New Particle Monitoring Strategies in UPW for 300 mm Fabs

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Presented at the 24th Annual SPWCC Conference, February 2005

Abstract

Process failures caused by particles in Ultra Pure Water (UPW) are relatively infrequent events, but with the move to 300 mm wafers and 90 nm line widths, they can have significant financial impact. Most particle monitoring strategies in the past focused on the use of the mobile and inline high sensitivity 0.05 micron particle counters. This paper examines the opportunities to improve performance, track data trends, and reduce cost by using a combination of both high sensitivity particle counters (to 0.03 microns) and less expensive lower sensitivity (0.1 microns) counters with very high sample volumes. The scope of this paper details the impact of size sensitivity, sample volume, and zero count level on particle counter performance. Practical monitoring considerations and particle distribution differences between poorly filtered and highly filtered systems are discussed.

Introduction

UPW is a vital component in semiconductor fabs. It is used in chemical make-up, CMP processing, and wafer cleaning and rinsing. In 300 mm fabs, as much as 1500 gallons of water are used to fabricate one wafer. As critical dimensions have decreased to 90 nm and smaller, the requirement for higher purity water and the need to monitor smaller particle sizes has increased.

In order to fully understand the benefits and drawbacks of any particle monitoring strategy, it is critical to understand the behavior of particles in UPW and some basics of particle counter design. Two of the most significant points are that particle concentrations in UPW are very low and that the volume of sample measured per unit of time (sample volume) by the particle counters can be very small. These two factors can result in poor counting statistics and poor particle counter performance. The push from the semiconductor industry to identify particles of smaller and smaller size resulted in the development of very high sensitivity instruments but at the cost of measurement precision due to small sample volume. Only recently has instrumentation been developed that is capable of meeting the strict sensitivity requirements while also improving the size of the sample volume. Since both particle counter size sensitivity and sample volume affect data variability, different counter designs may optimize one of these variables over the other. With an understanding of particle size distributions in UPW, less sensitive and less expensive particle counters can be quite useful in particle monitoring programs.

Particle Concentrations and Size Distribution in UPW Systems

Two criteria are needed to specify a particle concentration: 1) the number of particles/volume of fluid and 2) the minimum size of those particles. Particle concentrations in UPW system are very low, often less than 0.5 particles/mL for particles ≥ 0.05 microns. New 300 mm facilities are striving to achieve concentrations < 0.2 particles/mL for particles ≥ 0.05 microns. Particle concentrations are much greater for small particles when compared to particle concentrations for larger particles. In fact, particles distributions in filtered UPW systems follow a very predictable power law distribution, usually ~ $1/\text{diameter}^3$ as established by Knollenberg and Veal (1). This distribution results in a ratio of cumulative particle counts to particle size as shown in Table 1. Due to the exponential relationship between the number of particles and the particle size, graphs of particle size distribution are best expressed logarithmically (Fig.1).

Typical Particle Size Distribution in UPW	
Particle Size (μ)	Ratio of Particle Counts
0.2	1
0.1	8
0.065	29
0.05	64
0.03	296

Table 1: Concentrations of small particles in UPW are significantly greater than the concentrations of larger particles for a $1/\text{diameter}^3$ size distribution.

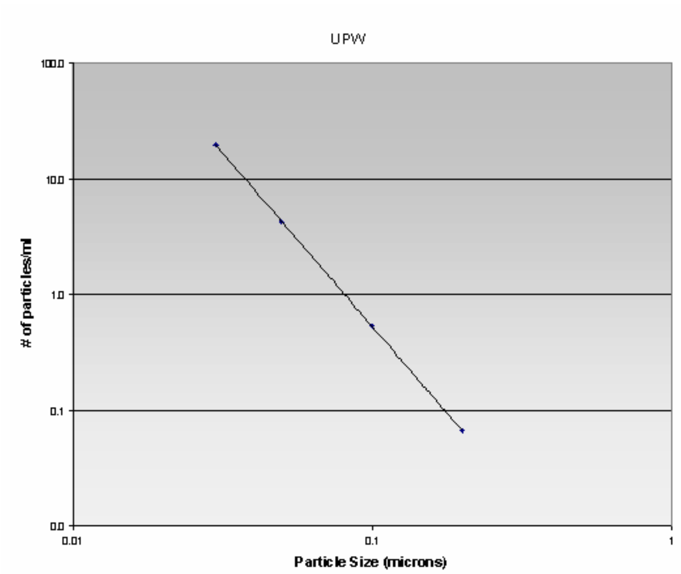

Figure 1: Particle concentrations in UPW show a predictable $1/\text{diameter}^3$ size distribution. This is useful in estimating particle concentrations at sizes below the particle counter's sensitivity limit.

Particle Counters

Optical Particle Counters (OPC) are widely used to monitor the cleanliness of cleanroom air, process gas and chemicals, vacuum environments, and UPW. This technology is usually considered the best available method for counting and determining particle size in UPW due to its high reliability and consistency. The results are independent of operator judgment and the technology is well suited to online process control.

