Point of Use Particle Counting In High Purity Applications

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Particle Measurement in DI Water Systems:

DI water is used extensively in electronics manufacturing. In semiconductor manufacturing it is used for cleaning and etching wafers. DI water is also used in CMP processes and the critical immersion lithography process. Up to 1000 gallons of water is needed to process a single 300mm wafer. Because DI water is used in many critical process steps and directly contacts the wafer, controlling its contaminants is critical to maintaining high yields. On line monitoring of particle levels is a common method of controlling contamination in DI water systems.

While these systems do a superior job of treating and filtering the water, the output needs to be monitored in the unlikely event there is a problem with the system. Particles can occur due to issues with the piping, ion exchange beds, pumps and or filters.

At the same time DI water management systems are being offered for local treatment of DI water just prior to the point of use (POU) in critical process steps. Filters for particle removal are part of these local DI water management systems. Therefore it may be desirable to monitor these locations as well to prevent process issues, however cost effective monitoring solutions will be required.

Particle concentrations in semiconductor DI water systems are extremely low. The ITRS goals specify less than 0.2 particles per milliliter at the critical dimension and most systems are monitored to a specification of 0.2 particles per milliliter at 50 nanometers today.

Particle distributions in DI water systems often follow an inverse third power law relationship. Figure 1 below shows a typical particle distribution on a DI water system. Because of the power law relationship between particle counts and size it is often best to plot it on a graph with a log-log scale.

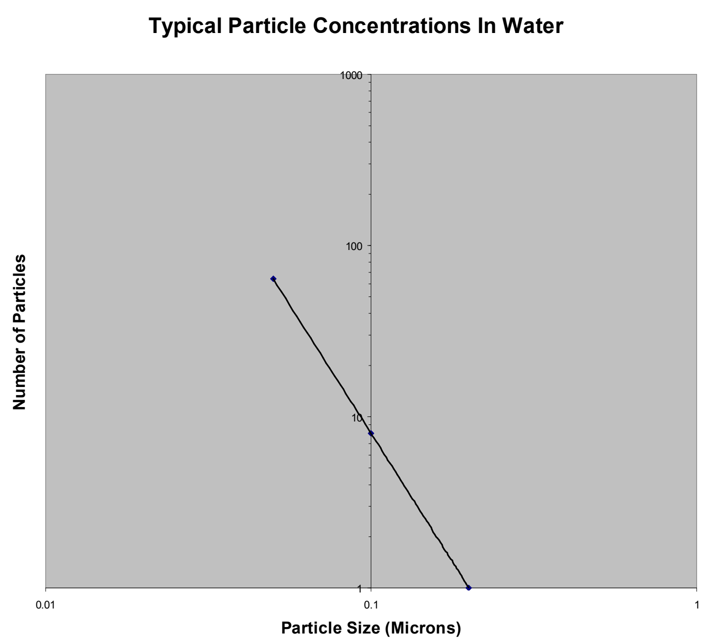

Figure 1 – Typical Distribution of Particles in DI Water

Liquid Particle Counter Technology

Optical particle counters measure particles individually one at a time. Historically two particle detection methods have been employed for monitoring particles in liquids. Light extinction is the first method and is often referred to as light blocking. It has also been referred to as bright field detection in the past. Here a light source is directed at a photodetector. A particle blocks the light or casts a shadow on the photodetector to create a signal by attenuating the light hitting the photodetector. This method is typically used to monitor large particles and is used pervasively in applications for monitoring drinking water, hydraulic fluids, and pharmaceuticals used in injections.

The other method of single particle detection is referred to as light scattering. This method of particle detection is also sometimes referred to as dark field detection. Here the particle redirects light from a light source when the particle intersects the light. This redirected or scattered light then causes an increase in the light that hits a photodetector thereby creating a signal for particle detection.

Light scattering is typically broken into two regimes referred to as Mie scattering and Rayleigh scattering. Mie scattering refers to scattering by particles of a size on the same order as the wavelength of the light source. The response throughout the particle size range can be very non-linear depending on the viewing angle and solid angle of light collected.

Rayleigh scattering describes light scattering from small particles as compared to the wavelength of light. Here the signal is proportional to the sixth power of the particle diameter. Therefore the signal decreases dramatically for particles of smaller sizes. Measurements in DI water are described by Rayleigh scattering.

