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Particle Detection in Liquids with High Viscosity or High Molecular Weights

Introduction

Laser-based particle counters have been used extensively to detect particles in DI water and process chemicals such as hydrofluoric acid. Not as many users have applied these tools to monitoring contamination in liquids with higher viscosity and/or molecular weights such as photoresist and anti-reflective coatings. Monitoring particles in such fluids is important for both the manufacturers and end users of these products. Manufacturers must ensure the cleanliness of their products before they leave the factory. End users need very clean materials which will not increase product defect densities. Traditionally, the cleanliness of photoresist and ARCs has been measured by applying them to a silicon wafer and inspecting the wafer on a laser-scanning system. This is wasteful of valuable chemicals, silicon wafers, coating system time and operator effort. A better method would be to use a particle counter on the liquid itself.

However, photoresist and other light-scattering media present numerous challenges to particle monitoring equipment. Some sensors can be overwhelmed by the light scattered from the fluid and produce inaccurate results. Other systems work very well for sampling out-gassing liquids but use a debubbling sampler which can be wasteful of very expensive fluids. Furthermore, since the liquids to be sampled were designed to coat surfaces, debubbling samplers can be difficult and time-consuming to clean between samples.

The solution is to use an LS-50 sampler and a LiQuilaz®-S02 sensor which has been optimized for the parameters of the liquid. The LS-50 sampler has a 25ml syringe and is available in materials compatible with Hydrofluoric acid. It has a smaller foot print than a debubbling sampler and does not require a source of pressurized gas for its operation. The high sampling volume of the LiQuilaz® sensor and the minimal internal volume of the LS-50 result in a cost efficient method of gathering particle data. The selectable thresholds of the LiQuilaz® sensor allow it to be used with fluids that have high molecular scatter coefficient.

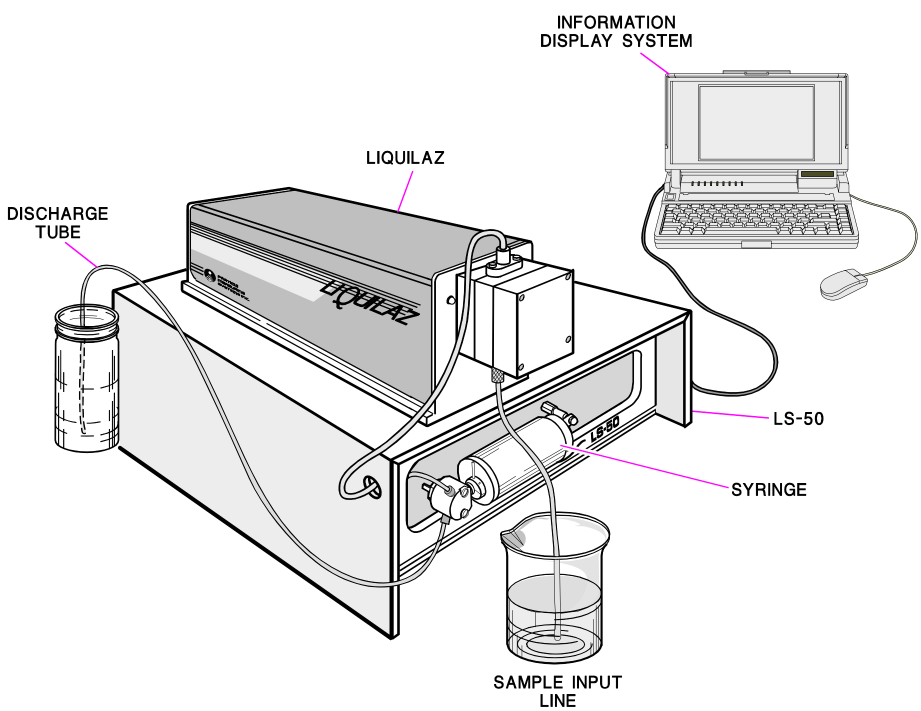

Figure 1

Optimizing the LiQuilaz®-S02 Sensor

The sensor must be optimized with regard to flow rate and sensitivity. A low flow rate of 10 milliliters per minute allows for the sampling for viscous fluids and reduces the possibility of cavitation. The sensor should be optimized and calibrated by the factory for this flow rate (standard sensors are designed to operate at a flow rate of 50 mL/min.).

