Managing Semiconductor Manufacturing Risk through Improved Control of Nano-particles in Ultrapure Water

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Abstract

This paper describes the current state of particle detection technology in ultrapure water (UPW) with respect to the needs of advanced semiconductor manufacturing. The International Technology Roadmap for Semiconductors (ITRS) has defined particles in UPW as a critical parameter associated with the risk of wafer defects in semiconductor manufacturing. This risk is driven by the increasing sensitivity of advanced semiconductor manufacturing processes, such as critical dimensional etching, and the limited ability to control particles in UPW and on the wafer. Despite relatively robust filtration capabilities, it is impossible to guarantee high-yield manufacturing without capable UPW particle metrology.

This paper presents a new approach for characterizing “killer” nano-particles. The approach has been adopted by the ITRS UPW Committee and takes into account theoretical deposition physics (diffusion, drying, etc.). A particle depositionstudy is under way to validate the theoretical model.

The ITRS UPW Committee has conducted a benchmarking study to determine the chemical composition of commonly occurring particles in semiconductor UPW systems. The benchmarking study uses a new, experimental device for the rapid collection and agglomeration of sub-50nm particles onto a Scanning Electron Microscopy (SEM) filter. After the particles are collected, their chemical composition is identified by X-ray spectroscopy.

Introduction

The ITRS UPW technical working group [1, 32] has defined particle detection below 30 nm to be one of the more important challenges in UPW technology. The group has also reported that there are no measurement devices currently available that can detect particles at this range.
Particles introduced during semiconductor manufacturing processes can cause a variety of wafer defects, including the following:

- Micro masking for critical dimension etch processes, thus driving the need for smaller particle detection.
- Bridging and gaps in lithographic features, traditionally driving the critical particle size.
- Chemical effects caused by the composition of the particles (such as metals or sodium chloride salts). Such effects can occur at smaller particle sizes than the critical particle size since the defect cause is not only size-driven. Due to the inability to detect particle composition, these effects are only discussed as a theoretical possibility. However, inability to detect the chemical composition of particles could become a critical road block to modeling particle deposition on wafers.

Particles on the wafers can come from a variety of sources. Since wafer cleaning occurs before critical process steps, ultrapure water and chemicals should be considered as important sources of the contamination. Cleaning, and therefore UPW quality, will become more challenging in the future for the following reasons:

- Megasonic cleaning can damage surface structures such as 3D gate structures (to be introduced in the near future).
- Under-etching (a key particle removal step in current technologies) will no longer be feasible with newer technologies, since there will be insufficient material between critical layers.

The lack of suitable particle metrology and particle chemical characterization (enabling the characterization of process variables, such as wafer movement, drying, etc.) has a severe impact on the ongoing development of deposition models.

Killer Particle Measurement

"Killer" particles are defined as those particles with minimum size causing a significant effect on the yield of semiconductor manufacturing. The size of killer particles has decreased as new semiconductor architectures, with ever-smaller line width, are introduced.

Generally, when dealing with particles, a spherical shape or an optically equivalent diameter of the particle is assumed. A previous ITRS publication [32] discussed different particle size definitions. However, a more useful definition of particle size is still undetermined because adequate metrology is not available.

Another important parameter characterizing the particle is its chemistry. Knowledge of the chemistry can improve the possibility of particle detection, can help to prevent particle generation, and can enable effective treatment. The ITRS UPW group conducted the benchmarking study to characterize the typical chemistry of the fine particles occurring in UPW.

ITRS Benchmarking Study

Methodology

The ITRS UPW group conducted a benchmarking study at five different sites. Balazs Analytical Services, Fluid Measurement Technologies Inc. (FMT), and Lighthouse supported the study by contributing their analytical instruments and services. The new device used for the benchmarking study was the *nano*-Particle Collection Device (*n*PCD - formerly known as the Nanalyzer).. The *n*PCD was installed at two sample locations (before and after final filters – some ultrafilter and some cartridge filters) at each site to collect material (particulate and possibly some high molecular weight compounds). Collection times were approximately 12 hours and 24 hours, respectively. Release time was 9 minutes for all samples and the released sample was collected on SEM filters.

To validate the data, two additional filters were placed at the first location, again for 12 and 24 hours before and after final filters. These filters were analyzed at a different SEM lab from the other filters. Initial results from both sets of filters were similar. The benchmarking sites were Intel, SUMCO, TI, IBM Fishkill and IBM Burlington.

Polycarbonate 0.1 µm track etch membrane filters (25mm) were pre-coated with gold and placed into filter cartridges. A control filter was retained at the SEM lab. Two fields on the filters were selected for analysis - one on the edge and one in the center of the filter. Particles were manually counted and selected particles were analyzed by X-Ray Diffraction Spectroscopy (EDS) by the same SEM analyst. Results were not blank corrected.

The goal was to perform EDS on 10 selected particles in each field in the range of approximately 0.5-1 um where possible, and up to 1.5 µ if needed. A control EDS was also performed on a blank area of each filter. The blank was analyzed last. Two images were performed one on the edge and one on the center. All SEM/EDS analysis was performed with a Zeiss EVO 50 with integrated IXRF EDS.

Results

All control filters showed the presence of C, O, and Au only, which is expected. The other samples showed different results depending upon their source.

Representative particle release data is presented in Figure 1. The data demonstrates the ability of *n*PCD to concentrate particles. Figure 2 presents a representative SEM EDS chart indicating the chemistry of the

collected particles. Figure 3 demonstrates the appearance of the particles under SEM microscope. Figure 4 illustrates the chemistry of the EDS analyzed particles.

