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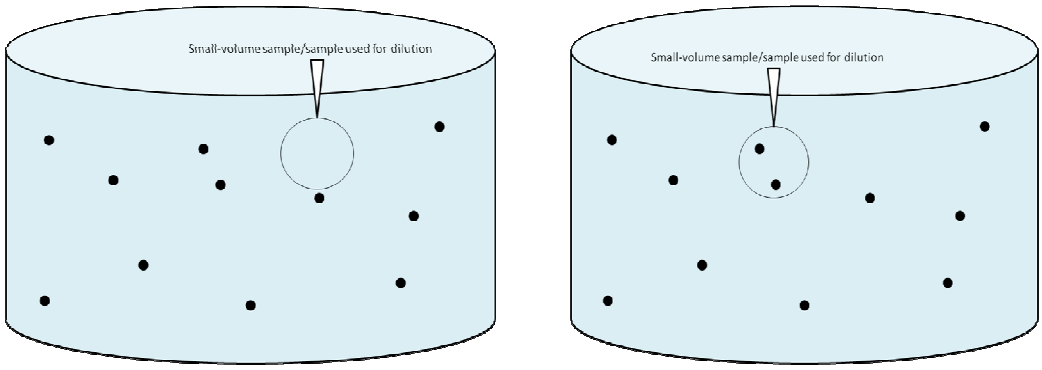
The Perils of Low Particle Counts

January 23, 2012

Traditional methods of measuring the large particle counts (LPC) in CMP slurry rely on dilution to reduce the number of particles passing through an optical sensor at any one moment to a manageable level. To maintain reasonable productivity, a slurry sample <1 ml is diluted 10x to 10,000x and only a portion of the diluted mixture is measured to represent to bulk slurry particle size distribution (PSD). Dilution with water can change the chemistry and the tendency of particles to agglomerate, possibly contributing to misleading albeit consistent results. Sample dilution will not be addressed in this Applications Note.

The combination of small sample size, dilution and measuring less than 100% of the diluted sample volume can lead to irreproducible results, depending on the particle size range. Particles smaller than 1 µm are abundant and are well represented in samples of any size. Particles in the range of 1 µm to 5 µm are less abundant and are not as well represented statistically in the liquid volume measured. Particles larger than 5 µm are even less abundant and are likely to appear as discrete random events from one sample to the next, if they are detected at all. This is illustrated in the following diagrams.



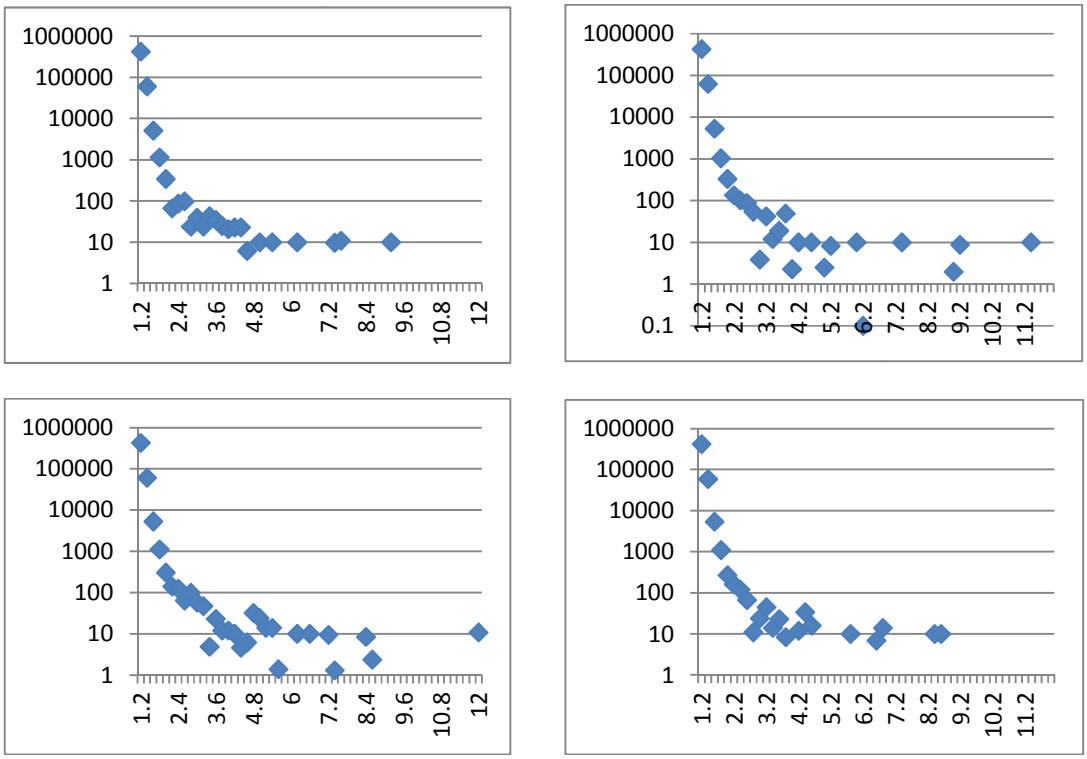
For large dilution factors, most LPC samples will look like the diagram on the left. The odds of capturing a rare large particle are low, even if there are hundreds of particles per milliliter of slurry.

