CLEANROOM MONITORING NEWS
ELECTRONICS

PARTICLE MEASURING SYSTEMS

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- **Airborne Particle Measurements of 100 LPM vs. 1 CFM Counter in a Semiconductor Cleanroom Environment**

UPCOMING EVENTS

- **AAAR 29th Annual Conference**
  Portland OR - October 25-29
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NANOVISION TECHNOLOGY™ - LIQUID OPTICAL PARTICLE COUNTERS ENTER THE DIGITAL AGE

For approximately four decades, liquid optical particle counters (OPC) have been built around six main components:

- A single wavelength laser
- Beam-shaping optics
- A measurement region
- Collection optics
- A photo-detector
- An analog-to-digital converter

Over the years, advancements in geometry, optics, and detector design have pushed the limits of detection down to 50 nanometers (nm); however current instrumentation can monitor only a small percentage of real-world particles at this size.

NanoVision Technology, a revolutionary technological breakthrough, brings particle counters to the digital age. Utilizing a high density two-dimensional array of high-efficiency detector elements, this new technology now pushes detection limits to 40 nm and with future improvements, to 30 nm. *Click to Continue*

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SYRINGE SAMPLING FOR COUNTING PARTICLES IN FLUIDS

A syringe sampler represents the simplest system for precisely delivering a sample to the particle counter. The precision motion of the syringe drive produces a steady flow of fluid that is free of pulsations that are typical of most pumps suitable for chemical delivery. Additionally, the elimination of any valves or complex sample plumbing ensures rapid clean-up between samples. *Click to Continue*

LIQUID PARTICLE COUNTING CONSIDERATIONS

This paper covers several basics of liquid particle counting. It starts by providing examples from industries currently using particle counters and suggests how this can be used in other areas. Also discussed here are the advantages of using an optical particle counter (OPC) and the considerations of sample volume and sensitivity. *Click to Continue*

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September 2010 - Issue 2, Volume 1

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NanoVision Technology™

Liquid Optical Particle Counters Enter the Digital Age

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There are many industries that use OPC's to monitor trace particle contamination in liquids. Pharmaceutical companies producing protein-delivered drugs monitor for particles that the proteins can agglomerate around, inhibiting their effectiveness. Other pharmaceutical companies detect low index particles such as bacteria in water treatment systems. Semiconductor manufacture requires extremely pure liquids for cleaning, etching, and developing silicon wafers. Particle contamination in these fluids can cause a variety of defects and once deposited, is very difficult to remove. The continuous miniaturization of semiconductor circuits as predicted by Moore's Law requires continuous improvement in the purity of these fluids. Monitoring these ever increasingly clean fluids requires constant improvements in monitoring technology. (1)

To illustrate the purity of these fluids in more well understood units, if the concentration of particles by volume is converted to parts per trillion (ppt), 200 particles per liter greater than 50 nm equates to less than 0.1 ppt by volume. Other contaminants in ultra pure water such as non-volatile residue, metallic, and other ions may exceed the concentration of particles by more than 4 orders of magnitude. In the world of analytical instrumentation, there are very few techniques that approach this degree of sensitivity.

OPC Design, History, and Evolution

There are basically two types of liquid optical particle counters: volumetric and non-volumetric. These two approaches balance tradeoffs between sensitivity and particle size resolution. Volumetric OPC's size particles very accurately, while non-volumetric OPC's detect very small particles by sacrificing size resolution. These instruments measure the amount of light scattered by particles as they pass through a laser beam in essentially the same manner that your eye sees household dust particles as they float through a beam of sunlight. (2)

Volumetric optical particle counters

Volumetric OPC's analyze the entire sample by optically shaping the laser beam profile to illuminate the entire measurement capillary. Particles traveling through any portion of the sampling region are illuminated with approximately the same intensity of light, which results in a high resolution measurement, where the amount of light scattered is proportional to the particle size. The sensitivity is limited by laser power, the amount of molecular scatter caused by the carrier fluid, and the scatter caused by the measurement capillary walls. Current technology is limited to about 100 nm particles. (2)

Non-volumetric optical particle counters

Non-volumetric OPC's are typically much more sensitive because the laser is focused into an intense but small region of the measurement capillary. Laser beam profiles are intrinsically more intense at the center of the beam, and less intense near the edge of the beam. Because of this intensity profile, non-volumetric particle counters exhibit a phenomenon called sample volume growth. Sample volume growth occurs when a large particle travels through the lower intensity outer region of the laser beam. This scatters a small amount of light, and thus the particle is sized as a smaller particle. Small particles traveling through the same low intensity region do not scatter enough light to be counted. This effect results in an increase in sample volume that is proportional to the size of the particle. The fact that a large particle can be sized as a small particle demonstrates the lack of resolution of this approach. Non-volumetric particle counters are currently limited to approximately 50 nm particles.