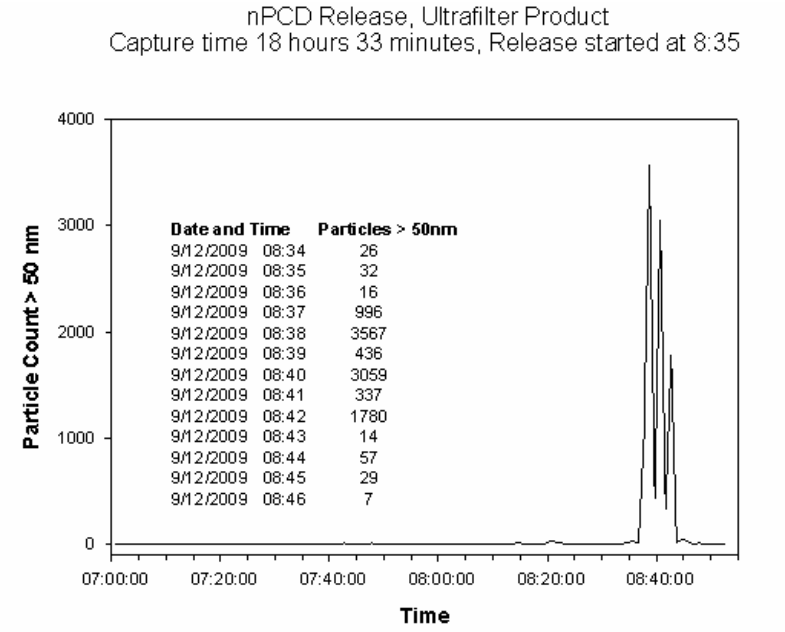
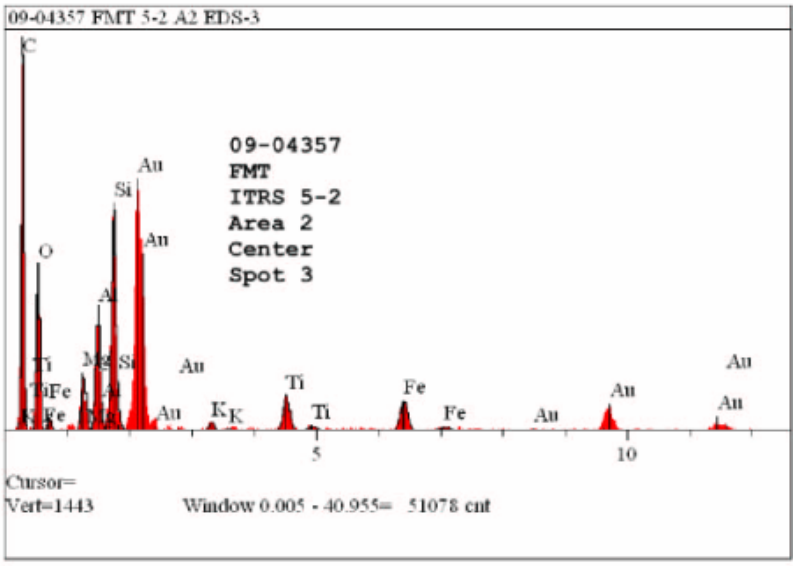


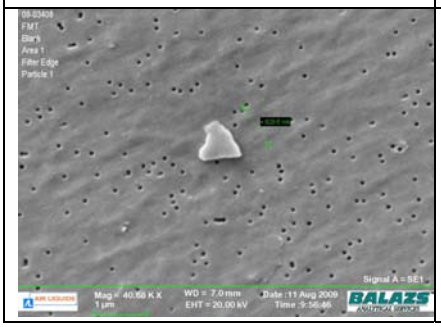
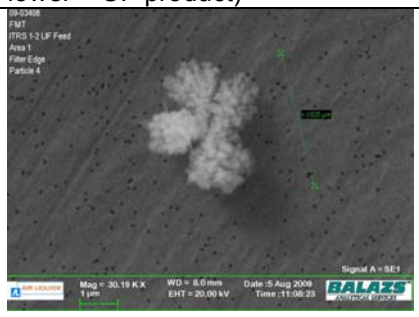
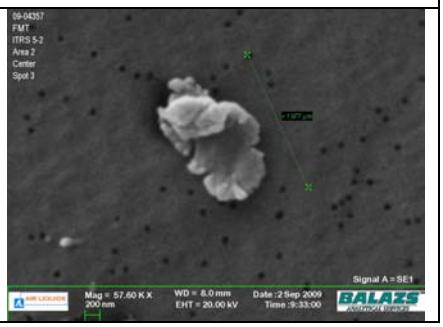
Figure 1. Representative OPC release data.



Elt.	Line	Intensity (c/s)	Atomic %	Conc	Units	Error 2-sig	
C	Ka	151.75	62.574	50.184	wt.%	1.052	
O	Ka	64.86	27.981	29.893	wt.%	0.958	
Mg	Ka	29.04	1.333	2.164	wt.%	0.104	
Al	Ka	62.68	2.287	4.120	wt.%	0.134	
Si	Ka	124.62	4.085	7.660	wt.%	0.177	
K	Ka	4.85	0.121	0.317	wt.%	0.037	
Ti	Ka	26.78	0.700	2.237	wt.%	0.112	
Fe	Ka	25.79	0.918	3.425	wt.%	0.174	
Au	La	0.00	0.000	0.000	wt.%	0.000	
			100.000	100.000	wt.%		Total

kV 20.0
Takeoff Angle 35.0°
Elapsed Livetime 60.0

Figure 2. Representative EDS data of the released *n*PCD particle

Blank particles	Location 1 typical particles (upper - feed to the Ultrafiltration (UF); lower – UF product)	Location 2 and 3 typical particles
		