The SlurryScope measures undiluted slurry continuously at a rate of 15 ml/minute. For a data collection period of 1 second, this corresponds to data for 0.25 ml of slurry per line of data in the bin file. This is comparable to a typical sample volume used for AccuSizer measurements, though the SlurryScope measures 100% of this volume without dilution. A segment of a representative bin file data set for ceria slurry is shown in the tables below. Each column represents a specific particle size bin, and each row represents the total number of particles per milliliter measured during the 1 second interval (equivalent to 0.25 ml measured volume). The columns on the left represent particle counts in the range of 1.0-2.6µm. These counts are relatively consistent from sample to sample, though the

2.6µm bins are already noticeably more variable than the 1.0µm bins. The columns on the right show data for the 10.2-11.8µm bins. Many of the data entries are zero, analogous to the behavior of small volume sampling as commonly practiced for AccuSizer measurements. However, particles are detected on occasion, leading to a high variance for the collection of measurements.

1.2	1.4	1.6	1.8	2	2.2	2.4	2.6	10.4	10.6	10.8	11	11.2	11.4	11.6	11.8
426003	60079	5919	1089	923	382	89	55	0	0	0	10	0	0	0	0
422701	60814	5606	1027	306	103	86	67	0	0	10	0	0	0	0	0
425011	59475	5518	1204	356	130	132	59	0	0	0	0	0	0	0	0
422218	60429	5187	1149	848	67	87	98	0	0	0	0	0	0	0	0
426193	59804	5458	1217	417	164	110	56	0	10	0	0	0	0	10	0
422450	58841	5297	1040	283	82	87	23	0	0	0	0	0	0	0	0
428492	59527	5423	1293	388	197	155	60	0	0	0	0	21	0	0	0
425466	59205	5351	1146	290	119	118	28	0	0	0	0	0	0	0	0
424718	59069	5211	1141	377	157	58	44	0	0	0	0	0	0	0	0
423875	62059	5273	1091	991	135	102	88	0	0	0	0	0	10	0	0
424832	58876	5725	1341	347	69	78	66	0	0	0	0	0	0	0	0
424921	61402	5124	1016	828	109	90	66	0	0	0	0	0	0	0	0
425888	59954	5060	1105	887	111	61	45	0	0	0	0	0	0	0	0
425503	59088	5056	959	302	160	70	56	0	0	0	0	0	0	0	0
424301	58808	5248	1079	311	93	54	80	0	0	0	0	0	0	0	0
426447	60634	5287	1109	305	140	121	64	0	0	0	0	0	0	0	0
426835	60968	5483	952	340	191	102	67	0	0	0	16	0	0	0	0
421106	60913	5558	1089	868	114	87	77	0	0	7.57	5.74	0	0	0	0
420119	59380	5473	1207	405	175	149	49	0	0	0	0	0	0	0	0
426245	61286	5526	1351	975	117	60	82	0	0	0	0	0	0	0	0
425984	58961	5020	969	814	121	56	68	10	0	0	0	0	0	0	0
421781	59382	5488	1103	271	162	120	67	0	0	0	0	0	0	0	0
428509	59148	5520	1162	559	101	94	58	0	0	0	0	0	0	0	0
423015	59581	5250	1068	351	160	119	132	0	0	0	0	0	0	0	0

