High Concentration Single Particle Sizing and Counting Technology

ACCUSIZER

The Accusizer FX
& FX~NANO~

Particle Sizing Systems

The AccuSizer FX and FX**NANO** are high concentration single particle optical size sensors (SPOS) that have the sensitivity to detect small differences in particle size distributions. They were designed to allow particle size monitoring to go from the R&D laboratory to the process line with a minimum to system reconfiguration.

The AccuSizer FX utilizes a focused beam technology that allows the sensor to focus on a specific area of the flow channel to count and size individual particles starting at 0.15 microns and at concentration levels exceeding 10 million particles per mL. These are concentration levels that are millions of times higher than traditional sensors. Although this type of approach has been tried by other techniques to monitor high concentration process streams it has been plagued by low resolution ad accuracy. The FX utilizes a patented electro-optical configuration that maintains the same levels of resolution and accuracy found in our traditional SPOS sensors.

The FX**NANO** provides a summation signal that extends the lower size limit of the sensor below the standard FX sensor. Classically one of the limiting factors in the lower size limit of single particle optical sizers was the need to find diluents that are clean enough so that they do not add or make up most of the background counts of the sample being analyzed. The tremendous increase in particle levels with the FX sensors makes it easier to reach lower sizes where background count levels are no longer a concern. In the past a few thousand counts in the background would cause great concern but now with concentrations exceeding 10 million a few thousand background counts do not affect the overall distribution. This becomes critical when looking at nano particles below 0.5 microns.

These sensors can also provide quantitative information on the stability of colloidal systems such as emulsions and dispersions. By determining the levels of coagulated droplets whose presence has a direct effect on the speed of separation the stability of product can be quantified.

The unique design also allows for a more stable platform that does not require the levels of cleaning and maintenance that standard light extinction sensors demand. A longer life span and ease of use make these sensors a perfect match for online monitoring applications.

These sensors can be used in conjunction with our existing fluidics modules by employing a few small upgrades to the electronics and software interface. They can also be combined as a integral part of our new fluidics modules that take better advantage of the high concentrations and features that the sensors provide to our particle size analyzers.

These unique sensors can be combined with a host of new automated sampling systems to provide extremely accurate size and count information on a variety of colloidal systems. They possess the sensitivity to detect low levels of aggregates several standard deviations away from the mean of a distribution. These LPC (Large Particle Count) levels are often a key indicator on the quality of CMP Chemical Mechanical Planarization slurries and their presence has been directly linked to damaged wafers (scratches/chip defects) in the semiconductor process line.

The new AccuSizer FX technology takes this measurement one step further by making the measurement more robust and easier to perform at much higher particle concentrations. Figures 4-6 illustrate the use of the FX technology when monitoring the tail of a silica based slurry at FULL CONCENTRATION (NO DILUTION). The slurry was analyzed from a single point in the process taking multiple measurements to illustrate the sensitivity and reproducibility of the measurement technique. Figure 4 is a graph of the complete distribution on scale from 0.7 to 20 microns; note one overlay is almost indistinguishable from the next. Figure 5 is the same graph but expanded by a factor of 1000 on the y-axis to highlight the low levels of oversize particles greater than1 micron. Again, the reproducibility is superb even out at 10 microns where the statistical sampling due to the low levels of particles is very poor.

Figure 1 shows a trimodal mixture of a 1, 1.3, and 1.5 latex standard with reduction in the amount of the first peak and by half in each subsequent figure. Note the sensitivity is maintained even in the presence of a large particle peak.
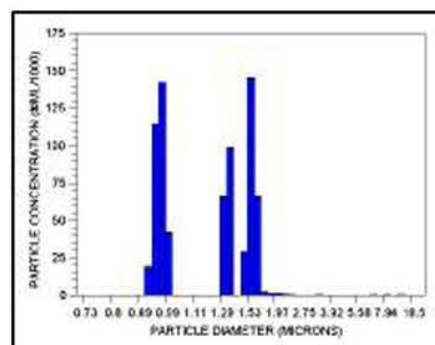

Figure 1 TRI-MODAL Mixture of 0.5 uL 1-mm + 1-uL 1.36-mm + 2-uL 1.59-mm latex (orig.).
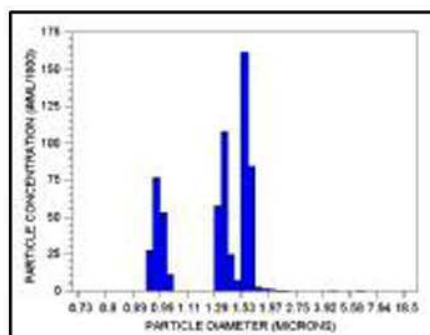

Figure 2 TRI-MODAL #1 - 0.25 uL 1-mm + 1-uL 1.36-mm + 2-uL 1.59-mm latex (orig.). Volume of small particle size standard cut in half 0.25 ul.
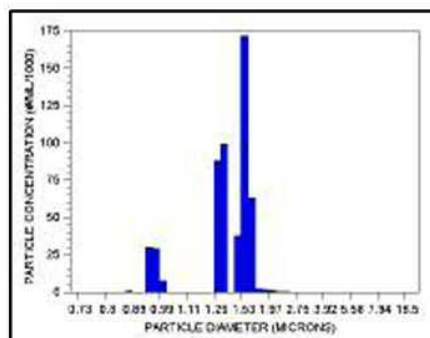

Figure 3 TRI-MODAL #1 - 0.25 uL 1-mm + 1-uL 1.36-mm + 2-uL 1.59-mm latex (orig.) Volume of small particle size standard cut in half again 0.125 ul.