Historically OPC detectors utilized a photomultiplier tube to quantify the scattered light. This approach was improved through the use of a photodiode as the detector, which had the benefits of increased long term stability and a smaller footprint. These technologies had a limited signal-to-noise ratio and the detection limit was limited by the molecular scatter of the sample matrix itself. This signal-to-noise ratio was eventually improved through the use of array detectors in 1990. (3) This approach enabled high-sensitivity counters by dividing the sample volume into smaller sections, which spread the background scatter among multiple detectors, and pushed the limits of the technology down to 50 nm. Despite all these improvements, OPC's have remained analog in nature. (2)

NanoVision Technology

The next major advance in detector technology brings optical particle counters into the digital age. This novel approach couples a high power laser with a high-density two-dimensional array of high-efficiency detector elements to digitally image the light scattered by particles as they travel through the measurement capillary. High-efficiency detector elements exhibit a high photon-to-signal yield. A digital imaging approach is similar to traditional diode array approaches, where noise is spread over an array of detector elements. The difference is that NanoVision Technology uses a significantly larger number of detector elements. Distribution of background noise in combination with high-efficiency detector elements results in a very high signal-to-noise ratio.

This digital imaging approach offers other distinct advantages over traditional analog-to-digital signal processing methods. Most notably, signal discrimination; the light scattered by a particle exhibits a predictable and repeatable fingerprint that travels in the direction of the flow. The particle size range (40 – 125 nm) is much smaller than the laser wavelength of 808 nm. Therefore, the digital image is a picture of the light scattered by the particle, or a fingerprint, and not an image of the particle itself. Utilizing proprietary signal processing electronics and software; it is possible to discern the light scatter fingerprint of a particle from both background molecular scatter, and other sources of background noise such as random, stray high-energy photons. Traditional analog-to-digital signal processing approaches have no mechanism for discerning these non-particle events from actual particles.

The particle fingerprints are a result of Rayleigh scattering. Rayleigh scattering is an elastic scatter, or scatter in all directions, from the particle. The amount of light scattered is dramatically reduced as the particle size diminishes. Water molecules in 1 mL scatter approximately two times more light than theory predicts will scatter from a 50 nm particle. Additionally, capillary walls, and possible contamination on those walls contributes significantly to the background light scatter. Analog techniques can only go so far to extract the signals from small particles out of the background noise. Both cell walls and molecular scatter are important limiters to sensitivity.

The ability to digitally image the illuminated region of the capillary allows portions of the sample region to be ignored. Light scattered from capillary walls or contamination on these walls does not affect other areas of the sample volume. Additionally, a proprietary signal-normalizing calibration procedure can be utilized to compensate for differences in signal strength from different regions of the sample cell. Because of this, there is zero sample volume growth with the digital imaging approach. Particle size resolution is approximately 5%, which is similar to volumetric-based approaches. Sensitivity, however, is improved to 40 nm in liquid chemicals, and with future development, 30 nm detection in ultra-pure water is expected.

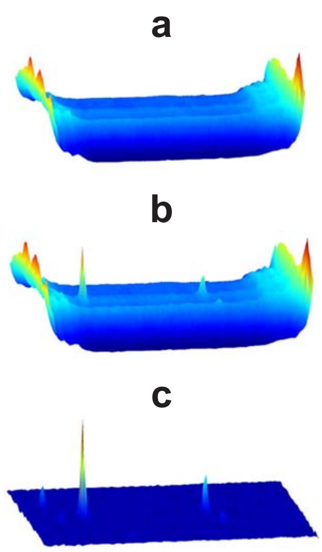

Figure 1. 3-D representations of imaging grid

Figure 1 displays a series of 3 dimensional representations of the imaging grid.