In a liquid particle counter, laser light is focused on a sample cell or capillary, through which a liquid is flowing. Particle counters are designed to measure the "equivalent optical size" of real world particles based on the principle of light scattering. Light scattering occurs when a particle crosses the laser beam. Particle counters use optics to collect scattered light and to focus the light onto a photodetector. The photodetector then converts the scattered light into a voltage pulse whose amplitude reflects the size of the particle. Light scattering of real particles in liquids is influenced by differences in refractive indices of the particles when compared to the calibration solution containing polystyrene latex spheres and water. While this affects the absolute accuracy of particle sizing, particle counters produce consistent and comparable results on a day-to-day basis.

Particle Counter Performance

The most frequent cause of variability in particle counter data occurs because there are so few particles in UPW. In other words, counting statistics becomes the limiting factor in OPC performance and therefore in understanding the true quality of the UPW. More repeatable measurements are obtained if more particles can be counted during the sample interval. The performance criteria affecting the number of particles counted per unit time by a particle counter are 1) sensitivity and 2) sample volume. As measurement precision increases and the understanding of process variations and excursions improves, tighter control limits can be set, and the time required for UPW facility certification is reduced. A more thorough discussion of particle counting statistics has been published by Mitchell (2).

The amount of light scattered is dependant on particle size. For particles less than 0.2 microns, Raleigh scattering is predominant and predicts that light scattering decreases exponentially by a factor of $1/\text{diameter}^6$. If we were to relate this strictly to laser power, a 0.05 micron sensor would require 4096 times more power than a 0.1 micron sensor. This is because the signal-to-noise ratio needs to increase by a factor of 64 (2^6), but signal-to-noise only increases by the square root of increases in laser power. Considering this relationship, it is not surprising that high sensitivity particle counters tend to be much more expensive than low sensitivity counters. Further improvements in particle counter performance beyond the current 0.03 microns sensitivity limit will require significant advancements in laser technology and collection optics.

Given a natural distribution of particles of $1/\text{diameter}^3$, a 0.05 micron sensor will count 8 times the number of particles as a 0.1 micron sensor. However, this is hardly ever the case due to differences in sample volume between different particle counters.

Sample volume or "sample rate" is the amount of fluid examined per unit time. Sample volume is just as important as sensitivity in affecting particle counter performance. Particle counters can be classified as:

- Low sample volume: 0.1 to 0.5 mL/min
- Medium sample volume: 1 to 10 mL/min
- High sample volume: 50 mL/min or greater

Low sample volume particle counters are reaching the limits of their usefulness, due to the ultra-cleanliness of process chemicals and UPW produced by increasingly better filtration. This is especially true if high precision is required. For example, given a particle concentration of 0.5 particle/mL > 0.05 microns in a UPW system, a particle counter with a sample volume of 0.1 mL/min will take about 20 minutes before it detects even a single particle. This results in poor measurement precision and high variability between samples.

The relationship between sensitivity and sample volume has implications for the measurement of UPW in 300 mm fabs. As critical dimensions shrink, smaller particles can impact the process, so high sensitivity particle counters are needed. However, due to the small numbers of particles present in UPW, high or at least medium sample volume particle counters are required if measurement precision is important. Finally, with knowledge of the size distribution, estimates of particle counts are possible at sizes smaller than the sensitivity limit of the counter. Because size distributions are normally well understood, high sample volume particle counters (even though less sensitive) are useful in alarming at less critical monitoring locations.

Particle Counter Designs for UPW Systems

Liquid particle counters are based on the following fundamental designs:

- **Non-volumetric:** high sensitivity but low to medium sample volume. These types typically measure less than 1.0 % of total flow through the instrument.
- **Volumetric:** high sample volume but lower sensitivity. By definition, this design measures 100% of total flow through the instrument.

Non-Volumetric Particle Monitors

The most common particle counter used for UPW is a non-volumetric design, commonly referred to as a monitor. The non-volumetric monitor (Fig. 2) shows a tightly focused laser beam with the sample flow into the plane of the diagram. The scattered light is collected from the center of the sample cell which is far removed from the glass optical interface thus reducing stray light and improving sensitivity. Non-volumetric particle monitors used in UPW have high sensitivity, 0.03 or 0.05 microns. The drawback to this design is that only a small percentage of total flow is analyzed, resulting in low to medium sample volumes. In order to count a statistically sufficient number of particles, low sample volume monitors require longer sample intervals. Medium sample volume monitors can count a statistically significant number of particles in less time but are more expensive to manufacture.

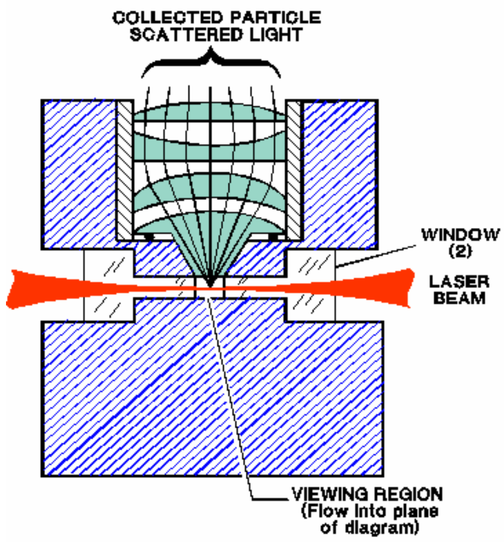

Figure 2: This non-volumetric monitor shows that the tightly focused laser is far removed from the glass optical interface to reduce stray light and improve sensitivity. Light is collected only in this small region, and much of the fluid does not pass through the laser beam. Note that the fluid flow is into the plane of the diagram (1).

