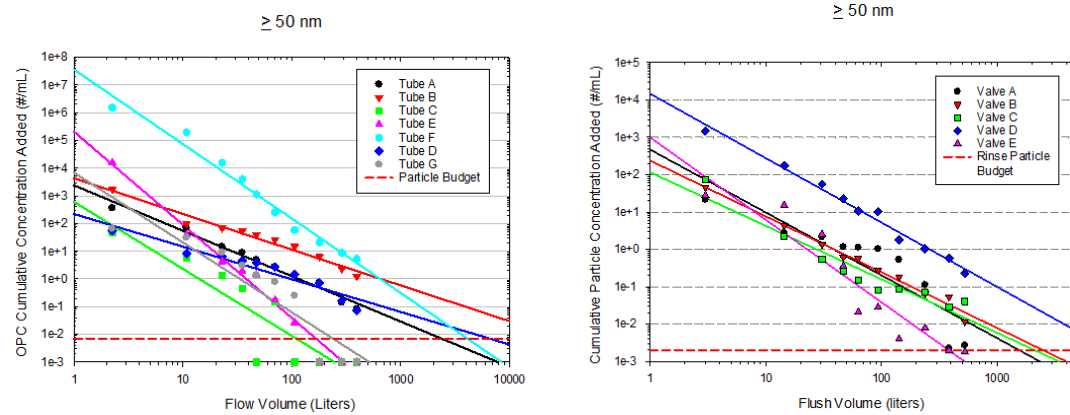


Figure 5. Tubing and Valve Rinse Results for Particles ≥ 50 nm (9)

Under cycling or dynamic conditions, particle shedding can become more problematic. A total of 7 valve types were tested during the study. Only four of the 7 valve types tested were able to comply with the particle shedding requirements established by power-law modeling of the IRDS requirements. Figure 6 presents the particle shedding results of two of the at-risk valves.

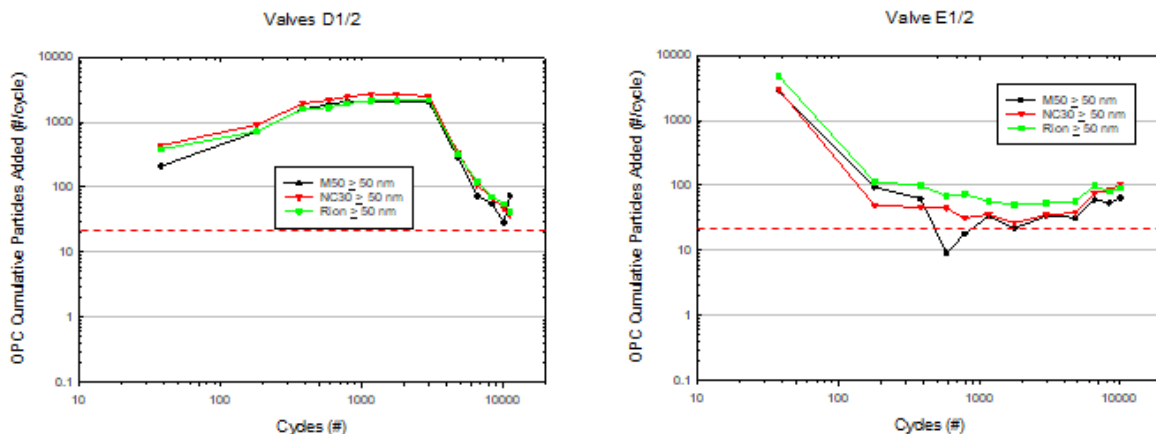


Figure 6. Particle Shedding from Two Valves Under Cycling Conditions (9)

In this study, the components having the highest particle shedding risk were pressure control devices operating with changing inlet pressure conditions. It can be seen that inlet pressure changes in the range of ± 10 psi (0.7 bar) can result highly elevated level of particle shedding and possibly particle generation from the device. The need for improved particle performance of these devices was quite evident.