- **Figure 1a** displays a three-dimensional representation of a background signal. Signal intensity is represented in the vertical axis, with red denoting a high intensity signal or high rate of light scatter, while blue

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denotes a low intensity, or low light scatter region. The red borders on each side of the image are a result of the light scattered from the capillary walls. The flat lower blue section corresponds to the light that is scattered by the sample fluid.

- **Figure 1b** displays a raw image of multiple particles as they are carried through the measurement capillary.
- **Figure 1c** displays the difference between the first two grids (**Figure 1a** and **Figure 1b**). This background subtraction clearly shows the particle fingerprints.

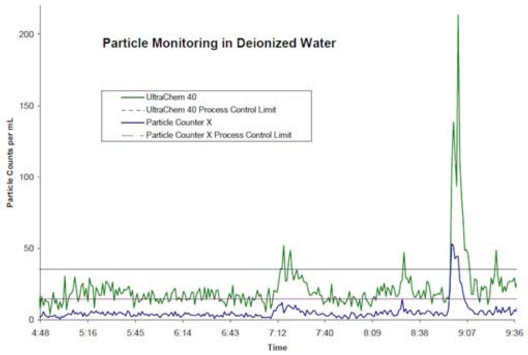

Figure 2. Head to head comparison of two particle counters that have different limits of sensitivity

Figure 2 shows a head-to-head comparison of two particle counters that have different limits of sensitivity. Particle counter X (bottom) has a limit of sensitivity of 50 nm, and represents the previous best available technology for monitoring small particles in clean systems. Particle counter Y is a particle counter that utilizes NanoVision Technology to detect particles down to 40 nm. In this example a deionized water system was monitored for about 3 hours. The ability to detect smaller particles is a major advantage when monitoring clean systems because with ambient particle size distributions, as particle size decreases, the number of particles increases. The ability to detect 40 nm particles in this comparison resulted in over 200% more counts per milliliter.

When utilizing statistical process control, counting more particles is a distinct advantage. The horizontal dashed lines in **Figure 2** show process control limits set at 3σ the normal noise of the system. Trends in particle concentration can be seen in both instruments; however the instrument with 40 nm sensitivity was able to detect 4 discrete particle spikes compared to 1 particle spike with the 50 nm sensitivity unit. This allows the manufacturing engineer to identify particle events or excursions in their process that previously would have gone undetected. This additional information provides greater opportunity to make continuous process improvements and put the necessary controls and procedures in place to prevent future events.

The continuous reduction of semiconductor feature size, along with more-specific drug delivery methods and other ultra clean industrial processes, have required continuous improvement in particle measuring technology to keep pace with cleanliness specifications. The monitoring of ultra pure process chemicals and water is essential for preventing defects, benchmarking new or additional procedures, and monitoring filter lifetimes and efficiencies. The NanoVision digital imaging approach currently demonstrates 40 nm sensitivity, and allows the counting and sizing of particles that until now were below the detection limit of the available technology.

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Syringe Sampling for Counting Particles in Fluids

A syringe sampler represents the simplest delivery system for precisely delivering a sample to the particle counter. The precision motion of the syringe drive produces a steady flow of fluid that is free of pulsations that are typical of most pumps suitable for chemical delivery. Additionally, the elimination of any valves or complex sample plumbing ensures rapid clean-up between samples. Care must be exercised when bubble formation is a concern; however, proper sample handling can usually overcome these issues. The syringe sampler is a valuable and low-cost tool to use for testing particle levels in fluids.

When particle measurements from an unpressurized source are required, a method for delivering the fluid to the particle counter must be selected. The choices available depend on the fluid that must be sampled. Water samples provide the greatest range of options, while fluorinated chemistries such as HF can restrict the choices significantly. Determining the best solution requires consideration of the compatibility of the wetted surfaces with the fluids that must be sampled. Additional consideration should be given to the possible combinations that can be created in the plumbing and pump cavity when switching from one chemistry to another.

Unlike peristaltic and diaphragm pumps, the syringe pump draws the fluid through the particle counter in a smooth continuous flow. Pulsations in flow that are typical with peristaltic and diaphragm pumps represent the increase and decrease in fluid velocity and this change will affect the instrument resolution. As a reminder, resolution is the ability of the particle counter to discern size difference between two particles close to each other in size. Gear pumps, while offering steady constant flow, can be limited in use due to fluid compatibility with the wetted components.