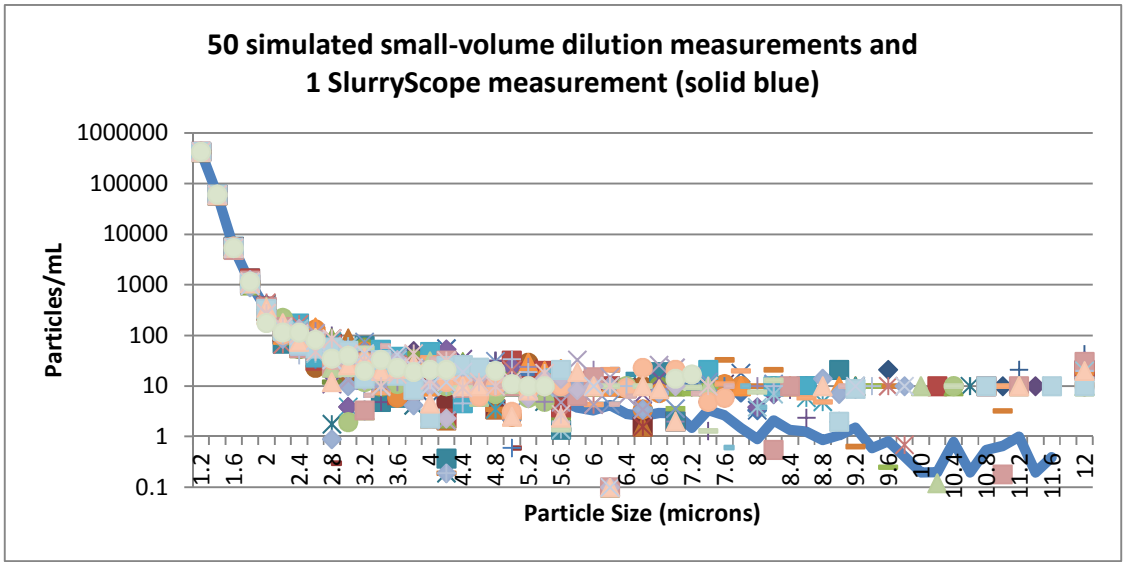
This increase in sample-to-sample variability can be visualized by treating each line in the SlurryScope bin file as a separate measurement. This is comparable to the 0.25ml sample size that is typical for individual AccuSizer measurements. In such a small sample volume, the variability in large particle counts from one individual measurement to the next becomes apparent, and is illustrated in the four charts below.



Each one of the charts above represents one line of SlurryScope data from a sample size of 0.25ml. Unlike an AccuSizer, the SlurryScope measures this sample volume without

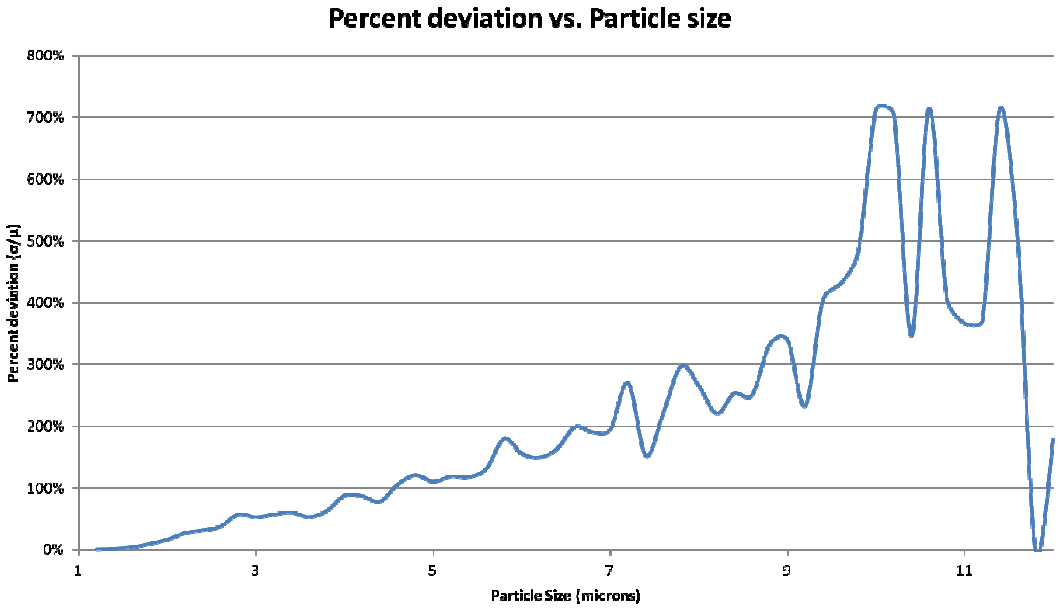
dilution in one second. As expected, the reported data is comparable from sample to sample for small particles which are numerous. As particle size increases and the counts drop toward single digits, the variability from sample to sample reflects the decreasing likelihood that an individual sample will provide a good statistical representation of the actual distribution of large particles in the bulk slurry. Recall that the SlurryScope, like the AccuSizer, reports the data as particles per milliliter, not as integer particle counts. This back-calculation explains the tendency for the data to appear to cluster around certain values for single digit particle events.

The 50 lines of SlurryScope data are plotted together on a single chart in the graph below, where the solid blue line is the SlurryScope PSD reported for the same data set.

