

A counting challenge

Dwight Beal and Jerry Gromala of Particle Measuring Systems look at optimising the performance of liquid compression samplers that utilise optical particle counters for the purpose of batch-mode measurements of effervescent and aggressive process chemistries

Table 1: Definition of the sequence of optimising a liquid compression sampler

<i>Sequence</i>	<i>Comments</i>
Set-up and Installation	Absolutely crucial for generating meaningful particle count data. The location of the sampler relative to the liquid that will be tested can significantly affect the time needed to perform the measurement as well as the results
Fill	Process of drawing the liquid into the sampler using vacuum
Compression	Step where pressure is applied to the sample, forcing bubbles back into solution prior to measuring particles
Tare	Important step prior to sampling, needed for the proper normalisation of data
Sample	Step where particle count and size data is generated
Inter-sample Delay	Used to control the amount of data collected and chemical used

Effervescent and hot process chemicals used in the semiconductor industry pose unique challenges when trying to measure particle concentration levels. Compression liquid samplers are widely used for both mobile batch sampling and continuous on-line sampling of these chemistries because of their ability to control bubble formation and minimise the effect they have on the resulting data. Samplers also offer tighter process control capabilities by monitoring in close proximity to the product, as in the case of a production wet-bench.

Liquid samplers must be optimised for the chemical of interest based on its viscosity, density, vapour pressure and effervescence. Physical parameters such as the location of the sampler from the chemical source, length and diameter of the sample tubing, and fluid elevation changes will affect the settings of the sampler, and therefore must be considered. It is not uncommon for a single sampler to be used with five to 10 different chemistries having vastly different physical properties. This broad challenge requires the operator of the

equipment understand that technique is a critical determinant in success or failure of the measurement. This article offers general guidelines based on field experience for configuring and optimising the performance of a liquid compression sampler. A detailed discussion defining a proven sequence for optimising a compression sampler is given, as well as suggested configurations for the most common process chemistries.

Compression sampling

Sampling chemicals and process baths for particulate contamination is an important part of ultra clean manufacturing processes. However, in many cases there is no reasonable method to provide a flow stream of chemical directly related to the product exposure site to an in-line style liquid particle counter. In these cases, a batch sampler is necessary to deliver the chemical to the liquid particle counter. A special type of batch sampler known as a compression sampler uses pressurised gas to force the chemical through a liquid particle counter, improving data quality by eliminating issues associated with bubble formation. The

ability to force chemical through the particle counter under pressure instead of pulling it through under vacuum like a syringe or pump system also allows the measurement of high viscosity fluids with the same instrument.

Because a compression sampler can be used with such a broad range of chemistries, it is important to properly determine the optimal settings for each chemical to be sampled. Failure to do so can cause increased data variability and produce less than optimal performance. Table 1 defines the sequence for optimising the configuration of a liquid compression sampler.

Set-up and installation

The location of the sampler relative to the liquid that will be measured will have a significant influence on the resulting data. In many instances adequate space is not available near the vessel where the measurement is to take place. Long lengths of tubing are then used to deliver the sample to and from the instrument. This is less than desirable because a fairly large volume of liquid can reside inside the tubing between the sampler and vessel. For example, a 4m length of 6mm diameter tubing will contain approximately 50ml of fluid.

Unless the sampler is designed to discard the first 50ml of fluid before drawing in the actual sample the measurement will include some of the fluid drawn from the bath during the last sample. This can also create timing problems. For instance, if the sampler must take two samples before it receives a representative sample from the vessel, but the product was in the vessel during the first sample, how does one correlate the particle data to process events.

Probably the most significant problem with long lengths of tubing is contamination remaining after the measurement is completed. The amount of time needed ►

◀ for tubing to clean up after a measurement is directly related to its length. Since the volume of liquid sampled in a batch mode is generally low, clean-up times can be significant after sampling dirty chemistries. Since the clean-up time is the limiting factor of testing throughput, this type of occurrence needs to be minimised. In some instances contaminated tubing is simply removed and disposed of rather than wasting the time to reduce background particle counts to acceptable levels. Tubing lengths less than 2m are generally suggested.

Equally important is sample location selection. In some wet bench processing, the particles generated are lighter than the fluid causing them to float. Monitoring for these particles anywhere but near the surface will result in artificially low readings. If the chemical contains heavy metallic particles, then the sample tubing should be placed near the bottom of the container. In most cases a general rule of thumb cannot be determined because too many variables exist. The only rule of thumb of value is to carefully consider the particles of interest and how to best find them.

