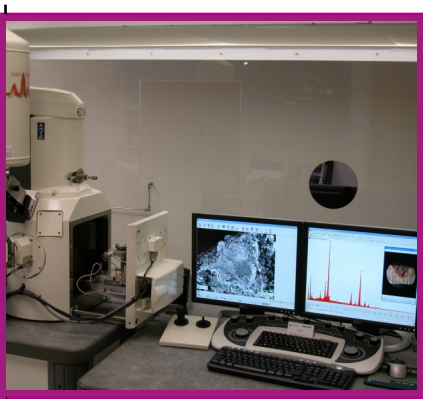
Particle Counts in Ultrapure Water



Particle Counts in UPW are Critical to Contamination-Free Semiconductor Manufacturing

As geometries shrink, particle sizing and analysis become even more important. Balazs™ NanoAnalysis offers several types of particle counts in ultrapure water. The Scanning Electron Microscope-Direct Count Method (SEM-DCM) is ideal for routine monitoring ultrapure water systems. Balazs provides SEM-DCM test, supporting specifications of ASTM D5127. This test is particularly important for the facilities, which do not use on-line optical particles counters (OPC) or need to validate the performance of the existing OPC instruments.

Scanning Electron Microscopy Direct Count Method (SEM-DCM)

The SEM-DCM is excellent for monitoring the overall efficiency of an ultrapure water system over time. It combines the advantages of on-line sampling with the power of counting at high magnification. Table 1 shows an example of a typical particle count performed by Balazs in ultrapure water. Note that the results include bacteria count and additional comments as well as error ranges. Furthermore, it is possible to obtain elemental composition of some of the particles by Energy Dispersive Spectroscopy (EDS). This information can be used to identify particles and locating their sources.

Table 1. Typical report from particle count performed in ultrapure water

Bacteria/100 mL	Particles/Liter				
	0.05-0.1 µm	0.1-0.2 µm	0.2-0.5 µm	0.5-1.0 µm	>1.0 µm
<1	140 +/- 8	35 +/- 2	12 +/- 1	24 +/- 1	32 +/- 2

*Typical Comments:

1. Even distribution
2. Edge weighted distribution
3. Center weighted distribution
4. Clumping (bacteria)
5. Clumping (particles)
6. Large particles (>5µm)
7. Small particle
8. Particles difficult to size
9. Particles difficult to distinguish
10. Pore clogging
11. Pore distortion
12. Filter mounted upside down

Note 1: +/- values are at 95% confidence level

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SEM-DCM Sampling

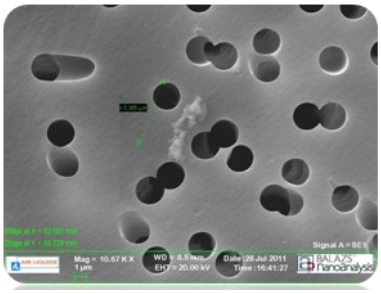

Figure 1. Online filter SEM particle

To prepare the sampling port, it is thoroughly flushed. Then the sampling device, a fluoropolymeric holder fitted with a polycarbonate sampling filter, is attached. The pore size of the filter can be 0.05 µm, 0.1 µm, or 0.2 µm. For final filter water, the recommended sample volume is 1000 liters.

Depending on the pressure of the water system and the pore size of the filter, sampling time may be anywhere from 3 to 30 days. If the pressure in the line fluctuates significantly, the flow must be monitored frequently in order to accurately determine the sample volume.

SEM-DCM Analysis

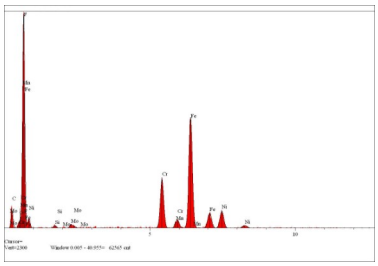

Figure 2. EDS of a Particle on filter

After sampling is completed and the filter holder is delivered to the laboratory, the filter is removed from the holder in a HEPA clean area. A thin coating of gold is applied to make the surface conductive. An area of the filter is then counted at 6,000 x magnification with a Scanning Electron Microscope (SEM).

At this magnification, there are approximately 487,000 fields of which up to 300 are typically counted, a statistically acceptable number. Results include particle size distributions and a total (live and dead) bacteria count.

The polycarbonate filters used can trap particles much smaller than the stated pore size making this method useful for counting very fine particles and colloids. At Balazs, an experienced SEM operator can spot subtle changes in the filter surface and/or clogging of filter pores, indicative of colloids.

Particle Analyses to Support State-of-the-Art Semiconductor Manufacturing: Nano Particle Capture Device (nPCD)

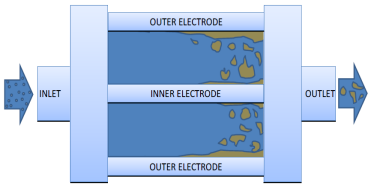

Figure 3. nPCD releases accumulated and agglomerated particles and directs flow to an SEM membrane for analysis

Balazs is actively involved in development of new methods and techniques via participation in ITRS and SEMI committee efforts. The latest particle monitoring advancement for ultrapure water incorporates an innovative sub-50nm particle agglomeration technique.

This test provides fast (less than 24 hour) collection of previously undetectable particles. When used with standard SEM EDS techniques, agglomerated particle composition data is available.

- The nano-particles agglomerate when they touch the electrode surface
- As UPW flows through the nPCD, a low-voltage electric field moves the charged nano-particles towards an electropolished stainless steel electrode
- After a “Capture” cycle the polarity of the electric filed is reversed, the agglomerated particles are released, swept out by the water flow and collected on a standard SEM filter

Optical Particle Counting Methods

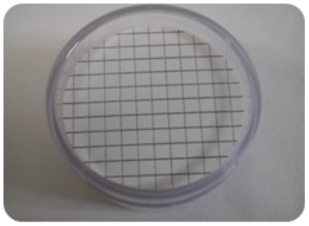

Figure 4. Optical monitor for particle counting

In addition to SEM-DCM, Balazs provides numerous other particle counting methods. They include rental service with the most advanced on-line particles counters, particles analysis in a grab sample (particularly important for the material cleanliness tests), and counting larger particles using optical microscope (i.e. ASTM F1094) method.