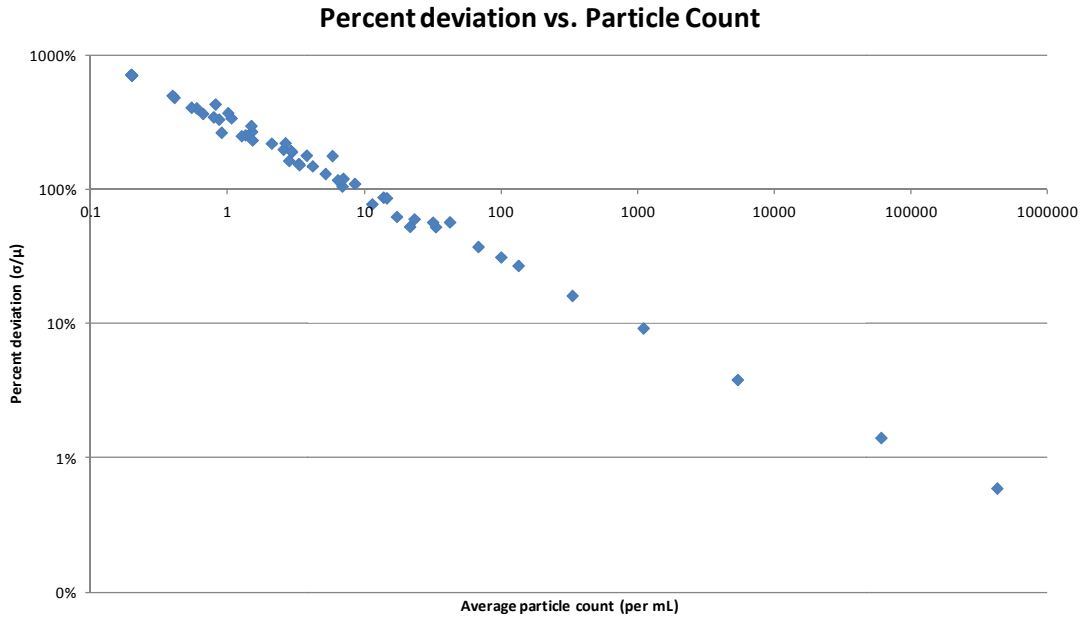


As expected, the data overlay well below 2 µm. Scatter increases in the 2-5 µm range as the particle counts decrease. Above 5 µm, the data becomes discontinuous as the number of zero bin counts per sample increases. The SlurryScope data shows that there is a low average number of particles per milliliter, as expected for a well behaved CMP slurry. The calculated particles per milliliter in the individual small sample volume data lines gives the impression that the large particle counts are much higher than they actually are, because each data set fails to take into account how many data lines detected zero particles. Only by accumulating the data over a large number of 0.25 ml samples is it possible to obtain a statistically meaningful representation of the LPC population in the bulk slurry.

The intuitive increase in data scatter at larger particle sizes and lower particle counts can be validated by representing the data set in a different way. The % deviation (ratio of the standard deviation to the mean) of each column of data (each particle size bin) is plotted below against the particle size, showing that the variability increases steadily up to about 9 µm. Above that, the counts are so sparse as to appear to fluctuate randomly. This would be expected to smooth out with a significantly larger data set.



The % deviation of each column of data (each particle size bin) can also be plotted against the average number of counts for a particle size bin. The results shown below confirm the intuitive expectation cited earlier: the variability from sample to sample is small when the particle counts are large, and the variability can become very large when the particle count is very low.



These data indicate that particle counts per milliliter as high as 100,000 are necessary for 250 µL samples in order to have sample-to-sample reproducibility of 1%. By the time the particle counts per milliliter drop to 1,000 – still a very high number in terms of potential wafer defects – the sample-to-sample variability is already up to 10%. Sample dilution

will further reduce particle counts per milliliter by the dilution factor, pushing the data to the left in the chart above, where the % deviation from sample to sample is even larger.

The accurate detection of rare large particles that are representative of the true LPC distribution present in the slurry is only possible when measuring a large total volume of slurry. The SlurryScope can fully characterize a discrete slurry sample using 15-25 ml of undiluted slurry in 1-2 minutes. This volume equates to 60-100 AccuSizer samples, but the LPC results will only be equivalent if the entire volume of the diluted samples is measured. However, the data above show that a count of 1,000 particles is required to achieve a 10% deviation in measurement results from sample to sample. In the 5 µm particle size bin, for example, this calls for a minimum sample size of 100 ml of undiluted slurry, the equivalent of 400 or more AccuSizer samples. The SlurryScope can complete this measurement in less than ten minutes, and the measured slurry volume can be returned to the slurry supply with zero waste because dilution is not required.

Continuous SlurryScope monitoring over the equivalent many of thousands of AccuSizer measurements provides a practical means to ensure that a slurry is acceptably free of large particles without slurry waste. Relying on small volume sampling to determine that a slurry is free of large particles is a high risk proposition.