Volumetric Particle Counters

The term *volumetric* refers to whether or not the particle counter is viewing the entire sample flow through the capillary. Most volumetric and near-volumetric particle counters use capillary designs. The laser beam in a volumetric particle counter is shaped so that its intensity is mostly uniform rather than tightly focused. Volumetric particle counters have traditionally been limited to process chemicals due to their lower sensitivities (0.1 microns). However, since they measure 100% of the fluid flow, they can have very large sample volumes (> 50 mL/min) and be very useful monitoring UPW. The large sample volume offsets the disadvantage of lower sensitivity, and this type of particle counter provides more repeatable results. Figure 3 shows Particle Measuring Systems' patented inviscid jet design used in the Liquistat 0.1 micron volumetric particle counter. This specific design does away with the capillary walls that are a source of scattered light, thus providing an improvement in signal-to-noise ratio.

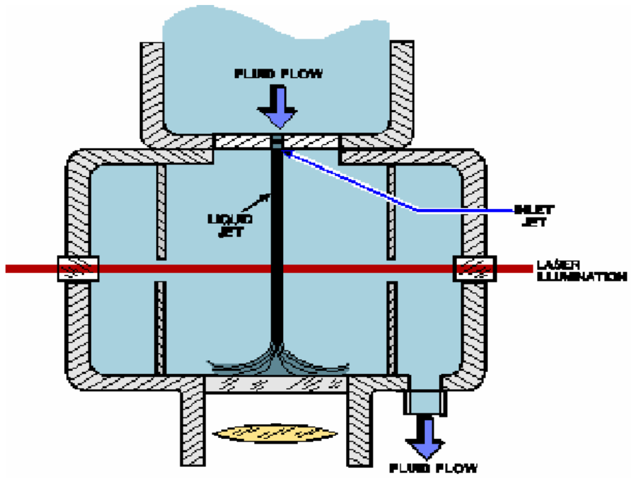

Figure 3: Volumetric designs measure 100% of the fluid and have improved sample-to-sample repeatability due to their large sample volume.

Monitoring Strategies

Particle counters can be further classified by their performance capabilities in UPW monitoring.

LEVEL 1: Trend analysis and alarm capability

A Level 1 counter can provide trend analysis and alarm capability at critical 300 mm Fab UPW locations. These counters have both high sensitivity (0.03 or 0.05 microns) and medium sample volumes. While these are the most expensive particle counters available, they do provide data trend information which can help identify and track process excursions.

LEVEL 2: Alarm capability only

A Level 2 counter only provides alarm capability and is typically used at less critical locations. It has the advantage of being less expensive. Traditionally, these have consisted of high sensitivity monitors with low sample volume. Another option exists, which is a lower sensitivity (0.1 micron) volumetric particle counter with a high sample volume. Volumetric particle counters produce statistically valid data more quickly and do not require long sample intervals.

Improving Measurement Precision

Both sample volume and size sensitivity affect the total number of particles counted and therefore the precision of the measurement. In other words, the time required to measure a statistically valid sample depends on the rate that the particles are counted. Since particles are counted according to Poisson statistics, increasing the sample rate by factor "x" will improve the relative measurement precision by the square root of "x". The relative precision (also called the *coefficient of variation*) is equal to the standard deviation/mean.

One method to improve precision is to increase the measurement time of each sample. This is the simplest method to improve results with no immediate cost implications. However, long sample intervals are often not an acceptable solution because during the sample interval, the process operation is blind. Also, modest particle excursions of short duration will be missed entirely.

Impact of Size Sensitivity on Total Number of Particles Counted

Since UPW systems have particle distributions around 1/diameter^3, we can estimate the particle concentration at any size, if the concentration at a less sensitive particle size is known. The concentration at the unknown particle size is a function of the power law distribution and the ratio of the two particle diameters.

For example, suppose it is known that a system has 1 particle/mL > 0.1 µ. It is then possible to calculate the number of particles > 0.05 µ:

# of particles > 0.05 µ = (number of particles > 0.1 µ) * 1/(ratio of diameters)^3
# of particles > 0.05 µ = 1 * 1/(.5)^3 = 8 particles/mL > 0.05 µ

An easy-to-remember rule of thumb is that if the particle diameter is halved, then the number of cumulative counts will increase by a factor of 8.

Impact of Sample Volume on the Total Number of Particles Counted

The impact of sample volume when monitoring UPW systems can best be understood graphically. In Figure 4, a Level 1 counter (Ultra DI® 50) is compared to a Level 2 counter (HSLIS M-50). Both particle counters have the same sensitivity but different sample volumes. In this example, the Level 1 counter has a sample volume of 3.75 mL/min and the Level 2 counter has a sample volume of 0.25 mL/min. This produces a rate difference of 15x. The Level 2 counter requires a much higher Upper Control Limit (UCL) due to the large sample-to-sample variation.