Optimal sensitivity is indicated by a low DC light and a correct distribution of particles with respect to size. The DC light parameter is an indication of how much light is being scattered off of the sampled fluid. High levels of this background light can make small particles difficult to detect just as bright room lights can make a dim computer screen difficult to read.

The user can adjust the sensitivity so that the sensor detects small particles but is not confused by background light. This is indicated by the distribution of particles with respect to size. Particles in nature tend to be distributed as shown in Figure 2. The particles totals for each size bin decrease with the cube of the sizes as shown by the excellent fit of a third order function.

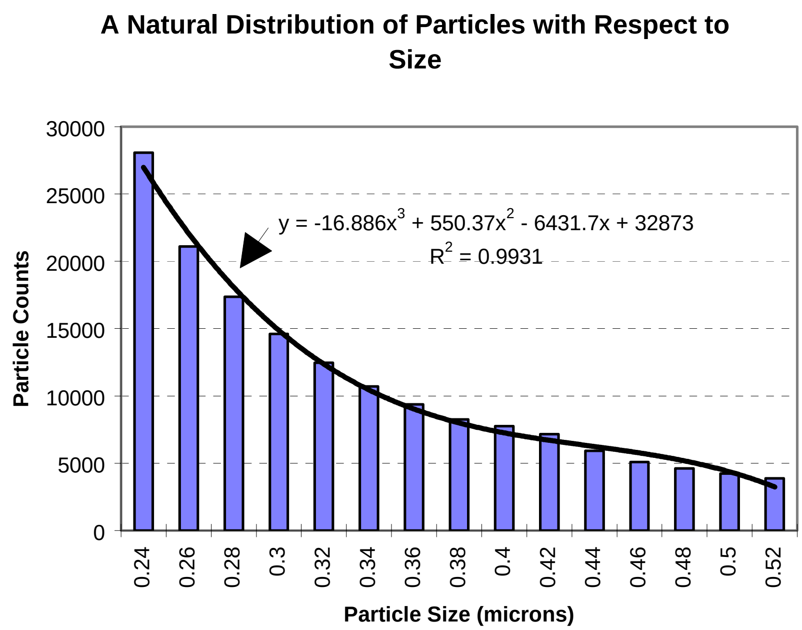

Figure 2

Figure 3 shows a particle distribution when the sensor is too sensitive. The particle counts in the first channel (size bin) are much higher than those for the other size bins. A third order function fits poorly as indicated by the low value of R-squared. A fifth-order function is needed to match the measured values.

By using the Operator Adjustable Threshold feature of the LiQuilaz® sensor, the sensitivity can be decreased slightly so that only real particles are detected. Figure 4 shows the particle size distribution when the same fluid is sampled with a minimum sensitivity of 0.22 microns instead of 0.20 microns. Again, the counts show a good fit with a third power function.

The user can be assured that the correct sensitivity has been reached when further adjusting the minimum particle size no longer alters the shape of the distribution curve. (For more information on using Operator Adjustable Thresholds see Service Bulletin SB950005 from Particle Measuring Systems).