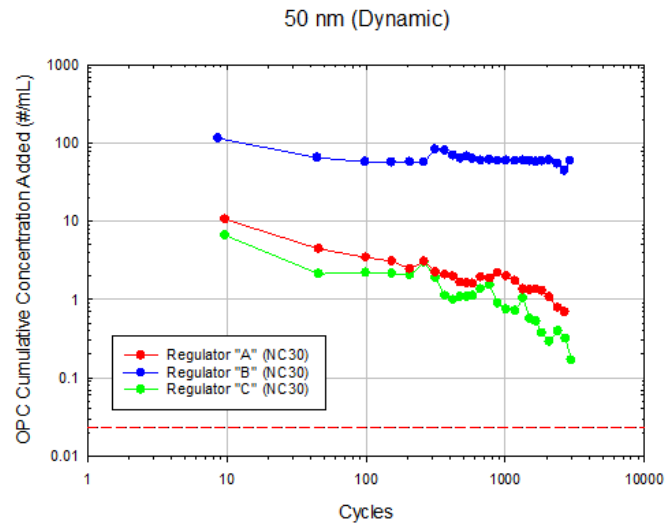


Figure 7. Particle Shedding from Pressure Control Devices

SEMI Standards – C79

The retention of critical-size particles and above is essential for the success of the Proactive Particle Control Methodology. If the filters are not capable of retaining a significant portion of the killer particles and larger, then the method cannot be successful. In addition, as the current critical sizes gets smaller, particle shedding from the filter as a particle source becomes more important. In the latest revision of SEMI C79, the method development emphasis was on reducing the particle size distribution of the colloidal silica challenge and measuring particle cleanliness of the filter to 7 nm. A new colloidal silica retention challenge was developed with silica particles as small as 4 nm (Figure 8.)

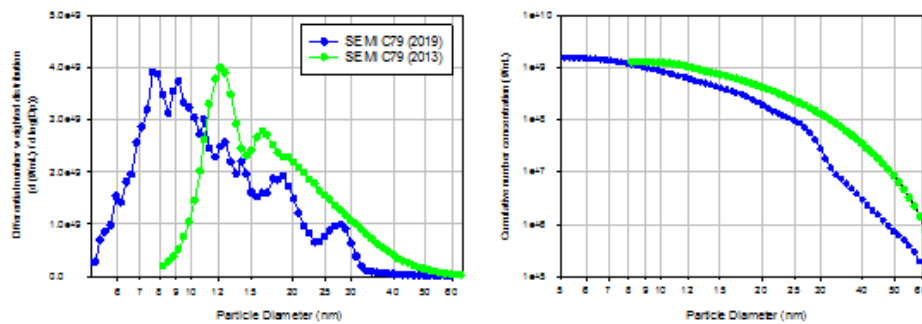


Figure 8. Comparison of Old and New SEMI C79 Silica (10)

A total of 9 filters were tested in the study. Three semiconductor filter manufacturers participated and the tests were run in triplicate. The filters provided were either commercially available filters or filters under development, all targeted toward latest node manufacturing.

A three-day rinse test was conducted on each of the filters prior to measuring the filter's retention. The filter effluent was monitored continuously for particle shedding using a Kanomax FMT Scanning Threshold Particle Counter and a Lighthouse NC30+ 30 nm OPC. The KFMT Scanning TPC was specially tuned to measure 7 nm particles. In addition to the Scanning TPC and NC30+; TOC and non-volatile residue were measured using a Suez Sievers 500 TOC analyzer and a Kanomax FMT NRM respectively. Sample results from the rinse study for each of the filters tested are presented in Figure 9. While only one set of data are presented, the rinse curves were consistent between each of the manufacturer's filters.

A significant observation in the rinse study is seen in Figure 9. When particle shedding is measured using the 30 nm OPC, the rinse-down profiles for all three filter type are very similar and quite low. However, when measuring particles to 7 nm, significant differences become apparent. Most striking is the difference observed with Filter A. In this case, large particle rinse-down was equal to or faster than Filters B or C yet small particle shedding was nearly 3 orders of magnitude higher for Filter A.