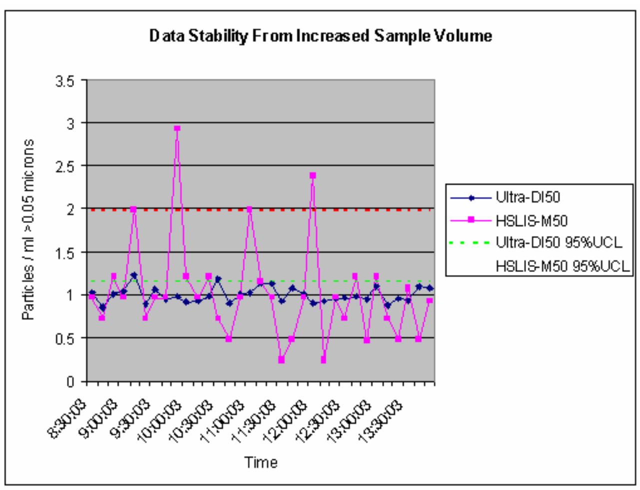

Figure 4: The Level 1 (Ultra DI 50) counter, with a larger sample volume, has less data variablity than the Level 2 (HSLIS M50) monitor of the same sensitivity. Tighter control limits can be established with the Level 1 counter.

The following example shows how upper control limits can be calculated for particle counters with different sample volumes.
Given:

- Customer's particle specification in UPW: 1 ct/ mL >0.05 microns (requiring the 95% UCL to be below this level).

- Average true UPW particle concentration: 0.5 cts/ mL >0.05 microns
- Sensors taking 10 minute samples over 500 minutes (about 1 day)
- Level 1 Counter: 0.05 micron @ 3.75 mL/minute
- Level 2 Monitor: 0.05 micron @0.25 mL/minute

The expected counts based on the true particle concentraton for the LEVEL 2 monitor:
= {(10min * 0.25mL) * 0.5 particle/mL} = 1.25 particles / sample interval
The expected counts for the LEVEL 1 counter:
= {(10min * 3.75mL) * 0.5 particles/mL} = 18.75 particles/sample interval

The 95% UCL can be calculated according to Mitchell (2):

UCL = number of expected counts + 1.65 * standard deviation (SD) of the expected counts. The SD is the square root of the expected counts according to Poisson statistics. A Student's t table shows that the 0.05 critical value is 1.65 (for the 95% UCL).

Level 2 monitor = 1.25 + (1.65 * 1.12) = 3.09 particles per sample = <u>1.23 cts/mL</u>
Level 1 counter = 18.75 + (1.65 *4.33) = 25.9 particles per sample = <u>0.69 cts/mL</u>

The Level 1 counter produces tighter control limits that are within the customer specification while the Level 2 monitor does not produce data that meets the customer specification. The proper choice of particle counters speeds the certification process of a UPW system and can save the UPW manufacturer thousands of dollars.

Size Sensitivity vs. Sample Volume

Since size sensitivity and sample volume impact the total number of particles counted, it is important to evaluate both of these parameters to see the combined effect on particle counter performance. The following example compares two Level 2 sensors:

1. Non-volumetric, high sensitivity monitor (0.05 microns) with a sample volume of 0.25 mL/min
2. Volumetric, medium sensitivity counter (0.1 microns) with a sample volume of 50 mL/min

We can estimate which one will perform better by first examining sensitivity and then sample volume. The volumetric counter would have 8x lower counts due to its poorer sensitivity but it would have 200x more counts due to its larger sample volume. The net result is that the volumetric counter has 25 times more counts when compared to the more sensitive non-volumetric monitor. The higher counts with the volumetric counter guarantees less data variability so tighter QA limits can be established (Fig. 5). The reduced standard deviation makes it easier to spot excursions.

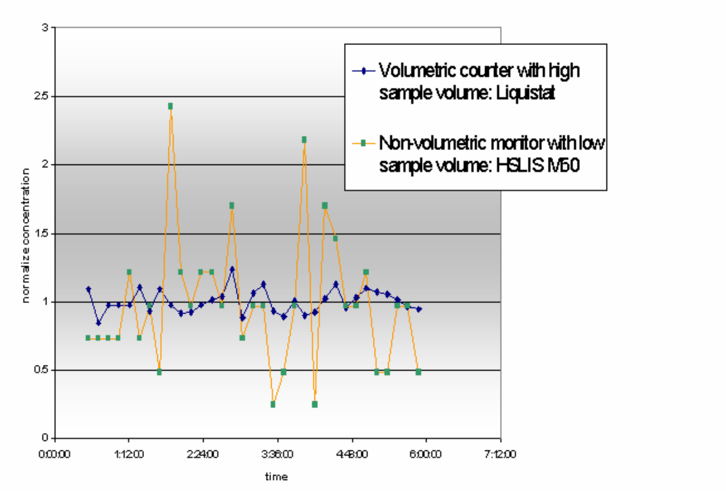

Figure 5: The high sensitivity (0.05 microns) of the non-volumetric Level 2 monitor does not guarantee better performance. The high sample volume of the volumetric Level 2 counter results in less data variablity. Tighter control limits can be established with the volumetric counter, even though it has poorer sensitivity (0.1 microns), Mitchell (2).