Another consideration for the sampler is the volume of sample available. Many particle counting applications only have a limited volume, yet making several measurements is desired for demonstrating sample accuracy. The ability of a syringe sampler to precisely measure volumes < 1 ml is very advantageous in this situation.

Additionally, the syringe sampler has the ability to delay initially while the flow ramps to speed and stabilizes at the particle counter's calibrated flow rate. This delay is often called the tare volume in particle counting applications. Coordinating such a delay in other pumping solutions could be difficult and costly to implement.

Unlike a compression sampler, which pushes the fluid through the particle counter, there are no valves or volume measurement containers in front of the particle counter. The additional plumbing associated with these types of samplers must be cleared of particles between every sample, which can potentially increase the time and chemical volume required to complete a series of samples. Since the syringe sampler draws the sample through under a vacuum, the plumbing that exists between the sample fluid and the particle counter is limited to the tubing connected to the sampler inlet. Cleanup between tests, purging the system of the previous sample, and other maintenance tasks will require fewer samples, which translates to less fluid waste.

Taken altogether, the multiple benefits of flow and volume accuracy, smooth continuous fluid motion, small volume capability, and coordination of particle counting with sample flow enables the syringe sampler to provide many attractive advantages for fluid delivery to a particle counter.

Figure 1. SLS-1200 Particle Counter

September 2010 - Issue 2, Volume 1

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Liquid Particle Counting Considerations

This paper covers several basics of liquid particle counting. It starts by providing examples from industries currently using particle counters and suggests how this can be used in other areas. Also discussed here are the advantages of using an optical particle counter (OPC) and the considerations of sample volume and sensitivity.

Benefits of Monitoring

The obvious reason for monitoring the fluids used during the manufacture of products is to somehow reduce costs and improve yields. In many cases the methods of accomplishing this are straightforward, yet some industries opt for predominately labor-intensive methods or wait until the product has been completely packaged before performing a final QC analysis on the cleanliness.

Learning from Industry

Much could be learned in many industries by performing a survey of best practices used in other industries that rely on clean manufacturing. For instance, semiconductor manufacturers learned many years ago that a significant savings could be realized in the factory by using particle counters to determine the frequency of filter replacement. Inevitably the pressure drop specification was set significantly towards the conservative side due to the extreme risk of being wrong. However, by using an appropriate combination of particle counters and other electronic monitors to provide a clear picture of fluid quality they discovered that filters did not require replacement until 2 – 3 times their original frequency. Given the cost of filters, the ROI on monitoring equipment can be as short as one year. Currently, the pharmaceutical industry continues to use the filter drop method of filter replacement and prefers to inspect quality into the system just prior to product shipment.

Data storage companies have been driven to compete on single digit margins, while producing some of the most dramatic increases in memory capacity. In the process they have forced the development of relatively inexpensive re-circulating, continuously filtered cleaning systems that insure the components that go into a disk drive have only a few particles per square centimeter larger than 0.2 microns. In many cases the cleanliness is automated and industry continues to use stagnant cleaning baths in many cases. Some have reported that cleaning is futile because the parts are more contaminated after leaving the cleaning bath than they were when they went in. Quality assurance is established by visual inspection, or gravimetric analysis of the residue removed from a part by means of spray extraction and collection on a filter patch.

Figure 1. Parts cleaned in various industries

When considering the benefits of switching to automated particle monitoring, you should look beyond the initial cost of equipment and evaluate all the ways the instrument can be used to save money. Several years ago, a larger semiconductor fab purchased several compression batch samplers. They were given to two different cleanroom areas run by different managers. One manager used the sampler almost exclusively to optimize filter selection for each process. The other manager integrated the sampler into the process equipment and monitored for any process upsets. Both reported that the equipment paid for itself within six months. The information learned was shared throughout the company allowing them to save a large amount of previously wasted money.

Figure 2. Compression Sampler

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Measurement Basics

A question that frequently arises is, "How do optical particle counters compare to other methods?" The answer is, "it depends." Consider, for example, the many different ways to measure a particle visually. Most naturally occurring particles are irregular in shape, so how do you classify the size?

Referring to **Figure 3**, is it the longest dimension? The longest dimension in one axis? Perhaps it should be the equivalent area? When examining most particles, each of these different methods will yield a slightly different answer.