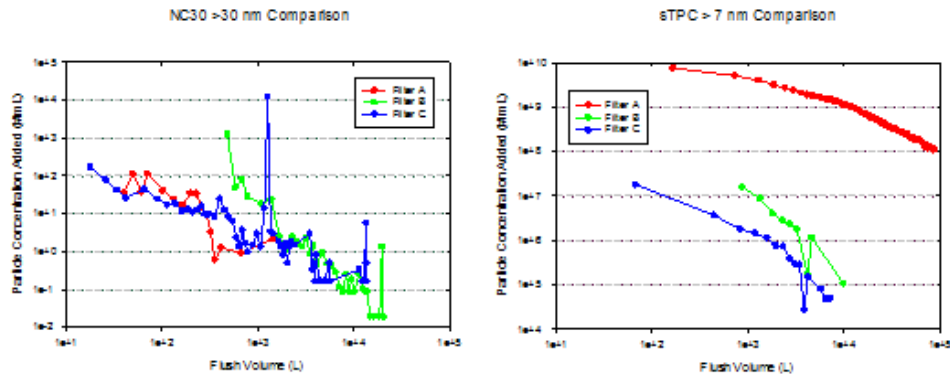


Figure 9. Particle Rinse Data from Three Filter Manufacturers (10)

Filter retention data are presented in Figure 10 for the three filters. Several interesting observations can be made. First, it is evident that at these particle sizes there are multiple particle retention mechanisms that are functional. For example, Filter C primarily retain via classic sieving, whereas Filter B is most retentive particle size of 15 nm.

The second observation is that all the filters lost retention capacity with loading. While this is not a new observation, this is the first data that supports this phenomenon for these size particles. It should be noted that for Filter A, this effect was greater for larger particles.

Finally, while Filter A had the highest particle shedding during rinse, it demonstrated the best retention of the three filters tested. This result strongly supports the benefit of adding the rinse testing to the SEMI C79 protocol.

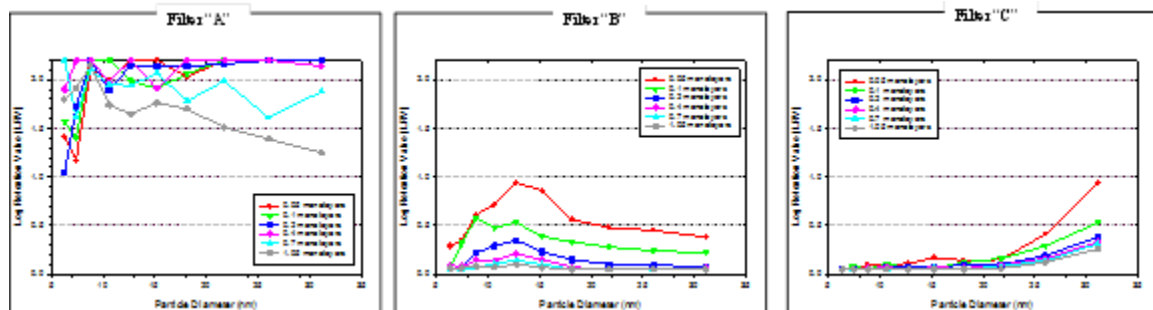


Figure 10. Silica Particle Retention to 5 nm - Log Retention Value (LRV)

SEMI Standards – C93

Ion exchange resin is the single largest surface area component in a UPW system. As such, small particle shedding from these resins present a significant risk to the final water particle quality. Ion exchange resins are also a significant source of what has traditionally been known as non-volatile residue (NVR), particularly during in the early stages of installation. As the critical particle sizes push below 5 nm, the industry is entering a region where particles, particle precursors and molecules, begin to overlap. A particle precursors are defined as a dissolved or suspended nano-material that when the liquid dries could result in a particle of critical size. While distinguishing particle precursors from solid particle is beneficial as mitigation techniques may be different, both present a risk for water quality and must be effectively measured and managed.