Performance Calulator

Particle Measuring Systems has developed a performance calculator which makes comparisons between particle counters straightforward (3). This particular tool calculates UCL as well as the number of alarms generated per day outside the control limits for particle counters with different sizing sensitivities and sample volumes. A better perfoming particle counter will have fewer alarms per day. An example is given below in Figure 6.

Criteria				
Smallest detected particle Size	0.030	microns		
Particle concentration	2.00	*total particles present in sample greater than 0.03 microns per ml of fluid*		
Sample Interval (seconds)	600	*seconds*		
Alarm Level	50.00%	*percent above expected particle concentration*		
Particle Distribution Power Function	-3	diameter^{x} (normally expect diameter^{-3} distribution)		
Particle Counter	**Instrument #1**	**Instrument #2**	**Instrument #3**	
Sample Volume	0.25	3.75	1.2	*ml/min*
1st Channel Particle Size	0.05	0.05	0.03	*microns*
Results				
Expected concentration >=1st Channel	0.432	0.432	2.000	*particles/ml*
Samples per day	144	144	144	*n*
Volume per sample	2.50	37.50	12.00	*ml*
Detected Particles >= 1st Channel	1.08	16.20	24.00	*per specifed distribution*
Detected Particles >= 0.03 microns	N/A	N/A	24.00	*per specifed distribution*
Standard Deviation >= 1st Channel	0.416	0.107	0.408	*particles/ml*
Standard Deviation >= 0.03 microns	N/A	N/A	0.408	*particles/ml*
P(sample exceeds Alarm Level)	0.3017	0.0221	0.0072	
Upper Alarm Level	0.65	0.65	3.00	*1st channel and greater per specif*
Alarms per day	43.44	3.18	1.03	*from statistical variation*
Possible Control Limits				
95.0% UCL >= 1st Channel	1.12	0.61	2.67	*particles/ml*
99.87% UCL >= 1st Channel	1.68	0.75	3.22	*particles/ml*

Figure 6: Performance calculator results for evaluation of particle counters with different sensitivities and different sample volumes. A better performing particle counter will have fewer alarms per day.

Zero Count Level

Another important criteria to ensure accurate counting at low particle concentrations in UPW is a low zero count. The “zero count level” or “background count” is caused by cosmic ray events which show up as particle counts. These occur on average about 1/minute and can have a pronounced effect on non-volumetric monitors due their low sample volume. A variety of methods are used to compensate for these false counts, including electronic circuitry and noise compensation algorithms (Fig. 7). Volumetric instruments with high sample volumes have inherently low background, so no compensation is needed. Background levels at critical monitoring locations in UPW facilities should ideally be less than 10% of the expected counts, to have a minimum affect on the data. Zero count levels < 50 counts/liter (0.05 cts/mL) are normally considered adequate.

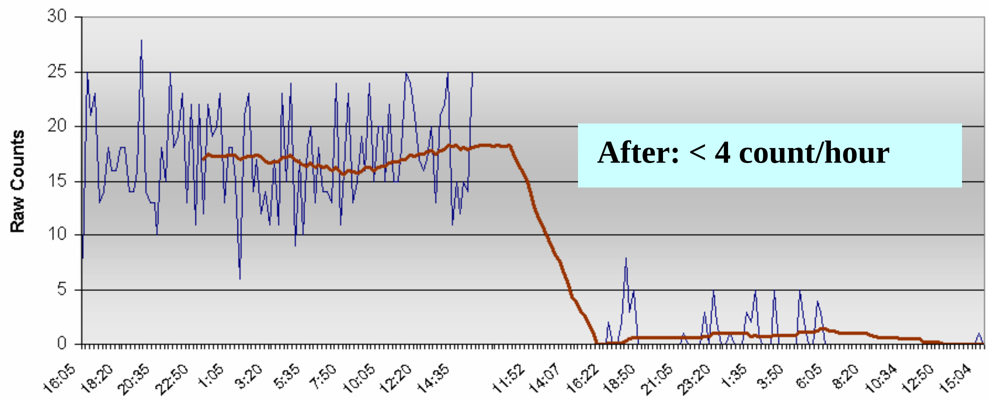

Figure 7: Zero count level being corrected using a noise compensation algorithm. Sample interval was 15 minutes. This data was collected on a LEVEL 1 counter with a sample volume of 3.75 mL/min. In this case, 4 counts/hour represents a background of 18 counts/liter.

UPW Manufacturing

Figure 8 shows a diagram of a UPW manufacturing facility. The pretreatment stage prepares water for the Reverse Osmosis (RO) system. Failures at the pretreatment stage can impact the life span of the RO system which can exceed $50,000 in replacement costs. Pretreatment filtration is normally to 0.5 microns, so particle monitoring at this stage can be accomplished with relatively inexpensive and much less sensitive particle counter than has previously been discussed.