Optical particle counters (commonly referred to as OPCs) work on one of two principals; they either measure the amount of light that is obstructed by the particle as it passes through a directional light source of known energy density (light obscuration), or they measure the amount of light that is scattered away from the directional light source (light scattering). The result is compared to the amount of light (obscured, or scattered respectively) by a calibration particle of known size and shape, typically polystyrene latex spheres (PSLs). In other words, an optical particle counter will report natural particles as the optical equivalent diameter of a PSL suspended in water.

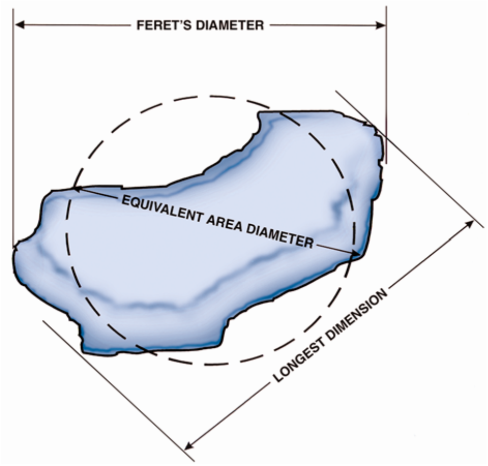

Figure 3. Measuring Particle Size

While this may again result in a slight difference in the measurement obtained by other methods, the repeatability of this measurement is highly reliable. Couple this with the relative ease of use, and reduced labor intensity and the benefits are generally believed to be significant.

There are a few considerations that should be given to implementing a monitoring program using OPCs. When monitoring on-line, care should be taken to eliminate any source of particle shedding upstream of the particle counter. Valves that are used to isolate the OPC when not in use at a given location should be opened 100% during monitoring. Flow control should be accomplished downstream from the OPC.

Partially open valves contribute to particle counts in two ways:

- First, they trap and release particles continuously over time, resulting in the appearance of elevated particle counts. Most valves present more surface area to the flow stream when only partially open. Pressure surges caused by operating other valves in the system will cause more particles to be released from the increased surface area.
- Second, depending on the extent to which the valve has been opened, it can produce a pressure drop that causes bubbles to form in the sample stream. OPCs cannot typically distinguish a bubble from a particle, so the data will appear to contain more particles than are truly present.

When performing batch sampling operations, it is important to allow sufficient time for the sampling apparatus to clean-up. Proper operation of the equipment is essential.

Following the manufacturer's recommendations will usually insure quick and simple testing with this type of equipment.

Sample Volume vs. Sensitivity

When selecting a particle counter, the value of total particles counted per unit time cannot be emphasized enough. Particle Measuring Systems has written several papers that discuss the reasons statistical significance is important, so this paper will not repeat them here. In general, it should be known that as particle counters approach the limits of technology, the volume of liquid assessed by the instrument will be less than 100% of the flow rate. Some manufacturers of particle counters do not publish this difference in their specifications, so it is important to ask, "What is the sample volume?"

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Figure 4. Particle Counter with High Sensitivity and High Flow Rate

If the application of interest requires high sensitivity (ability to detect very small particles) in a very clean fluid, you must evaluate the time required to count a minimum number of particles. In many ultra pure applications this time may be several hours. Unfortunately, a sample interval of this length will not allow the operator to detect temporary upsets of short duration. If the ability to detect these upsets is important, consideration should be given to sacrificing sensitivity for a particle counter with greater sample volume. It should be easy to understand that process upsets will not generate only one size particle, but rather an increase in particles at all sizes proportionately. Therefore, a less sensitive particle counter will be able to detect the upset and report it as it happens because it can be set with a sample interval of only a few minutes.

Conclusion

Optical particle counters are used in many industries. However, they are not used to their most effective benefit everywhere. Industry benchmarking could help many manufacturing companies that are just entering the ultra clean manufacturing environment to make significant strides forward.

While there are differences between OPCs and visual measurement techniques, they are not of significance and the highly repeatable nature of OPCs makes them an operator's tool of preference.

Finally, consideration must be given to the number of particles counted in the sample interval desired. If this number is not enough for statistical significance, the better choice is often a less sensitive particle counter with greater sample volume.

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