In light scattering liquid particle counters a laser light source is focused on an optical flow cell that the fluid travels through. As a particle in the fluid travels through the laser beam, it scatters light. The scattered light is then gathered by collection optics and relayed onto a photodetector. The photodetector in turn generates a current that is converted to a voltage and output to the counter circuitry. The voltage output is in the form of a pulse, the amplitude of which is proportional to the size of the particle.

The counter circuitry then counts and bins the voltage pulses into discrete channels associated with the particle's size through a calibration process. At the end of the sample time each bin contains the number of particles for that particle size.

It should be noted that the light scatter signal is a function of the shape of the particle. The signal is also proportional to the optical index of refraction of the particle divided by the optical index of refraction of the liquid. Therefore the signal changes depending on the exact composition of the particle and the fluid type being monitored. Liquid particle counters report an equivalent size to a calibrated spherical particle (typically polystyrene latex) in water. Still particle counters produce very reproducible measurements in liquid applications, including DI water monitoring.

There are other specifications that define a liquid particle counter's performance. One parameter is referred to as the view volume. It describes how much of the flow path is illuminated by the laser and therefore what fraction of the flow is monitored by the particle counter. Historically there have been two types of particle counters for liquids. The first type of particle counter is a full stream particle counter that has a view volume close to 100% and measures particles throughout nearly the entire cross section of the flow path. The other type of particle counter is a partial stream particle counter which has a very low view volume and counts particles confined to a small fraction of the cross section of the flow path. Partial steam particle counters have a small view volume to maximize sensitivity or the smallest size particle they can detect.

Full stream particle counters are characterized by having good size resolution since the intensity of the laser is fairly constant over the view volume. Partial sensors have a much reduced sizing resolution since the laser intensity varies greatly throughout the illuminated flow path cross section.

Another parameter to consider when selecting a liquid particle counter is the flow rate of the particle counter. This defines the rate of fluid flow through the sensor. Higher flow rate particle counters can reduce clean up times and eliminate significant lag times in response to changes in the process being monitored depending on their location.

DI Water Particle Counter Performance Considerations

In DI Water applications the particle levels are extremely low and therefore one must carefully consider other factors that affect the accuracy of the instrument. First, optical particle counters have a measurement uncertainty that is a function of the particle's time of arrival which is governed by a Poisson statistical process. Thus the accuracy of the reported number of counts has a direct relation to the number of observed events or particles in a sample. The standard deviation of the measurement error is equal the square root of the number of counts. Therefore, to improve the accuracy of a particle counter's measurement one must improve the number of particles counted in a sample by some means.

To understand the performance of a specific liquid particle counter in a DI water application one must consider the sample rate of the counter which is defined as the view volume times the flow rate. The sensitivity of the particle counter as well as the distribution of particles being monitored must also be considered. For example, if the distribution of particles follows the typical inverse third power law then there are eight times as many particles present at 50 nanometers than at 100 nanometers. Therefore if two particle counters have these size sensitivities and the same sample rates the 50 nanometer instrument would count eight times as many particles and have an inherently better statistical accuracy. In addition, the number of particles counted is directly proportional to the sample rate of the sensor. Higher sample rate sensors with equivalent sensitivity produce a more statistically accurate result.

Another point to consider is the fact that at low particle levels particle counters are also subject to cosmic ray noise due to cosmic rays in the atmosphere. Cosmic rays occasionally bombard the detector with enough energy that they create a voltage pulse with sufficient amplitude that it is falsely counted as a particle. The cosmic ray pulse noise limits the particle levels that the instrument can measure accurately. The specific design of a liquid particle counter can greatly influence this background pulse noise level which is frequently expressed as noise counts per volume of fluid measured.

DI Water Particle Counters Design Theory and Calibration

In general DI water particle counters employed today are partial stream sensors. The instrument is designed to maximize sensitivity to allow for monitoring the particle sizes required. Therefore the view volume is a small percentage. In addition the laser intensity illuminating the flow cell varies greatly and roughly follows a Gaussian intensity profile. Therefore a particle traveling through the center of the laser beam will register a larger voltage pulse than a particle traveling through the edge of the laser beam. The net effect is the sensor has reduced sizing resolution, and as the size of the particle increases a larger area of the flow path cross section contributes to output signals that are registered by the instrument.