The critical locations that are most suitable for Level 1 counters are the final filters and the DI water return. Monitoring pre and post final filters facilitates troubleshooting when particle problems are encountered. Monitoring the DI water return can help identify process excursions. Level 1 Counters can also quantify performance of 1) RO system (monitored below degasifier) and 2) roughing filters. Particle counting at these locations can facilitate an understanding of filter effectiveness and lifetime. While monitoring the pressure drop across a filter is commonly used to determine the need for filter replacement, particle counters have the advantage in that they are directly measuring the particles. A specific pressure drop across a filter may or may not signify the need for replacement as far as particle generation or filtering efficiency is concerned. Filters that are poorly seated following maintenance would likely have no pressure drop but be actively generating particles. Particle counters can be a key element in establishing a cost effective filter replacement program.

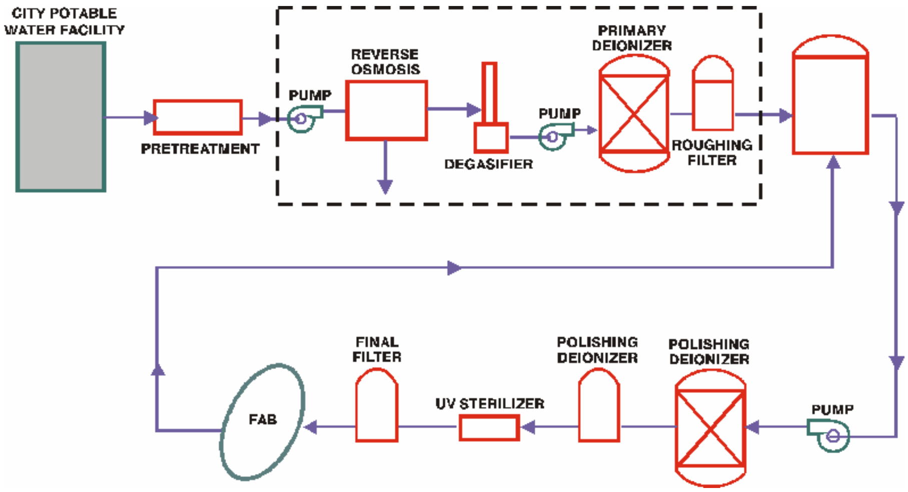

Figure 8: UPW manufacturing facility: critical particle monitoring locations are pre and post final filter as well as the DI water return from the fab.

UPW Distribution in a 300 mm Fab

Figure 9 shows UPW distribution from a UPW plant to a semiconductor manufacturing facility. The "mains" distribute UPW to the fab, and the lateral lines coming off the mains supply UPW to the individual tools. The "return" sends the UPW back to the daytank for further processing, including de-ionization, UV sterilization, and final filtering. In order to verify the incoming quality of the UPW, a Level 1 sensor is commonly used on the mains. This is the most critical monitoring location as a failure here can impact many tools. The laterals are considered less critical and suitable to a Level 2 sensor. Typical sample locations for the laterals would be near the intersection with the return.

Particle problems in the fab can be due to unbalanced flow. Low flow areas or "dead legs" are prone to the formation of bacteria colonies that can be dislodged due to pressure fluctuations. Installation or modification of tools, valving, and filters can also create particles. Permanent counters are most effective in identification of particle sources and bacteria colonies on the mains or the laterals. Mobile sampling carts with Level 2 sensors can be used for trouble shooting, though these are typically only useful in identification of gross particle problems. Mobile carts can suffer from bacteria build up when not being used and subsequently may require long cleanup times following installation. Volumetric Level 2 counters can provide more rapid results when troubleshooting, due to their ability to use shorter sample intervals when compared to non-volumetric Level 2 monitors.

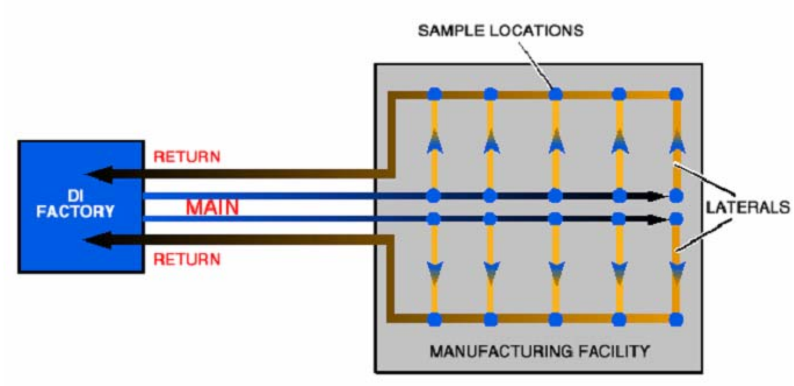

Figure 9: The diagram shows UPW distribution to the fab. Critical particle monitoring locations are the mains and the DI water return from the fab.