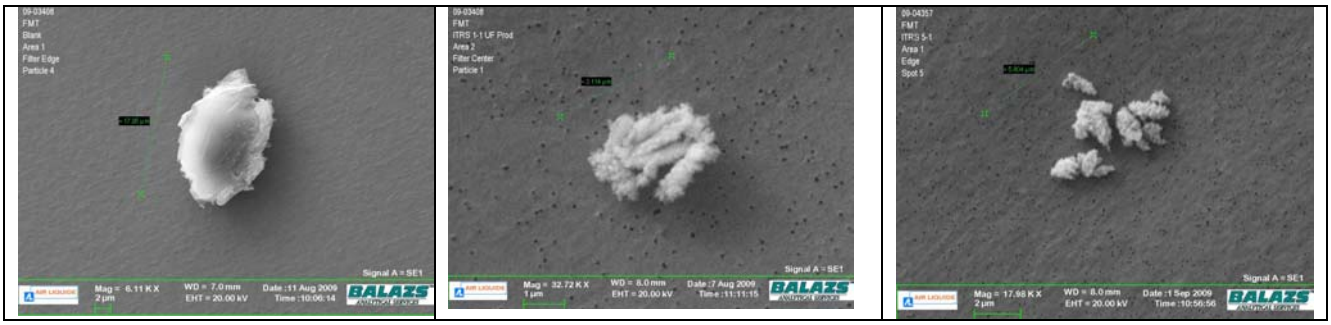
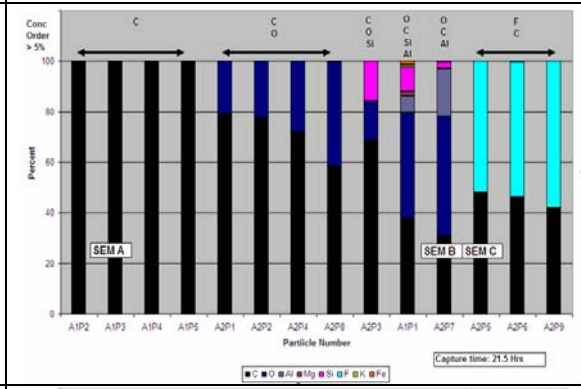
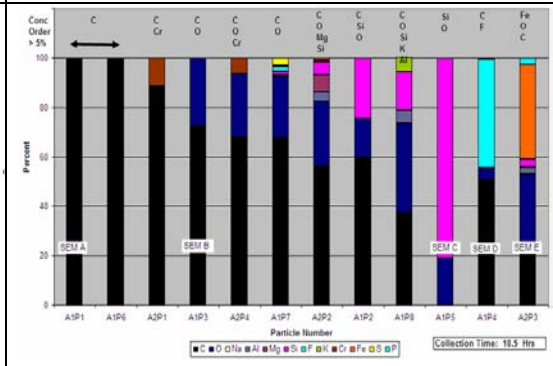
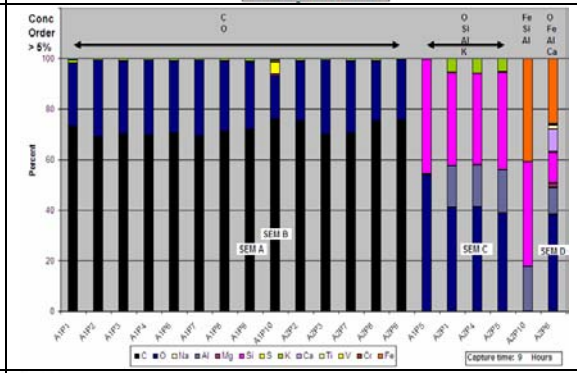
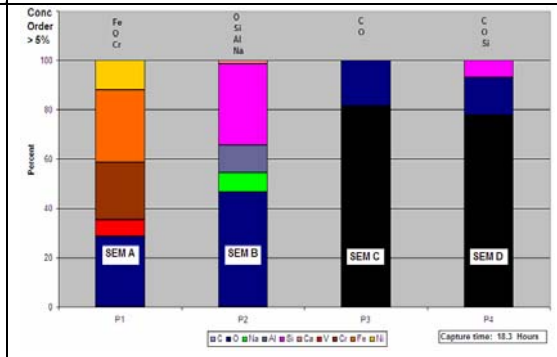
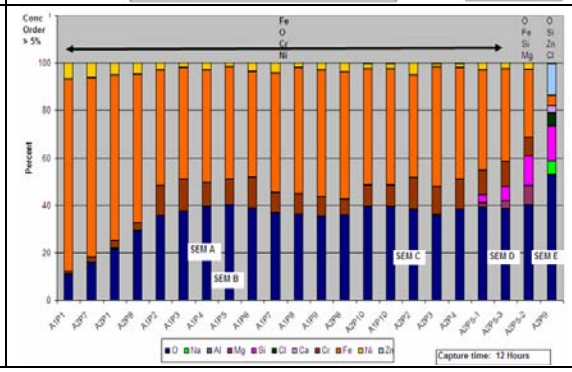
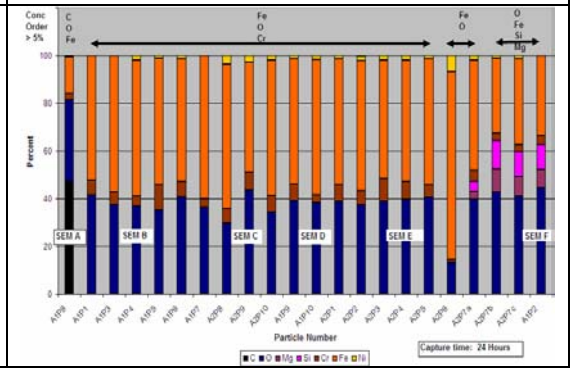


Figure 3. SEM images of representative particles.