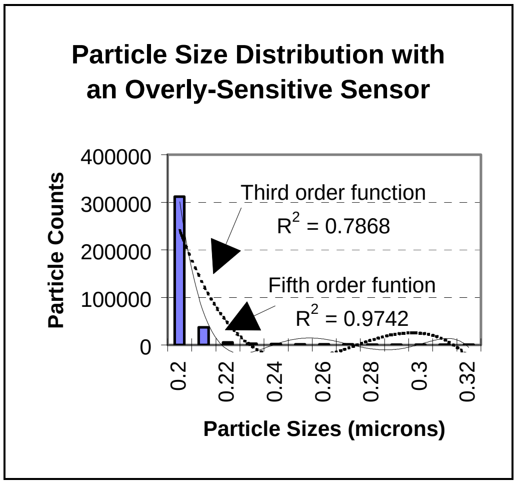

Figure 3

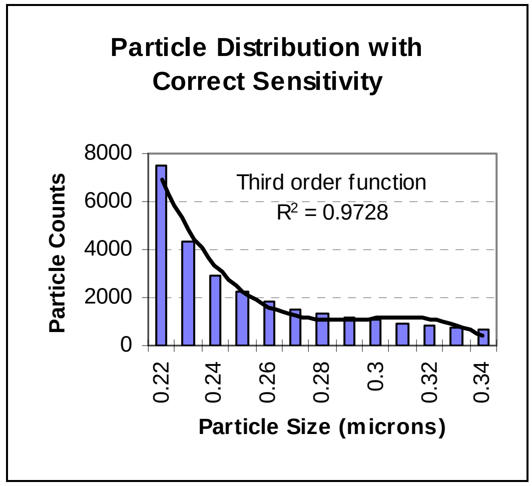

Figure 4

Testing Configuration

Eight different liquids were sampled with an LS50 and a LiQuilaz®-S02 sensor. The fluids were chosen because they were difficult to monitor with a debubbling sampler. Each sample had a volume of 10 ml. with a tare volume of 1 ml. Special inlet and outlet adapters were mounted on the LiQuilaz sensor. (option 90301015). This allows ease of tubing connection to the sensor. Inlet and outlet tubing lengths were about 24 inches and the tubing between the sensor and the sampler was about 18 inches. The tubing was a 3 mm outside diameter. A 25 ml. glass syringe was used. The system was operated with APPSVIEW software but could have used FACILITY VIEW or the recommended SAMPLER VIEW.

An acetone rinse was used between each sample to isolate the chemistries in the waste bottles. After each sample series, the instrument was allowed to draw two samples of acetone and the waste was delivered to the sample waste bottle. The inlet was then placed in the next product to be sampled. In a few cases, this transition caused the DC light to become exceedingly high, probably due to the coating abilities of the chemical allowing some residue to remain in the capillary of the sensor. In these instances, the unit was returned to a recirculating acetone rinse cycle until a low enough DC light level was attained to allow 0.2 micron sensitivity. The sampler was then returned to the desired sample. At no time was a sample allowed to remain stagnant in the sensor. The reason for this was to minimize the chance that the internal capillary of the sensor would be coated with the product being tested.

Results

Figure 5 shows the particle count results for eight fluids which are difficult to sample with other particle monitoring systems. For each liquid, the sensitivity was optimized using the method described above. At a flow rate of 10 mL/min a minimum sensitivity of 0.2 microns was attainable for each fluid except the AZ Anti-Reflective coating. Note that particle levels should not be considered characteristic of a manufacturer's product as the original sources of liquid were of various levels of cleanliness.

The LiQuilaz sensor required only a brief cleanup period between samples. Cleanup times for the various fluids were less than half an hour.

Table 1 shows the times for the first five fluids sampled. The cleanup time for the first liquid was the longest because the cleanup methodology was being developed during the testing.