Practical UPW Monitoring Considerations for 300 mm Fabs

Particles smaller than 0.1 microns are more difficult to flush away and can require long cleanup times. This is true whether installing a new process tool or a new particle counter. Even with new tubing and valves, clean up times after installing a particle counter can easily exceed 12 hours in order to reach baseline particle levels. These long cleanup times impact the usefulness of strategies that rely heavily on mobile monitoring programs.

Particle distribution can be somewhat variable depending on the degree of filtration being employed. Multiple 0.03 micron filters (or smaller) in series can create steep distributions ($1/d^4$) while poor filtration creates less steep distributions ($1/d^2$). Figures 10 and 11 show examples of particle distributions from two different UPW systems. While normally the distribution in a 300 mm UPW facility is known and is close to $1/d^3$, it is still important to verify the concentrations of particles below 0.1 microns at the critical locations. Monitoring with a high volume (and less sensitive) LEVEL 2 counter while beneficial in rapidly alarming on problems, does not provide the information needed to fully understand all aspects of the UPW.

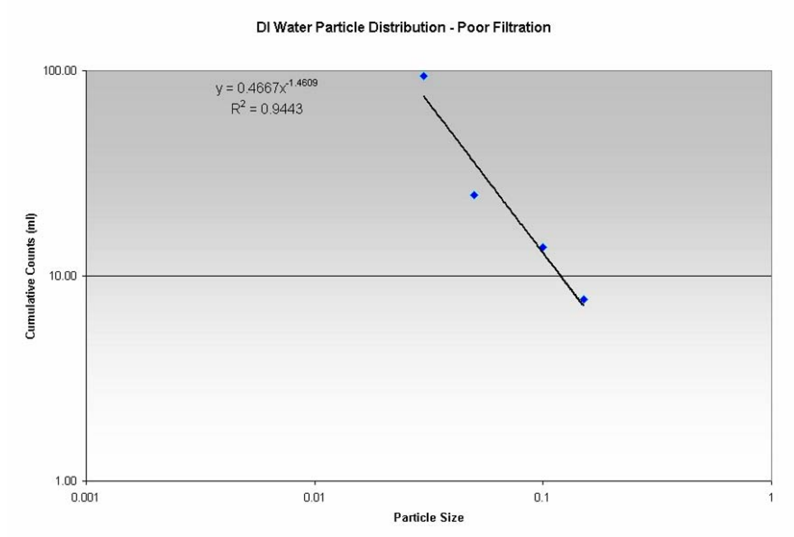

Figure 10: Poor UPW filtration is identified by high cumulative counts near 100 particles/mL > 0.03 microns. The particle size distribution is very low ($1/d^{1.5}$) with a correlation coefficient of only 0.94.

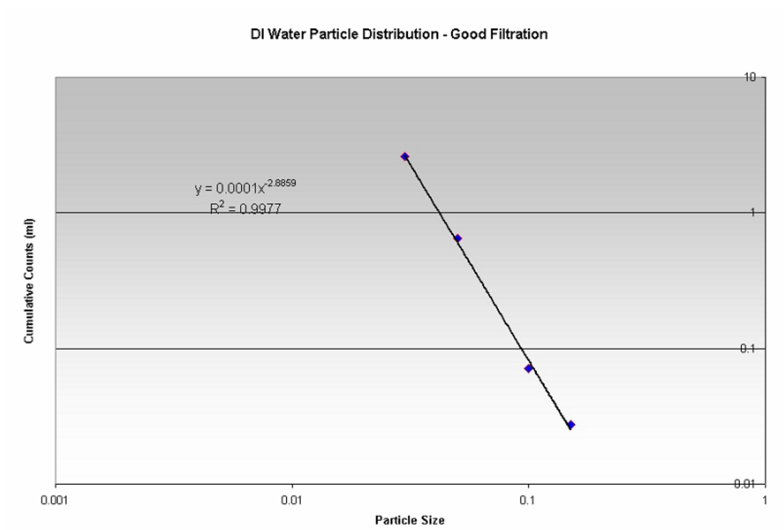

Figure 11: Good UPW filtration is identified by low cumulative counts near 2 particles/mL > 0.03 microns. The particle size distribution is much more typical ($1/d^{2.9}$), and the correlation coefficient is 0.99.

Comparison Data from a Semiconductor Facility

A LEVEL 1 monitor (sensitivity to 0.03 microns and 1.2 mL/min sample volume) and a LEVEL 2 monitor (sensitivity to 0.05 microns and 0.25 mL/min sample volume) were connected in parallel at a large semiconductor facility. This example (Fig. 12) shows normalized cumulative results collected on the LEVEL 1 monitor over 8 hours, using a 15-minute sample interval. There is low variability to the data due to the large number of counts measured (250 counts) during the sample interval. The LEVEL 1 counter is capable of monitoring trends in the data as can be seen from the gradual rise in particle counts slightly before 5:00 followed by a steady drop in counts. This is evident on both the >0.03 micron and the >0.05 micron channels.