In the recent update to SEMI C93, six semiconductor-grade final polish resins were tested for dynamic rinse performance and static extraction analysis. New to this study was the addition of an ultrafine nebulizer configured with a 4 nm condensation particle counter (CPC). The rinse-down of the 6 resins as measured using the ultrafine nebulizer combined with a 4 nm CPC and a Scanning TPC are presented in Figure 11. Both instruments utilize a liquid nebulization approach. By managing the nebulization droplet distribution and the counting method, the instruments allow for segmentation existing particles and particle precursors and dissolved contamination. The Scanning TPC is designed to isolate and measure predominately existing particles in the liquid while the ultrafine nebulizer with a 4 nm CPC captures these particles plus particle precursor and dissolved contaminates. The combination of these two instruments provide useful data to determine when a resin can be released to production and monitoring the resin health during operation. It can be seen Figure 11 that particles ≥ 10 nm rinsed to below the detection limit in roughly 130 liters of flush volume (~ 200 bed volumes) yet continued to shed a significant level of particle precursor after 2000 liters of flushing (3000+) bed volumes.

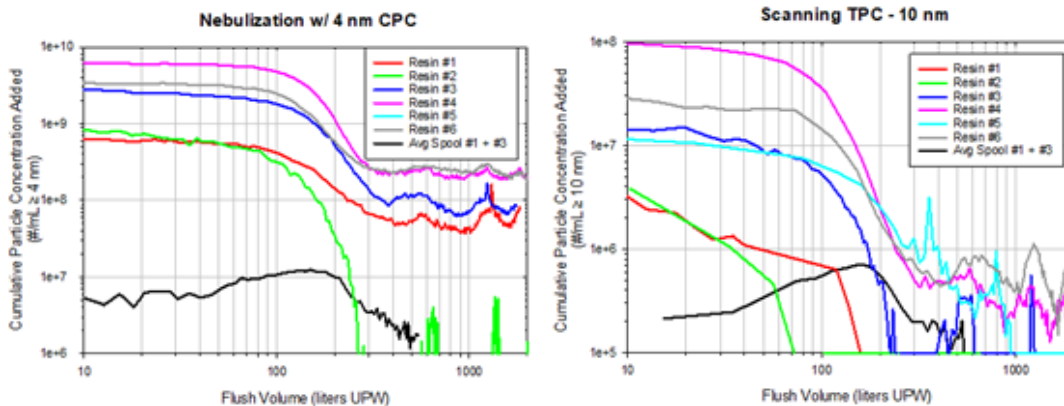


Figure 11. NVR and Particle Rinse at 4 and 10 nm of Virgin Ion Exchange Resins

IRDS Studies – Round Robin Test for Advanced Particles Metrology

	Site 5 IN	Site 5 OUT		Site 5 IN	Site 5 OUT		Site 5 IN	Site 5 OUT
Al			Si			Ti		
Particle Conc. (particles/ml)	564	282		8442	6841		564	423
Median Size (nm)	15	16		37	37		14	14
BED* (nm)	6	6		21	21		6	6
Mg			Zn			Fe		
Particle Conc. (particles/ml)	282	141		705	282		282	0
Median Size (nm)	27	19		23	23		13	0
BED* (nm)	5	5		9	9		4	4
B			V			Cr		
Particle Conc. (particles/ml)	423	0		282	0		73	0
Median Size (nm)	27	0		14	0		14	0
BED* (nm)	10	10		5	5		5	5