Table 1

Media	Cleanup Time (minutes)
10i Olin Resist	30
620 Olin Resist	11
Brewer ARC	10
Agent T	20
Sumitomo Resist	15

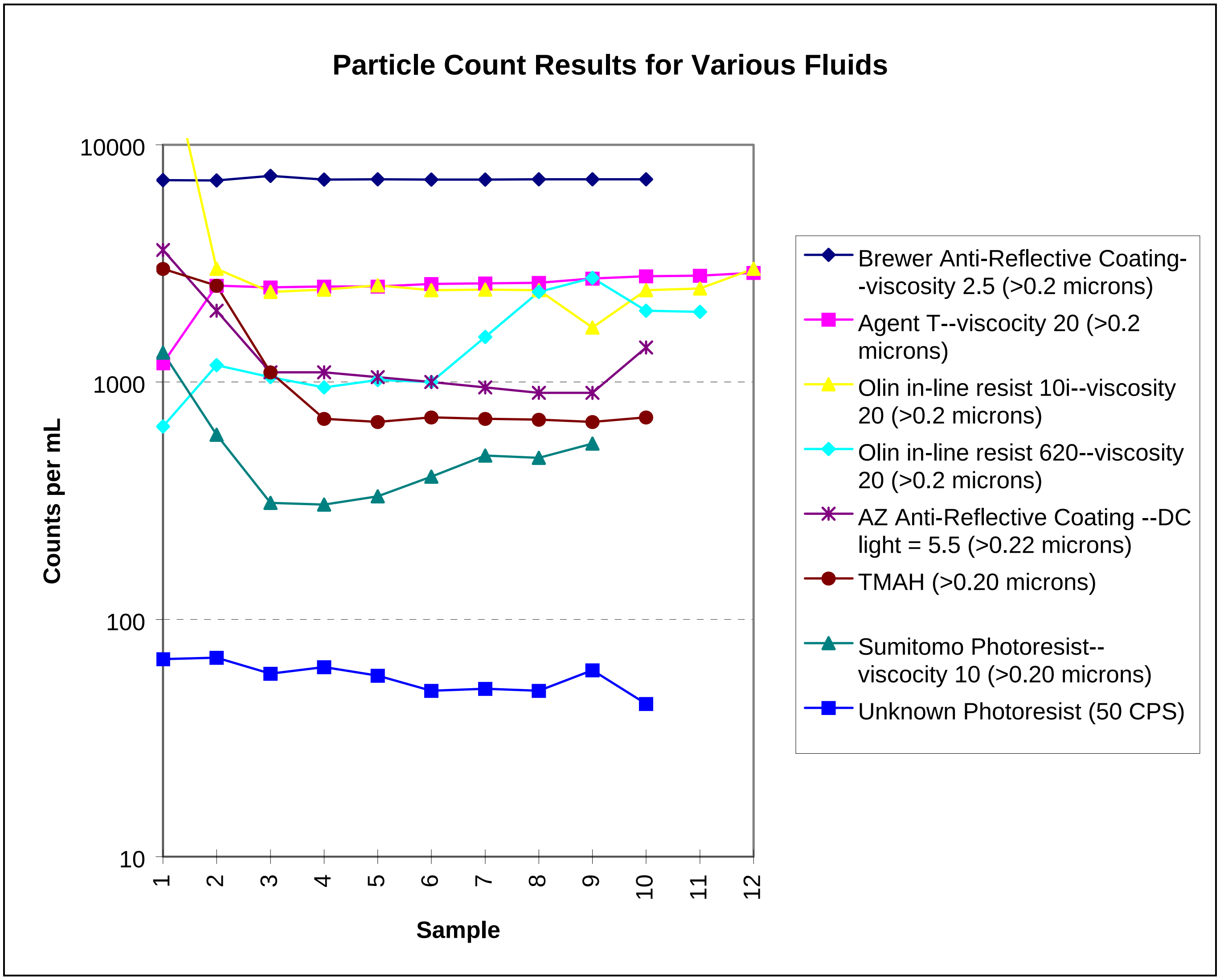

Figure 5

Conclusion

Photoresist and Anti-Reflective Coatings can be effectively monitored for particle contamination using an LS50 sampler and a LiQuilaz-S02 sensor if the sampler and sensor are optimized for these liquids. Optimization involves adjusting the flow rate of the sampler and the sensitivity of the sensor. An optimized system produces accurate, repeatable readings with short cleanup times between samples.

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