Source	Feed to Ultrafilters	Product of Ultrafilters
Fab 1		
Fab 2		
Fab 3		

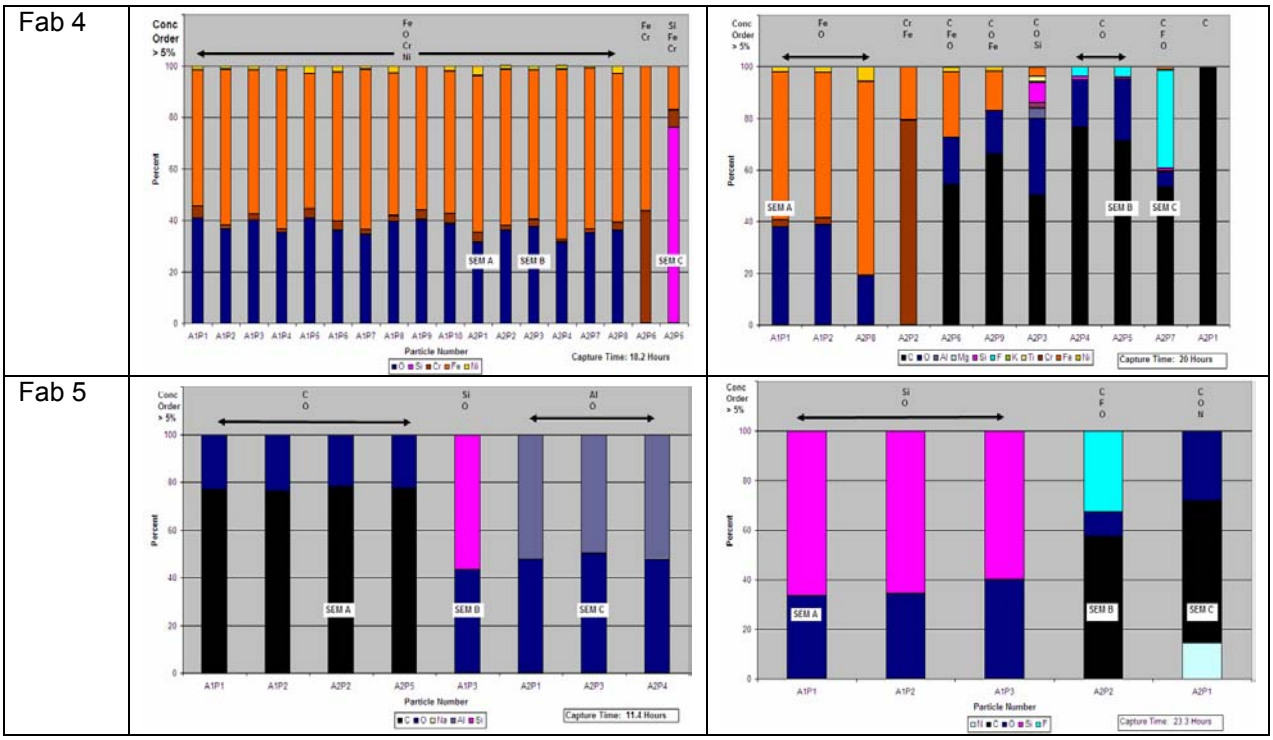


Figure 4. Summary of the particle chemistry. (Filter supply on the left of the diagram; filter product on the right.)

Benchmarking Study Summary

The benchmarking study findings indicate that UPW particles have diverse chemistry. The most common particles contained carbon, iron, aluminum, fluorine, and silica. The following glist describes the probable origination of the particles:.

- A significant portion of the metallic particles probably originated from stainless steel components because they contain iron, chromium and nickel.
- Fluorine particles are common in UPW – originating from the material of construction of the high purity piping.
- Silica particles are commonly found in UPW but were not as common in the benchmarking results, probably because of the limited ability of *n*PCD to concentrate Silica-based particles.

As was expected, the *n*PCD agglomerated particles of different chemistries (not usually found in a single particle). This ability to agglomerate smaller particles could be observed on many of the SEM images. Specific examples are the Fe/Si/Mg particles found in Fab 3 or the C/Mg/Si particles found in Fab 1 (shown in Figure 4 above).

Although it is unclear what the original size of the particles was (before agglomeration), it is important to emphasize the following:

- Baseline Optical Particle Counter (OPC) data was within, or close to, the baseline of the on-line instrument used to monitor water quality. OPC was not able to detect individual particles before agglomeration by the *n*PCD;
- Chemistry of the particles is consistent between the feed and the product of the ultrafilters;
- The overall amount of released particles was similar (same order of magnitude) in both UF feed and product samples (based on OPC release chart and SEM particle counts);
- Three of the photos in Figure 3 show that the individual particles in the agglomerations were smaller than any particles that ultrafilters can remove.

The current UPW roadmap indicates a need to control smaller particles than can be detected by existing OPCs and wafer particle detectors or semiconductor manufacturing will be at risk. The ability of the ultrafilters to control particles smaller than those detected by OPC is not proven.

SEM particle counts of the agglomerated particles measured on the SEM filters are shown in Figure 5, indicating that the UF filters removed particles.