Figure 6 is a tabular representation of the data where certain trend sizes were being monitored. What is important to note is, in a full concentration CMP Silica slurry sample (12% solids content), the AccuSizer FX was able to accurately count over 800,000 particles in one size class while having the sensitivity to count less than 10 particles in another.

The AccuSizer SPOS technology has played a major role in the Chemical Mechanical Planarization (CMP) market where the use of CMPs is important in the production of highdensity integrated circuits. CMPs are used in many stages of the production process, often complex slurry distribution systems are employed to transport these slurries from their holding day-tanks to the various points of the process where they are used. Slurries must be handled extensively and are exposed to many different environments. All of these issues impact thestability of the slurries, which often results in aggregation or contamination from the various filtration and pumping steps in the process. The AccuSizer has been an effective measurement technique in determining the low levels of out-of-spec particles which have been directly linked to the yield of the chip manufacturing process.
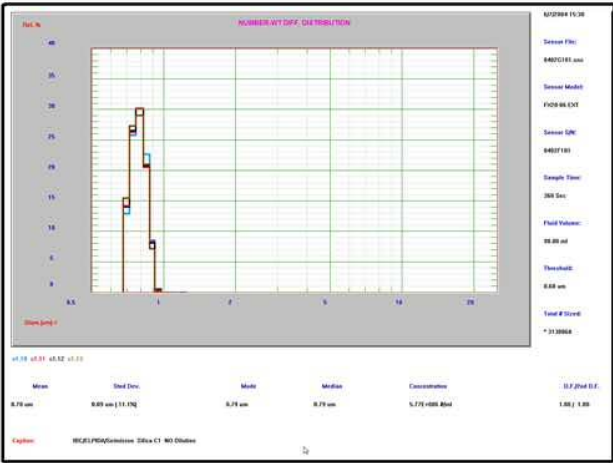

Figure 4: Silica C1 Overlay of the PSDs obtained from four consecutive analyses The agreement at the lowest size channels (0.72- and 0.82-mm) is good, considering the significant influence of fine-particle coincidences expected at these small sizes.
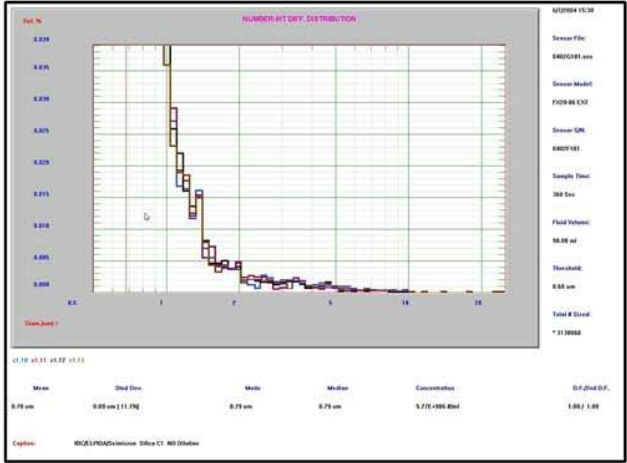

Figure 5: Silica C1 Same as Fig. 1, but with an additional 1000´expansion of the Y-axis (total of 1000´). Very good reproducibility is confirmed above 1.04-mm (details below).

Size Microns	Run1 Part/mL	Run2 Part/mL	Run3 Part/mL	Run1 Particles Sized	Run2 Particles Sized	Run3 Particles Sized
0.82	1,458,992	1,407,491	1,489,869	857,461	827,370	844,558
1.04	2,835	2,720	2,493	1,923	1,849	1,697
1.92	352	276	284	315	247	255
5.1	85	65	79	89	67	82
9.4	10	6	15	13	8	19

Figure 6: Silica C1 Differential data reported in particles per mL and total particles sized at 5 common trending points.

FEATURES:

AccuSizer FX

High concentration over 1 Million / ml
Wide dynamic range : 0.7 – 20 microns
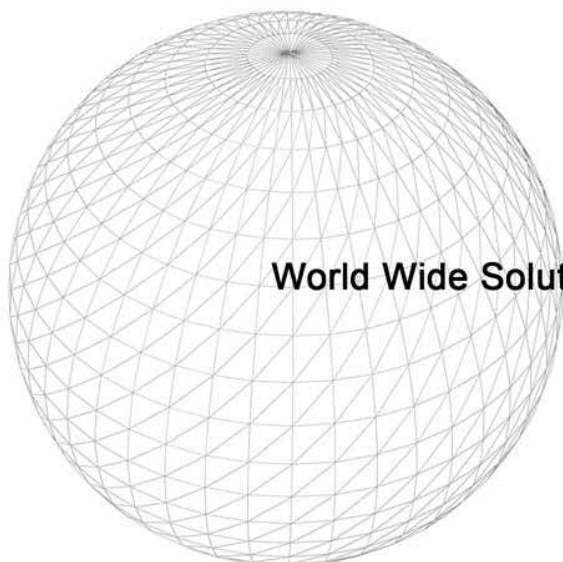
Applications:

CMP Slurries
Inks, Pigments
Emulsions
Dispersions
Pharmaceutical Injectables
Stability Analysis
Ceramics / Abrasives
Polymers
Nano Particles

AccuSizer FXNANO

High concentration over 10 Million / ml
Wide dynamic range : 0.17 – 20 microns

Sampling Systems:

Lab Small Volume Syringe Sampler
Single Stage Autodilution System
Online Point of Use Monitor
Online Multi-Point Multiplexing Monitor
Batch Auto Sampler

World Wide Solutions