The primary controls for a liquid compression sampler are generally located on the front panel of the instrument for convenient access. As previously discussed, adjustments will generally need to be made if sampling a different chemical is required or if there are physical changes to sampling setup. Fig. 1 shows the front panel and primary controls for a Particle Measuring Systems CLS-1000 liquid compression sampler. Adjusting the fill time, flow rate and compression pressure is accomplished with front panel controls. Setting compression delay, tare and inter-sample delay is usually made in software. All liquid plumbing connections are made from the rear of the unit.

Fill

Vacuum is used to draw the chemical from the source into the sampler. Increasing the vacuum reduces the time required for sample intake, or enables the sampler to aspirate more viscous fluids, such as sulphuric or phosphoric acid. Changing the height of the sample, i.e. from a container located on the floor to a container located at tabletop level or vice versa, may require changing the vacuum setting.

The time required to fill the compression sampler's internal sample burette is the primary control of the vacuum adjustment common to all compression samplers. Filling too fast can cause splashing which could trigger the level detector before the

Fig. 1: Example of a liquid compression sampler and PC used for control and data collection



burette is actually full resulting in an inaccurate sample volume. Alternatively, it can also cause cavitation points that may not be flushed properly to eliminate residual contamination from previously sampled chemicals. Both of these situations will result in unpredictable data variability. On the

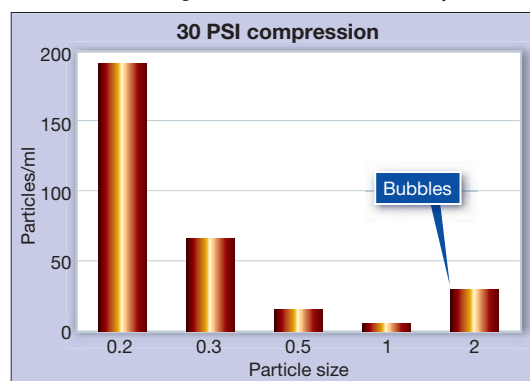


Figure 2: Particle distribution affected by bubbles

other hand, filling too slowly reduces throughput. A general rule of thumb is to adjust the sampler fill time to approximately 10 to 15 seconds using the vacuum control.

Compression

Compressing process chemicals prior to measuring particle concentration eliminates bubbles that would otherwise invalidate the resulting data or make interpretation of the data a more difficult task than it needs to be. This is particularly true for wet etch applications and chemistries with ozone or peroxide additives.

It is important to note that non-effervescent chemistries may still suffer from bubble formation. For instance, overflowing wet benches often entrain air in the fluid. The liquid particle counter counts these micro-bubbles as if they were particles. Therefore, it is always necessary to have

some pressure on all samples, and to be prepared to adjust it based on the chemistry. There will always be a greater number of smaller particles than larger ones given an ambient distribution. Any time a liquid particle counter displays a particle size distribution that includes more large particles than smaller size particles it is reasonable to expect that bubbles are present.

There are two parameters associated with the compression mode of a sampler: compression pressure and compression delay. A reasonable approach when working with a new chemistry is to start with a nominally low compression pressure and a short (15 seconds) compression delay. A good starting point for the pressure setting is 10 to 15psi. If the particle size distribution looks like Fig. 2 after a few samples, bubbles are likely still present in the liquid.

To determine if this is the case, the compression pressure on the sampler should be increased. In an unknown situation, it is suggested that the pressure be increased a significant amount initially, i.e. from 15 to 50psi. The pressure can be reduced until the distribution changes and bubbles just begin to form. It is a good rule of thumb to add an additional 5psi to assure bubbles will be eliminated as a final optimisation step and establish this value as the pressure setting for the chemical under test. Fig. 3 shows a particle size distribution optimised using the process described.

It should be noted that compression delay has less of an impact on eliminating bubbles than compression pressure, but is still an important part of optimising a sampler's settings. The following example illustrates the interaction between compression pressure and compression delay.

As stated previously, the first step to take when bubbles are present is to increase the compression pressure for several samples

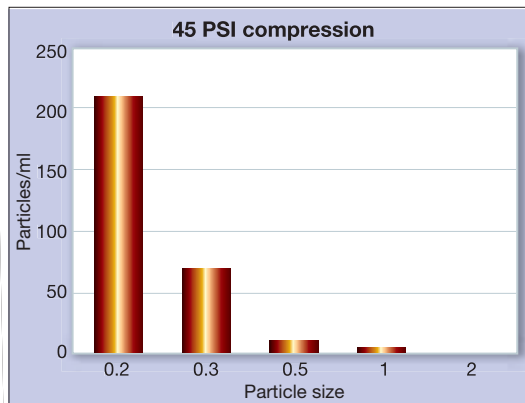


Fig. 3: Particle distribution that is free from bubbles

while monitoring the resulting distribution. Short compression delays (15 seconds) facilitate rapid sampling so more data can be generated.