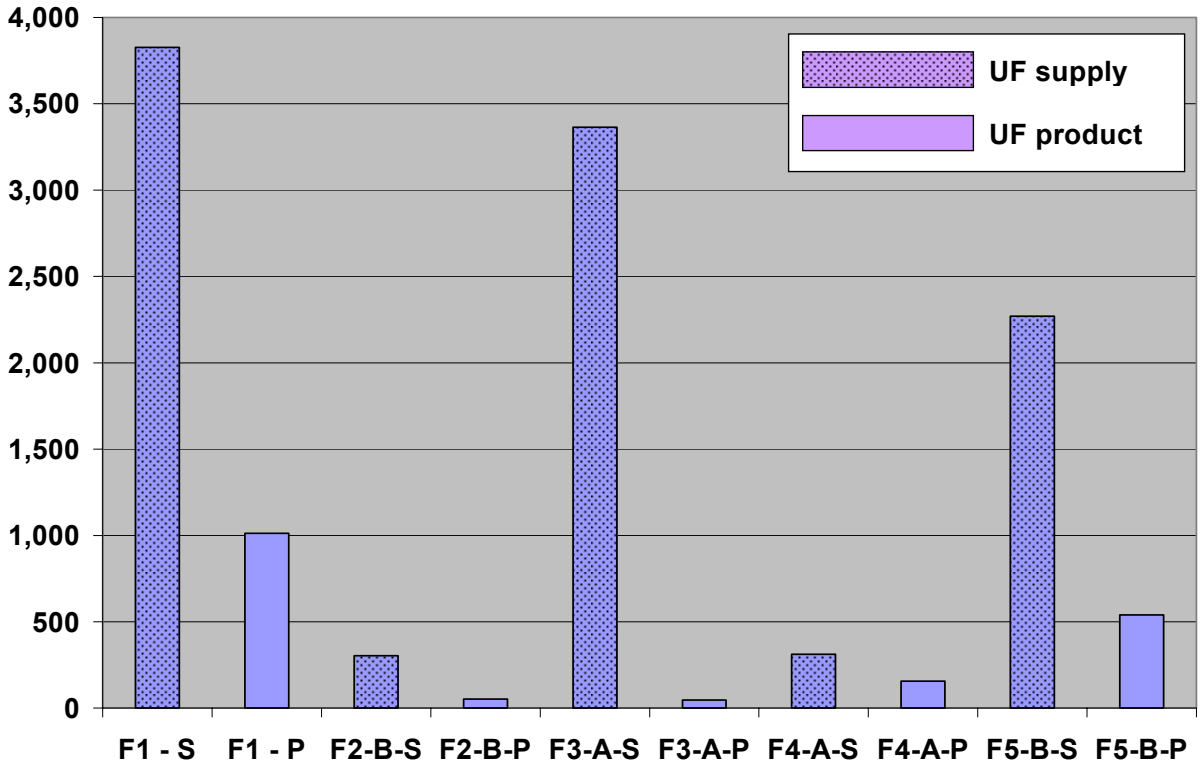


Figure 5. Particles counts estimated by SEM. (Note: Values are an approximation.)

Particle Occurrence

Evaluation of the actual risks associated with particle contamination in high-volume manufacturing environments must consider the probability of occurrence as well as the possibility of particle detection failure. The previous UPW ITRS report [32] provided an overview of the possible causes. However, the results of the benchmarking study confirm that significant concentrations of small particles exist downstream of the final filters. There is also a concern that particles may be generated in the process tools downstream of the final filters. Therefore, given the current limited ability to monitor particles at the critical size, the risk to semiconductor manufacturing is increasing.

Particle Size

The critical particle sizes, as calculated for Flash memory devices, are shown in Table 1 [1]. The equivalent feature sizes for other semiconductor technologies are provided as comparison.

Table 1: Critical particle sizes for different technology nodes. (Source: ITRS [1])

Year of Production	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022

Year of Production	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022
Flash ½ Pitch (nm) (un-contacted Poly)	**54**	**45**	**40**	**36**	**32**	**28**	**25**	**23**	**20**	**18**	**16**	**14**	**13**	**11**	**10**	**9**
DRAM ½ Pitch (nm) (contacted)	65	57	50	45	40	36	32	28	25	23	20	18	16	14	13	11
MPU/ASIC Metal 1 (M1) ½ Pitch (nm)	68	59	52	45	40	36	32	28	25	23	20	18	16	14	13	11
MPU Printed Gate Length (nm)	42	38	34	30	27	24	21	19	17	15	13	12	11	9	8	8
MPU Physical Gate Length (nm)	25	23	20	18	16	14	13	11	10	9	8	7	6	6	5	4
Critical particle size (nm)	**33**	**29**	**25**	**23**	**20**	**18**	**16**	**14**	**13**	**11**	**10**	**9**	**8**	**7**	**7**	**6**

There is some debate as to the validity of the assumption that the critical size is ½ of the feature size. In future roadmaps, more aggressive assumptions are expected. For example, during ITRS discussions in San Francisco (2009) it was reported that:

- For epitaxial processes, particles of 1/200 of the size are critical.
- In chemical deposition (CD) vias, the percentage of particles causing yield loss is more relevant than a single physical dimension (such as particle diameter). Designing redundant vias adds complexity to the calculation models for acceptable defect density.

Particle Concentration

Another important parameter in particle detection and control is the minimum concentration of particles that may affect wafer manufacturing. The ability to detect small particles is not enough; they must be detected at sufficiently low concentrations. The calculation of the critical particle concentration was presented in the previous ITRS UPW report [32]. The report described a new approach to estimate the minimum allowed concentration of particles in water. In the Front End Processing (FEP) chapter of the ITRS roadmap, maximum acceptable particle concentrations on the wafers are provided. As well as yield assumptions, feature, cell, and die-size are incorporated into the ITRS models..

It is important to understand how these values can be correlated to the particle numbers in the UPW chemical-cleaning solutions. (Note: UPW used as dilution for chemicals may contribute particles causing more damage to the wafers in the cleaning process than from direct contact.). The model with the best results is based on a two-step (or two-mechanism) approach [33]. The basic assumption is that after drying, a layer of water/process solutions remains on the wafer, depending on the drying method. This layer can vary from 10 nm (after Marangoni drying) to 2000 - 3000 nm (for conventional spin drying). During cleaning, particles from the solution will diffuse onto this remaining layer depending on the particle size and liquid characteristics driving the diffusion coefficient.