Figure X. Representative Result of the Particle Analysis by ICPMS

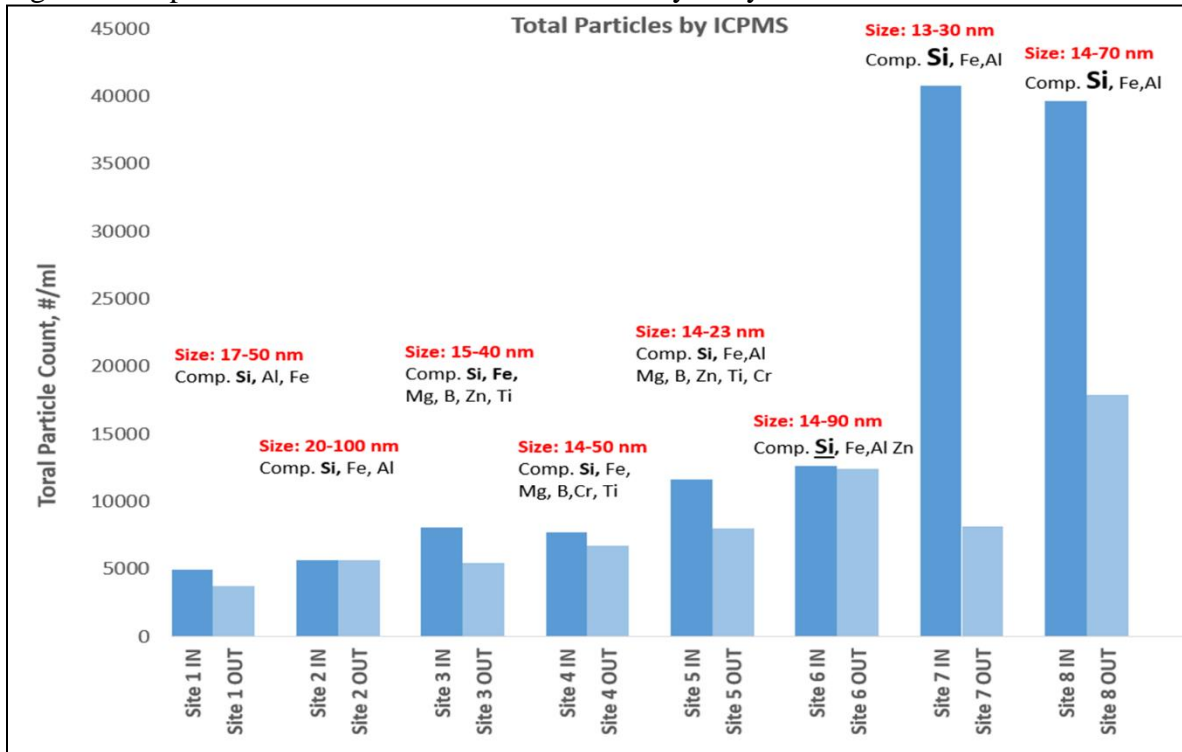


Figure X. Round Robin Study Results of the Particle Analysis by ICPMS

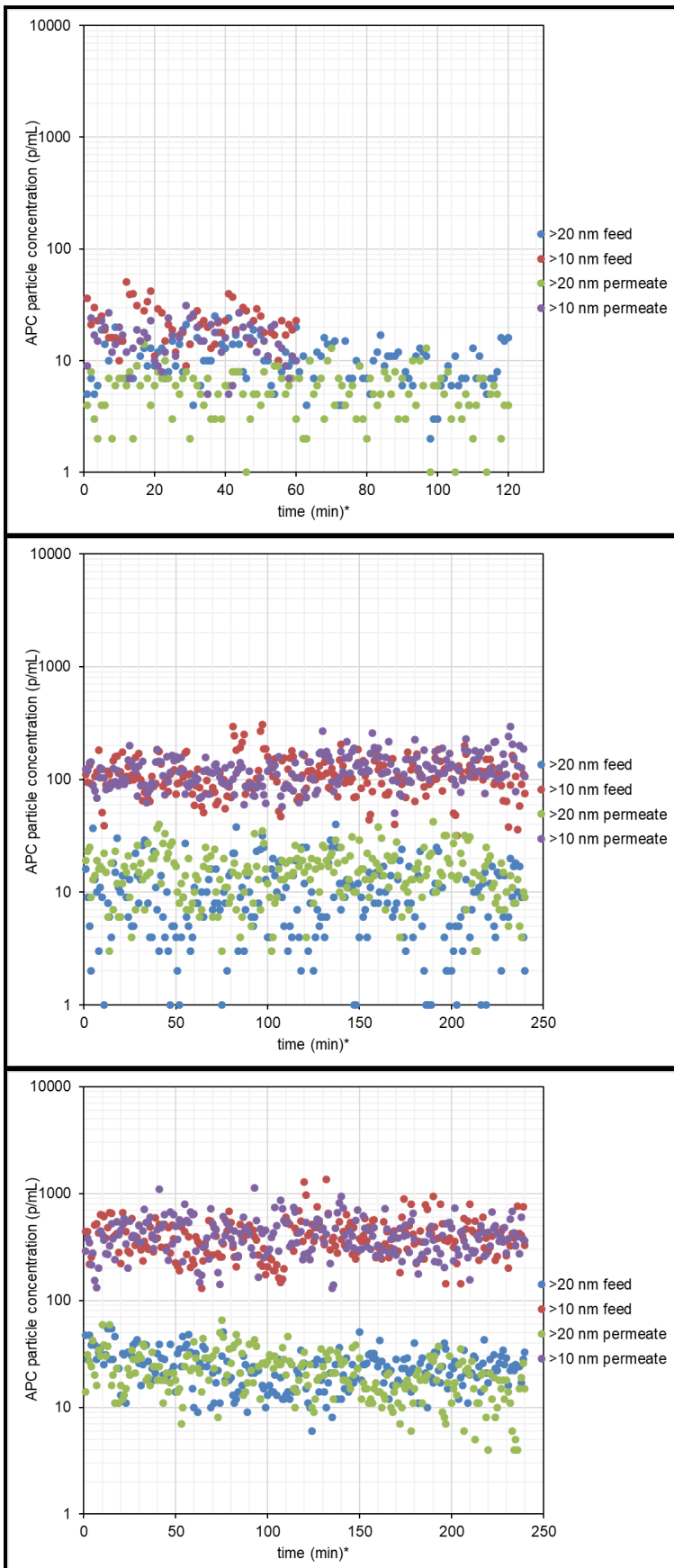


Figure X. Round Robin Study: Typical Results of the Particle Analysis by APC

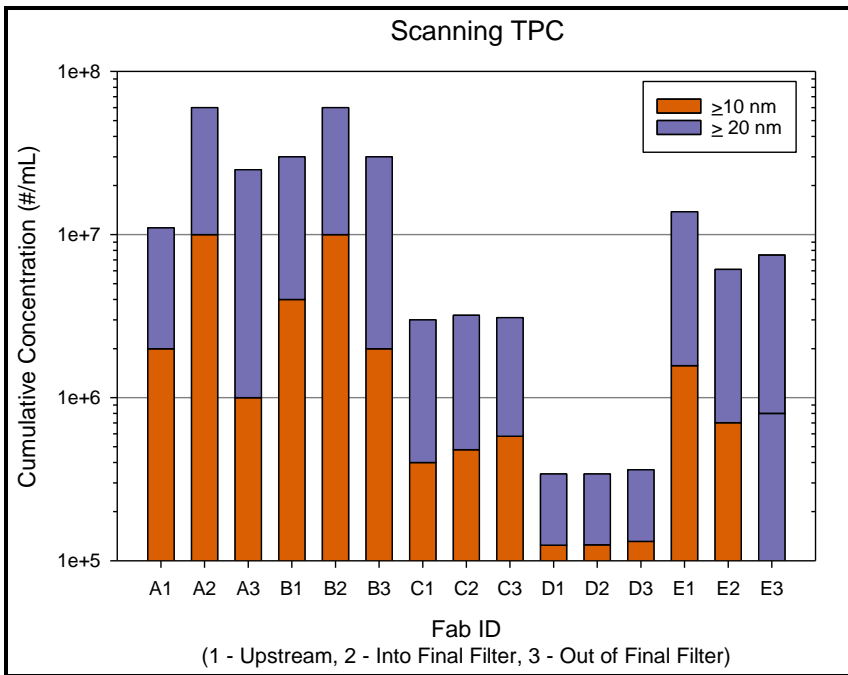


Figure X. Round Robin Study Results of the Particle Analysis by sTPC

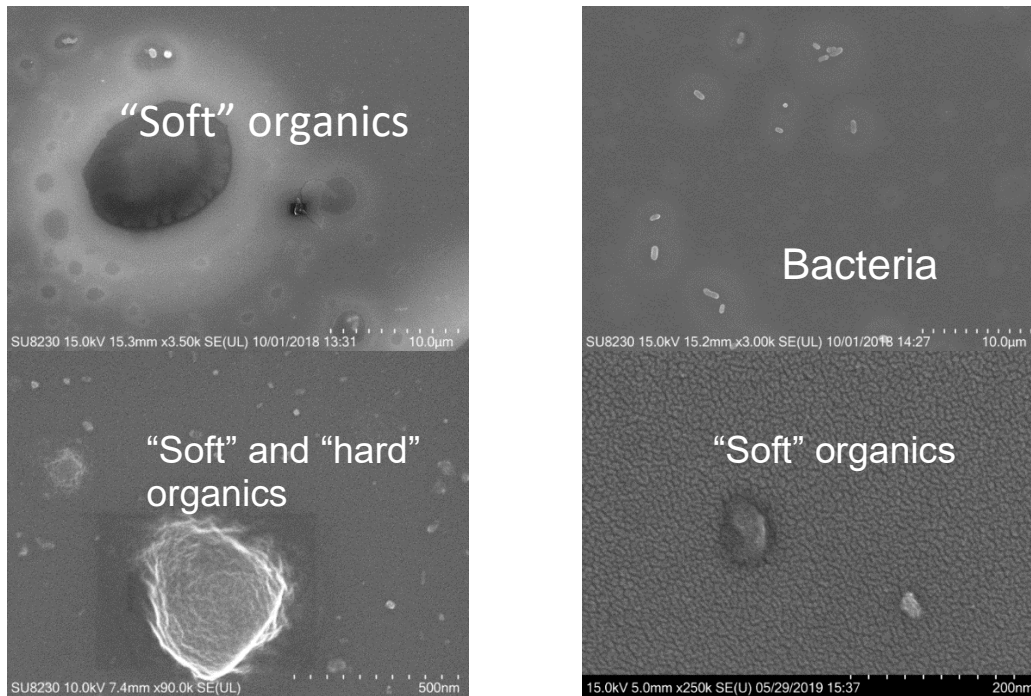


Figure X. Round Robin Study: Typical Results of the Particle Analysis by FAD
 The results of the IRDS Round Robbing study in the figures above indicate that All instruments provided meaningful data.

1. Acoustic particle counter showed that particles in the feed to the final filters is higher than the product. The values are in the range typically observed using laser particle counter (also consistent with literature data (8)) but extending the detectability range into the 10nm particle size.

2. ICPMS results not only showed similar trend of particles reduction by the final filters, this method also provides an interesting information about particle composition.
 - a. Silica particles dominated in all samples. This is particularly concerning, because silica particles are expected to be more difficult for removal by the final filters (2).
 - b. Surprisingly, ICPMS reported boron particles in some of the samples. It is commonly expected boron to occur in the form of an ion. At this point, the boron particles are assumed to be IX resin particles carrying boron ions. It should be noted that ICPMS does not detect carbon, the type of the particles that is expected to represent majority of the particles expected in UPW.
 - c. Other particles elemental composition is generally consistent with what is typically found in UPW.
3. In contrast with the 2 methods above, sTPC did not show correlation between the feed and the product of the respective sites' final filters. This is likely due to the fact that the process of measurement includes a step of nebulizing the sample that may lead to particle formation out particle precursors. This would explain why sTPC particles are not removed by the final filters. This mechanism also explains high number of particles reported compared to other methods.
4. Focused Aerosol Deposition provided ways to collect very small particles, concentrate them, and then deposit on the specimen stab for subsequent SEM-EDX analysis for elemental composition. In contrast with a typical SEM analysis, where the particles are collected on the relatively large pore size filter, this method is not sensitive to the particle size, while the sample collection is much faster.