If bubbles are still suspected to be present after reaching the maximum pressure the sampler allows, increasing compression delay is the next step. The quickest way to find the optimum setting that eliminates the bubbles is to increase the compression delay from 15 seconds to several minutes.

Again observe the size distribution for several samples. If the size distribution does not change, the sample does not contain bubbles. Return to the shorter delay time and continue sampling with confidence that bubbles are not affecting the results. If the distribution gets worse, additional bubbles are forming because of chemical reaction time, and a shorter delay time should be chosen than the original starting point. If the distribution changes for the better, begin reducing the compression delay until the bubbles reappear.

For best results, use a divide and conquer approach. Cut the compression delay in two, and observe several samples. If the sample distribution continues to look good cut the delay in half again. Continue in this fashion until reaching the point that bubbles are just beginning to form. Then add a 10% margin for variations between samples.

Finally, consider the application where viscous chemicals need to be sampled. Compression liquid samplers have been proven to sufficiently suppress bubble formation from occurring when dissolved gasses are present. There have been instances where viscous chemicals were either aggressively agitated, or forcefully aspirated into the sample container. In

these cases the bubbles formed were not the result of a natural occurrence, but artificial introduction. It is unlikely that any amount of compression pressure or delay will adequately suppress these bubbles to prevent the particle counter from reporting them. Only time to allow the bubbles to rise to the surface and exit the fluid will stabilise the data. A better solution is to transfer the fluid more carefully to prevent the introduction of the bubbles in the first place.

Tare time

The tare time is the delay between when the sample begins to flow through the particle sensor and when the sensor begins recording particle counts. The purpose of this parameter is twofold. First, it is to allow the short piece of tubing between the sample burette and the particle sensor to clear itself of the previous sample and present the particle counter with fluid from the current sample. Second, it allows the sample fluid to accelerate to the sensor's calibrated flow rate. This ensures that the sensor accurately reports particle size information during the particle counting interval. Generally, one to two seconds is sufficient for viscosities less than 10 centipoise. Experience has shown that five seconds works well for chemistries having viscosities greater than 10 centipoise.

Sample

All optical particle counters are calibrated at a particular flow rate that optimises the particle size measurement. Operating at flow rates other than that specified by the manufacturer will invalidate the measured data and make the overall task more difficult and confusing than it needs to be. Setting and maintaining the desired flow rate is straightforward for most online applications. It can be quite difficult however in a batch sampling mode with viscous process chemicals.

Experience suggests that the flow rate be confirmed for each liquid that is sampled after the fill and compression configuration steps have been completed. The flow rate should be adjusted to $\pm 5\%$ of the manufacturer's specification for best results.

Inter-sample delay

This delay will cause the sampler to delay between the beginnings of each sample for a designated length of time. The purpose of this delay is to reduce the number of samples taken in a given length of time, for instance, samples per day. It is an artificial method of slowing down the sampler from taking as many samples as it could normally take without the delay. This is probably the only parameter whose setting is completely arbitrary and depends upon the specific application.

The best approach

The most desirable plan when setting the compression pressure utilises the lowest pressure that will provide accurate data. This reduces stress on the internal components of the liquid sampler thereby reducing maintenance. Table 2 summarises liquid compression sampler settings for some of the more popular chemicals used in the semiconductor industry.

Effervescent and hot process chemicals used in the semiconductor industry pose unique challenges when trying to measure particle concentration levels. Eliminating bubbles from these chemistries allow particle contamination to be quantified, which aids in their production, delivery and use. This article has discussed the benefits liquid compression samplers provide, as well as a general guideline on how to optimise their associated configuration for best results.

Table 2: Suggested compression sampler settings for common process chemistries

Chemistry	Fill Time	Compression Pressure	Compression Delay
DI Water	≥ 10 seconds	30psi	10 seconds
Dilute HF	≥ 10 seconds	30psi	15 seconds
B.O.E.	≥ 10 seconds	55psi	60 seconds
SPM and SOM	≥ 10 seconds	55psi	5 seconds
Nitric	≥ 10 seconds	30psi	15 seconds
Phosphoric	≥ 10 seconds	50psi	30 seconds
SC-1	≥ 10 seconds	45psi	15 seconds
SC-2	≥ 10 seconds	45psi	15 seconds

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