Figure 6 is a particle deposition model based on the following assumptions:

- Particle diameter in nm: 200
- Diffusion coefficient incm2/s: 2.14E-08
- Evaporation thickness in nm: 2000 (spin rinse dryer with high rotation)
- Particle concentration in liquid in #/Liter: 200
- Probability of particle attraction in percentage: 10% (Note: Factor can range from 0 - 1 and characterize the mainly electrostatic attraction, i.e. is equivalent to the % of particles which remain in the relevant boundary layer.)

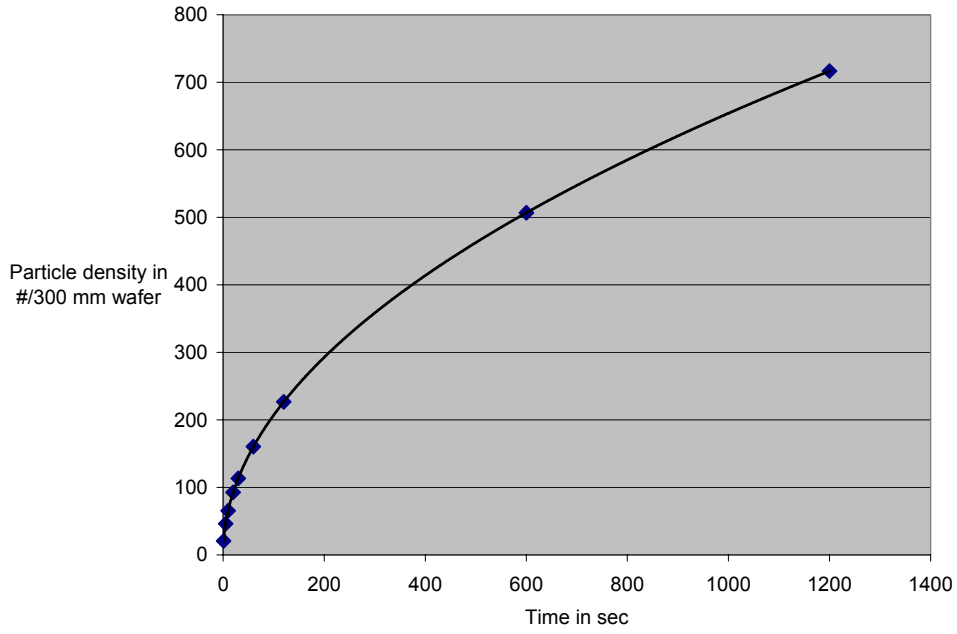


Figure 6: Results of the particle deposition model calculation.

The model represented in Figure 6 assumes an indefinite reservoir of particles. Therefore, depending upon the equipment design, the curve is capped at a certain level. Note: This is only relevant for large processing times. Since the processing times are only in the tens of seconds to a few minutes range, this was not considered relevant, but needs to be considered in further testing.

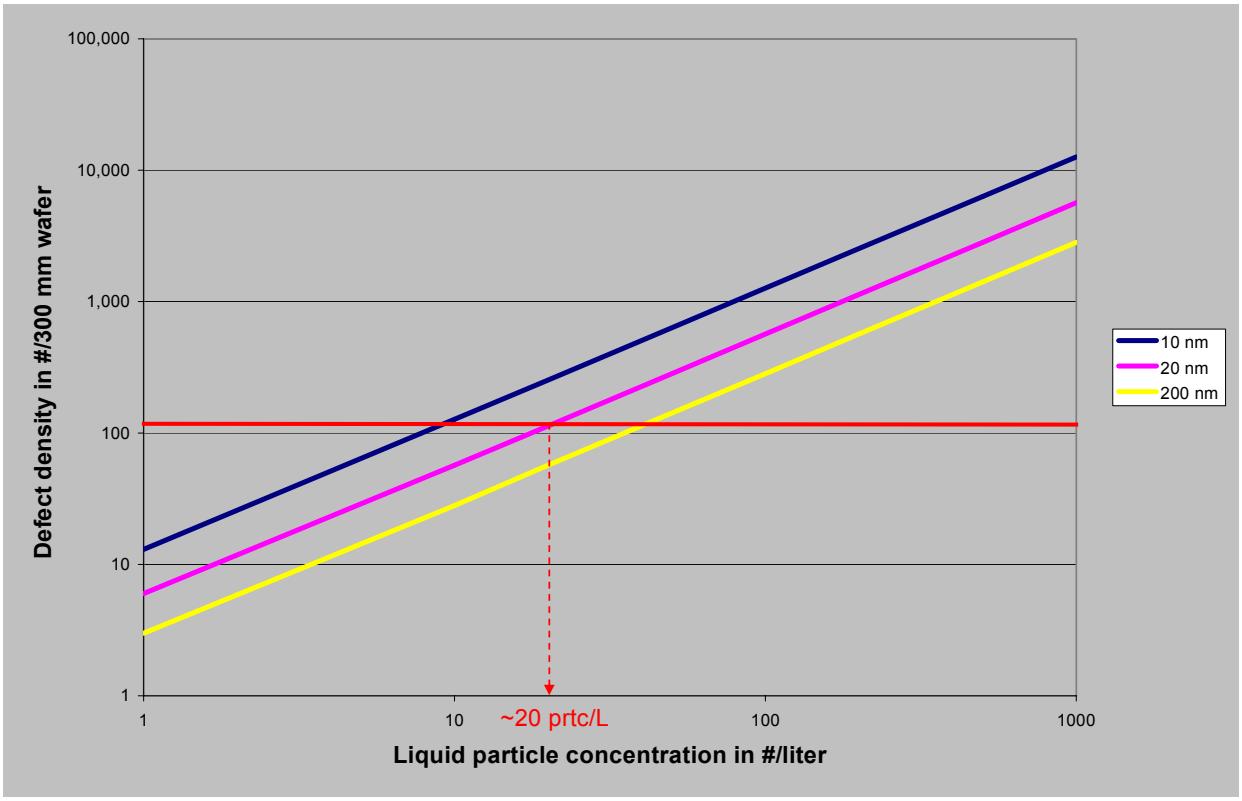


Figure 7. Particle concentration in water estimated by the model shown in Figure 6 (corresponds to 2011 ITRS value of acceptable level of defects on the wafer, 105 part/wafer)

Based on the results indicated in Figure 7, the level of particles required to support the ITRS needs in 2011 is approximately 20 particles at 20 nm per liter.