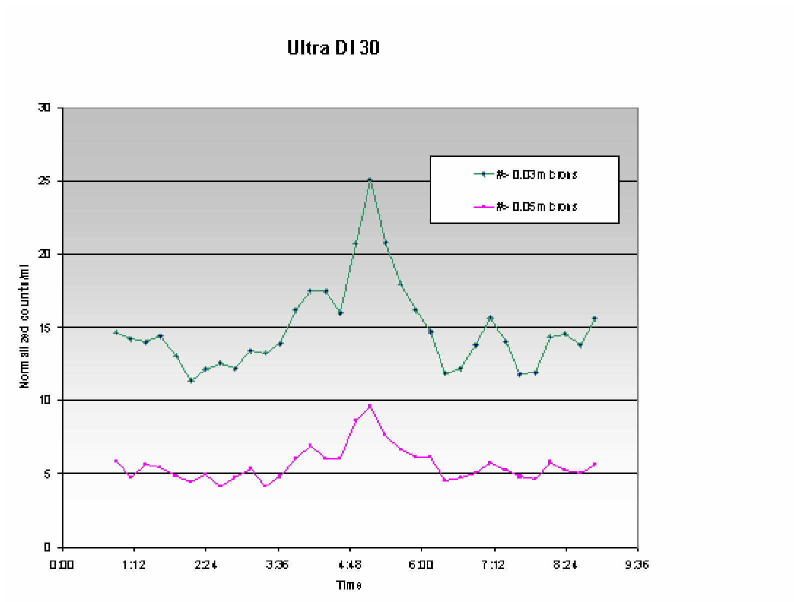

Figure 12: Particle trending information observed with a Level 1 particle monitor with high sensitivity and medium sample volume. Low sample-to-sample variability is due to the high number of counts per sample interval.

Data from the Level 2 monitor is included in Figure 13. The Level 2 monitor data (HSLIS M-50) is more variable due to poorer counting statistics. During the 15-minute sample interval, the Level 2 monitor measures an average of only 13 counts. The low sample volume, less sensitivity, and higher background levels contribute to the poorer precision. No data trend is detectable with the Level 2 counter.

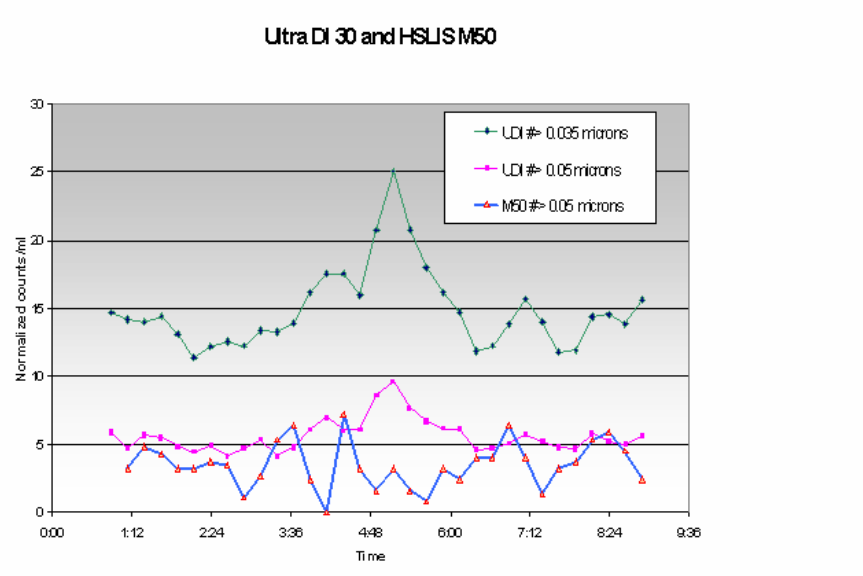

Figure 13: Particle trending information cannot be detected in the low sample volume high sensitivity Level 2 particle counter (HSLIS M-50).

Summary

High quality UPW is of vital importance in the production of 300 mm wafers with line widths at the 90 nm node and below. Particle monitoring strategies in the past focused on low sample volume and high sensitivity 0.05 micron particle monitors. However, improved performance and the ability to track data trends can be accomplished using a combination of both medium volume, high sensitivity particle monitors (0.03 or 0.05 microns) and less expensive high sample volume, lower sensitivity (0.1 microns) particle counters. The key measure of performance of any OPC is total particles counted. Sample volume and background noise levels are as important as particle size sensitivity when selecting a particle counter for tracking data trends and detecting excursions. Good counting statistics provide tighter control limits and a better understanding of process variations. In order to keep costs down, strategies can be employed that combine sensors for alarm purposes mixed with more expensive particle counters capable of trend analysis.

Biography

Ed Terrell is the Liquid Line Product Manager at Particle Measuring Systems. He has worked in the field of particle measuring for five years and has over 20 years experience in Chemical Analysis Instrumentation with ICP Mass Spectrometry, X-ray Spectrometry, Optical Emission, and ICP Spectrometry. He is currently a member in the SEMI Liquid Chemical committee and the SEMI Facilities committee with active participation in various task forces, including “Slurry Characterization,” “Particle Measurement in Process Chemicals,” and “Ultra Pure Water”. He has his Bachelor’s degree in mathematics from California State University.

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