At first glance this may make one wonder how to quantify the number of particles since the number counted varies with size. Fortunately in DI Water applications we are monitoring a power law distribution in the Rayleigh regime where the response of the instrument follows a strict power law. Therefore rigorous analysis shows that if the instrument is calibrated such that the size channels follow the Rayleigh scattering response, the reported counts at each size are a consistent fraction of the true counts in the fluid and a single view volume can be used to normalize the counts for all sizes. This view volume is easily established by comparing the counts at an overlapping size at the upper end of the range of the instrument with the counts at the same size of a full stream sensor on a DI water system.   The resulting computed sample rate dictates the count rate and the resulting statistical accuracy of the sensor.

Requirements and Design of a Point of Use DI Water Particle Counter

Some key requirements for a point of use DI water liquid particle counter include:

1. 50 nanometer (0.05um) sensitivity for semiconductor processes
2. Adequate view volume for statistical accuracy
3. Low pulse noise to minimize the background false count level
4. A small footprint to facilitate integration into process equipment in point of use applications
5. An optimum flow rate for process control applications
6. A fast clean up time for portable applications
7. A low cost of ownership including annual maintenance and calibration costs

To achieve a sensitivity of 50 nanometers starting with 100 nanometer technology requires the signal to noise of the technology be improved by a factor of 64. To achieve this factor of improvement a number of technologies must be utilized simultaneously.

The first improvement factor comes from the utilization of a high brightness diffraction limited single mode laser diode. A maximum brightness or optical throughput is achieved by using a single mode laser diode rather than a higher power multimode laser diode. In addition this design approach lends itself to a relatively low cost of ownership since this light source is typically less expensive than the alternative technologies. Next a low aberration optical system allows one to produce a very bright laser spot in the center of the flow cell from the single mode laser diode.

Another improvement factor comes from using a high gain photodetection system. This offers a significant signal to noise improvement over conventional photodiodes. A final critical element of the design is a very high numerical aperture light collection optical system that has minimal aberration and a low magnification. The low aberration and magnification permit the use of a small photodetector to reduce the background noise and stray light in the optical system. This design approach helps the system achieve the required signal to noise and it also minimizes the pulse noise from the photodetector to allow the system to achieve very low background pulse noise levels.

Figure 2 below is a DI water counter designed to meet the requirements outlined previously in this section.

Figure 2 – A DI Water Liquid Particle Counter

Figure 3 below demonstrates the performance of the instrument described above. Very repeatable particle readings are achieved at extremely low particle levels.

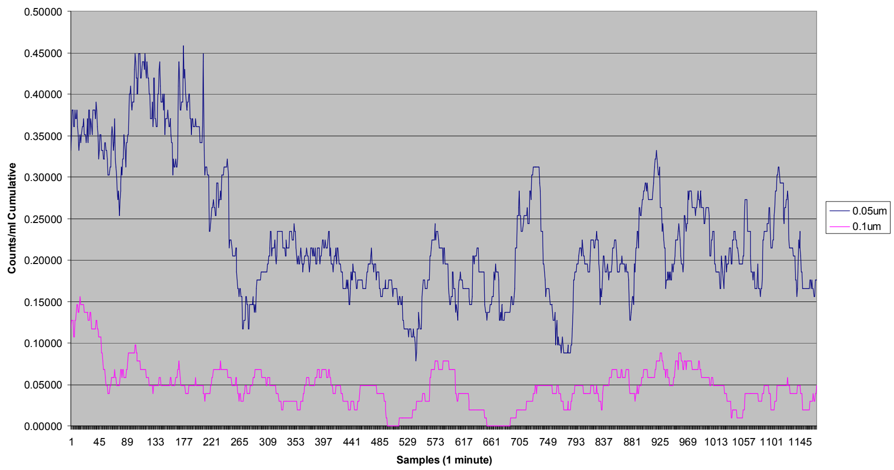

Figure 3 – Test Results From a DI Water System

Summary

Clean DI water is critical to semiconductor manufacturing. Liquid particle counters are used extensively to monitor contamination in DI water. The sample rate, size sensitivity and background noise level are critical specifications for a DI water counter and generally determine their performance in these applications. Point of use particle counting imposes additional requirements on these instruments including a low cost of ownership and small footprint. Achieving the requirements for these instruments requires that several technologies be utilized simultaneously to maximize sensitivity and view volume while minimizing the instrument's background pulse noise.

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