Current Particle Removal Techniques

The previous report [33] provided a summary of existing particle removal technologies. Although reverse osmosis and nano-filtration provide filtration ratings far beyond the needs of advanced semiconductor processes, they cannot guarantee complete particle removal due to the defects in the membranes. Microfiltration and ultrafiltration technologies are considered to be more robust, providing more efficient particle removal.

The data reported by the ITRS benchmarking study indicates that a significant concentration of particles downstream of the UF system is still possible. It is also possible that all (or a majority) of the detected particles are ultra-fine particles (smaller than the rated pore size of the UF). However, the ability of ultrafilters to efficiently control particles smaller than those that can be detected by OPC is not proven. Therefore, along with the need for new particle detection devices, there is a need to focus on particle treatment in UPW (both operational and technology measures). Operational measures may include timely filter change-out or integrity tests. Technology measures may require development of finer, and higher efficiency, filters. Recent development with cartridge filters capable of treating particles down to 10 and 20 nm [36, 37] is consistent with the need, but it is still unclear whether or not these new filters are sufficient to mitigate the risks. Improved system design and materials of construction should help to reduce the number of particles challenging the final filters. Metal particles, even of very small size, present a high risk to the semiconductor manufacturing therefore stainless steel components should be eliminated in the polish loop of UPW systems **.**

Metrology: Particle Counting Methods

The following section provides updates to a particle technology survey which was presented as part of the previous report [32]. The following technologies were examined:

- SEM
- Dynamic light scattering.
- Laser Diffraction.
- Laser Interferometric Detection.
- Laser Induced Breakdown Detection.
- Capillary Hydrodynamic Fractionation.
- Acoustic Attenuation Spectroscopy.

Table [3] of that report summarized the various advantages and disadvantages of each of the techniques.. The report highlighted the fact that the established practice of extrapolating particle count data based upon an assumed power-law conversion was questionable, especially as the particle size of interest had become more and more distant from known data points. The work concluded that techniques with sufficient sensitivity were still commercially unavailable and that to enable effective semiconductor manufacturing at 45 and 32nm line widths, more advanced particle counters were urgently required. In addition to sensitivity to smaller particles (10nm – 50nm range), the identification of chemical composition was also cited as being of comparable importance.

A description of the following techniques is included in this paper because new or promising technology now exists for them:

- Induced Grating Spectroscopy
- Enhanced Particle Collection Device (PCD) to augment SEM
- Digital Imaging

Induced Grating Spectroscopy

In Induced Grating (IG) Spectroscopy, a diffraction grating is created, based upon entrained particle concentrations, by applying an ac voltage on an electrode array which is immersed in the sample volume.

Dielectrophoresis draws particles toward the electrode array forming a particle density grating. When a laser beam strikes the grating at a specific angle of incidence, it gets diffracted and the diffracted light is detected by a sensor. When the ac voltage is turned off, the dielectrophoresis effect ceases and the particles start to diffuse away from the electrode array. The decaying intensity of diffracted light is a function of time. Using algorithms, this time-dependant behavior can be converted to sample particle size and distribution information.

Induced Grating (IG) Spectroscopy	
Disadvantage	Advantage
• Low sample volume. • Measurement time.	• High sensitivity. • Refractive Index independent.

SEM Analysis and capture time

Grab sampling and subsequent measurement using SEM still remains the fundamental method for particle counting and analysis in UPW, and the technique exhibits many advantages. For example particle shape information, coupled with subsequent EDS/EDX analysis, can provide insight on the source of the particles. However, the long capture periods necessary for statistically valid samples are a fundamental disadvantage of SEM techniques. The completed benchmarking study, using the *n*PCD, has yielded the significant results described in this paper.

Advantages and disadvantages of *n*PCD

*n*PCD Sampling and SEM	
Disadvantage	Advantage
• Cross-contamination. • Inability to detect particles of specific size. • Possible selective preference for concentrating specific chemistries of the particles.	• Short sampling time for statistical valid samples. • Ability to concentrate very small particles (probably at the range of 1nm). • Large sample volume.

Digital Imaging

Digital imaging technology employing relatively large photo-detector arrays, coupled with image analysis algorithms, may provide significant improvements in particle size sensitivity as well as rudimentary morphology. The technique is similar to conventional laser-based OPCs in that suspended particles are directed through a flow capillary to interfere with an incident laser light source.[Needs a reference number] Unlike traditional OPCs, where the light scattered by particles is collected and focused onto a single or small number of photodetector elements, digital imaging technology employs a large detector array consisting of many thousands (or even millions) of elements upon which a two-dimensional area within the flow capillary is imaged. Specialized signal-processing electronics and software algorithms are required to extract particle count and size information from the background noise. In addition to sensitivity improvements, noise discrimination techniques may be used to digitally filter out molecular background scattering as well as high-energy electromagnetic radiation (both primary sources of “false counts” in conventional OPC instruments). In fact, such effects have been the primary limiting barriers to further improvements in conventional OPC technology.

Advantages and disadvantages of Digital Imaging

Digital Imaging	
Disadvantage	Advantage
• Processing speed current limits sample measurement flow rate.	• Enables sensitivities lower than current 0.05um limits. • Can be applied to both UPW and liquid chemicals. • Particle sizing in addition to absolute sensitivity provides distribution data. • Higher sensitivity than direct detection OPC methods as noise is spread out over thousands or millions of detector elements. • Noise discrimination against molecular background scatter, high-energy electromagnetic radiation and scattering from flow path boundaries.