Different methods provided different results for the same sites
they likely measure different types of contaminants

Application of each method may help to address specific issues of concern

Understanding the effect of different type of contaminants to Yield is important to determine which method/s to use.

IRDS Studies – Particle Precursors

The SEM analysis of the 70,000 dalton HMWO material deposition is shown in Figure 17. 5 – 10 nm particles can be clearly seen. This size range is consistent with both the theoretical size of the molecules and the particle size distribution analysis measured from the stock solution (Figure 18). Additional work on-going to determine if these particles will deposit on the wafer surface when the solution is applied using a traditional spin-rinse process.

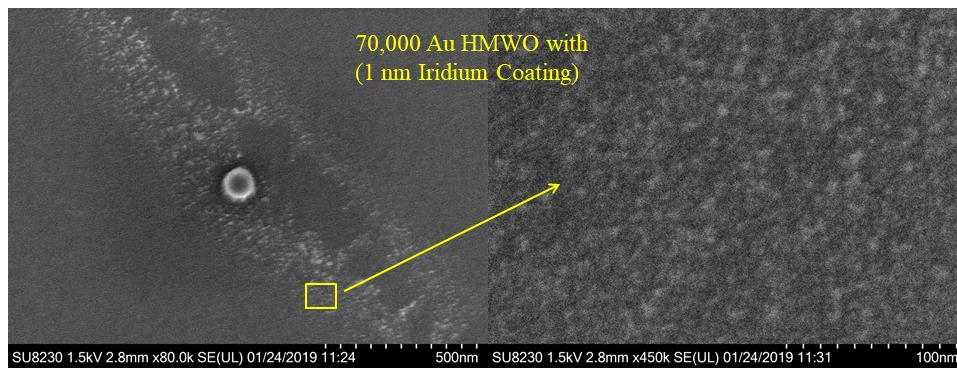


Figure 17. Detection of HMWO on Wafer Surface by FESEM

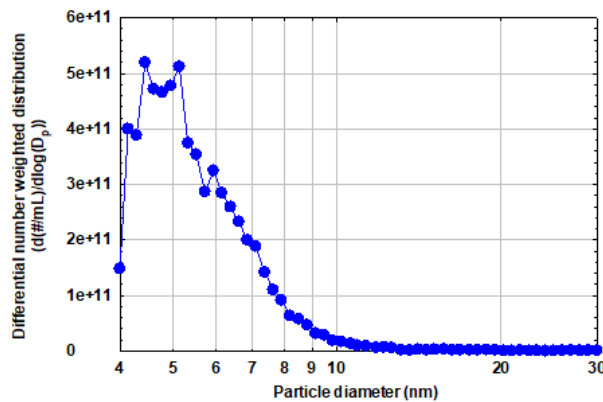


Figure 18. High Molecular Weight Organic Particle Size Distribution

Discussion and Summary

Industry data, as well as recently conducted experimental work suggest that significant collaborative effort is required to ensure high yield of advanced semiconductor manufacturing. Particles coming from the system materials and components must be reduced by improved proactive quality management throughout entire technology chain. Both suppliers and end users should leverage appropriate SEMI Standards to drive both quality control and continuous improvement of the respective technologies.

Ion exchange resin cleanliness should improve, and the quality regularly should be verified by end users. UPW filters should provide tighter particles control, including particle precursors. Application and quality of the regulators used in the liquid delivery systems should be reconsidered to reduce particle generation.

IRDS Round Robin study indicated meaningful capability by the recently developed particle metrology with detection sensitivities approaching the target size of the killer particles. Those instruments have potential for both online monitoring and particle excursion management. However, application of each method should consider the mechanism of the measurement, and as the result, their respective limitations. If target contaminant type and the purpose of measurement are defined those methods can significantly improve the ability to cope with particle challenge.

Acknowledgments

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Complete citations should appear at the end of the text. Use the reference style that is shown below for all references.

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