Technology Summary

Tables 2 and 3 summarize the current and potential metrology technologies.

It is still uncertain which, if any, of the advanced techniques may eventually be effective for practical applications in UPW. Even more advanced methods than the ones listed above may be required for monitoring critical particle size and identifying particle chemical composition in the 10-50nm size range in order to support semiconductor manufacturing at 45 and 32nm line widths.

The extrapolation of particle count based upon an assumed power-law conversion is now thought to be questionable, especially as the particle size of interest gets smaller and more distant from known data points. This, in addition to sizing and count inaccuracies resulting from different optical geometries and refractive indices, renders the particle metrology gap ever wider.

Table 2: Summary of current and future metrology technologies.

Technology	Applicable for					
	UPW	Chem	Slurries	Gas	Air	Wafer
Laser Light Scattering (Direct Detection)	50nm (30nm in lab)	65nm (50nm in lab)	50nm	100nm (50nm)	100nm (50nm)	NA
Digital Imaging	30nm	30nm	?	?	?	NA
Induced Grating Spectroscopy	1nm	1nm	?	NA	NA	NA
Condensation Nucleus counter (CNC)	NA	NA	NA	2.5nm	2.5nm	NA
Dynamic Light Scattering (Brownian motion)	NA	NA	1nm	NA	NA	NA
Laser Diffraction	NA	NA	1…5 nm	NA	NA	NA
Laser Interferometric Detection [21]	10nm	10nm	NA	NA	NA	NA
Laser-induced Breakdown Detection [22]	20nm	20nm	20nm	NA	NA	NA

Technology	Applicable for					
	UPW	Chem	Slurries	Gas	Air	Wafer
Surface Scanner	NA	NA	Scratches	NA	NA	28nm
Non-volatile Residue Monitor (TPC)	20nm	NA	NA	NA	NA	NA
Sampling &SEM	20nm	20nm	NA	NA	NA	NA
Capillary Hydrodynamic Fractionation	NA	NA	10nm	NA	NA	NA
Acoustic attenuation spectroscopy	NA	NA	5nm	NA	NA	NA
Using ICP-MS as particle counter	Detection limit	Detection limit	NA	NA	NA	NA
Using VPD ICP-MS as particle counter	Detection limit	Detection limit	NA	NA	NA	NA

Table 3: Advantages and disadvantages of different metrology technologies.

Technology	**Disadvantage**	**Advantage**
Laser Light Scattering (Direct Detection)	• Depends on refractive index; • Long counting periods for statistical validity; • Background counts.	• On-line monitor
Condensation Nucleus Counter (CNC)	• Maintenance; • No species/composition identification; • No size information.	• Low background count
Dynamic Light Scattering (Brownian motion)	• Depends on refractive index; • High concentration for small particles(10^10 particles per mL for 100nm PSL in water).	• Particle size distribution
Laser Diffraction	• Background count	• Particle size distribution.
Laser Interferometric Detection [21]	• Depend on refractive index (see light scattering).	• On-line monitor.
Laser-induced Breakdown Detection [22]	• Depend on refractive index; • Background counts.	• Particle size distribution.
Surface Scanner	• Depend on depostion; • Limited to blank wafers.	• Actual contamination.
Non-volatile Residue Monitor	• Unable to differentiate materials detected.	• Low detection limit.
Sampling &SEM	• Cross contamination; • Long sampling periods (potential to mitigate with PCD); • Labor intensive and expensive.	• Particle identification.
Capillary Hydrodynamic Fractionation	• Background counts.	• Particle size distribution.
Acoustic attenuation spectroscopy	• Background counts.	• Particle size distribution.

Technology	Disadvantage	Advantage
Using ICP-MS as particle counter	• Metals or several non-metals.	• Speciation analysis.
Using VPD ICP-MS as particle counter Induced Grating Spectroscopy	• Metals or several non-metals. • Low sample volume • Measurement time	• Speciation analysis. • High sensitivity • Refractive Index independent
Digital Imaging	• Processing speed current limits sample measurement flow rate.	• Sub 0.05um limits. • UPW and Chemical. • Particle sizing • Noise discrimination against molecular background scatter.

Summary

The ITRS UPW benchmarking study results, using the *n*PCD, indicated the presence of nano-particles in UPW. Although the absolute size of the particles could not be confirmed by the method applied, there is a significant risk to semiconductor manufacturing from these particles, confirming the urgency of improvements in metrology and treatment technology for UPW.. The ITRS is concerned that as the semiconductor industry moves forward with new devices, using ever-shrinking line widths, the inability to detect small particles will no longer be masked. Particularly troubling are the following factors:

- The integrity of the final filters cannot be guaranteed for 100% of the time.
- New filters may shed undetectable nm sized particles and require excessive rinsing and qualification before use to offer an acceptable level of nm particle removal.
- Particles may be generated downstream of the final filters.

Known colloids (including SiO2, Al2O3, CeO2, Alum, and CaF2), metal particles (including Fe, Ni, Cr, or Ti), and organic particles will require continuous monitoring in the future, but as yet remain undetectable at sizes below 40-50 nm. Ultrafiltration appears to be efficient down to 10 nm. Improved cartridge filters are available now with 20 nm rating [30], but efficiency of particle removal is still not well known and there is always a risk of potential integrity damage.

Despite marginal improvements, no particle measurement metrology is available that fully meets the roadmap requirements in terms of speed, sensitivity, and statistical viability. In order to enable effective semiconductor manufacturing, particle counters that meet ITRS specifications are urgently required. With new particles metrology filtration efficiency needs to be validated and maintained.

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