CMP Slurries

AccuSizer® SPOS System

THE EFFECTS OF CHEMISTRY AND ENVIRONMENT ON CMP SLURRIES: STABILITY AND TAILS

Chemical mechanical polishing (CMP) is an important set of steps in the production of high density integrated circuits. Because CMPs are used during various points in the process for usually short periods of time, they must be handled extensively and are exposed to many different environments. New batches must be prepared often from concentrate kept in storage. All of these issues impact the stability of the slurries, which in turn effect the yield levels of chips. What is required is a measurement technique that is sensitive to low levels of out of specification particles, which can be the result of contamination from handling or aggregation from applied stress. This technique would need to be used to do quality checks on fresh material, and have the ability to be placed online to monitor the slurry in situ. The goal of this paper is demonstrate the utility of single particle optical sizing (SPOS) for providing quantitative information about CMP slurry health.

CMP slurries are becoming important to the creation of high density integrated circuits, particularly PC processors and DRAM chips. There are two critical performance parameters for slurries: removal rate, and number of defects. As it turns out, both are dependent on the colloidal stability of the slurries. As the primary particles begin to aggregate, larger particles are formed. These outliers scratch wafers, consequently increasing the number of defects. These particles also are more likely to settle, lowering the percentage solids of suspended materials. This causes removal rates to decrease. The list of variables affecting slurry stability is long: pH, ionic strength, environmental exposure, contamination, zeta potential, and so on. In order to meet the ever escalating demands placed on CMPs, slurry stability must be monitored. There is only one established tool to determine the stability of colloidal systems and that is SPOS. SPOS can detect the presence of aggregates because it counts individual particles. By counting and sizing particles one at a time, excellent sensitivity can be achieved.

The SPOS technology can be utilized online or in the QC laboratory. It combines ease-of-use with the ability to obtain quantitative information about large particle tails. Several other technologies have been offered as solutions to slurry monitoring but all are ensemble techniques, which inherently have lower sensitivity, and are unable to provide quantitative information about actual amounts of particles. Laser diffraction is one such technique, and its use with CMPs is discussed in Application Note 168.

Another is acoustic spectroscopy, which uses sound waves to obtain particle size information. A relatively new technology, it, like laser diffraction, is an ensemble method and claims to make measurements at high concentrations. In a recent paper, several CMP slurries were examined by acoustic spectroscopy. It was determined that the acoustic spectroscopy had a detection limit of 1% relative solids content. But it has been determined by SPOS measurements that CMP slurries with large particle solid fractions as low 0.005% can produce unacceptable decreases in yields.

In this paper, CMP slurries were subjected to various chemical and mechanical stresses were tested with SPOS. The results will show that SPOS has the sensitivity to see differences in large particle counts brought on by handling which can adversely effect chip yields.

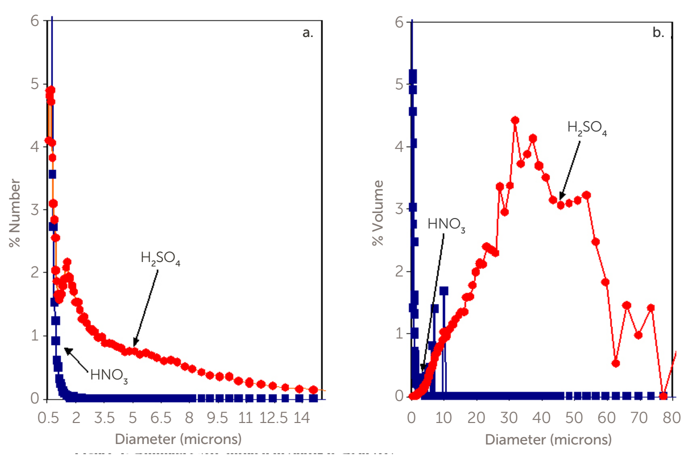

Figure 1. Alumina CMP diluted in sulfuric acid (red, circle) and nitric acid (blue, squares); a. Number weighted PSDs; b. Volume weighted PSDs.

CHEMICAL EFFECTS

Figure 1 contains the data obtained from an alumina slurry concentrate diluted in H_2SO_4 and HNO_3. The pH of each diluent was adjusted to a value of 3, so that the acidity was identical for each measurement. However, the results were quite different, where the slurry diluted in H_2SO_4 has a much broader tail than the slurry diluted in HNO_3. This effect is more dramatic in the volume weighted particle size distributions (PSDs). In H_2SO_4, particles as large as 70 microns are present while in HNO_3, there are no particles greater than 10 microns. So H_2SO_4 causes the slurry to aggregate, or flocculate, in an extreme way. As mentioned above, the pH value was the same for each measurement so this is not an effect of acidity. But the two diluents do differ in ionic strength. Why is this result important? Slurries must be prepared from concentrate before use; usually a chemical component is mixed with an abrasive component. It is important that the proper mixture is prepared. If not, the ionic strength might be out of specification, and this might cause the slurry to become less stable. It is much more difficult to test for ionic strength than it is to test for large particles, so SPOS provides an easy way of determining whether a new batch of slurry was properly mixed.

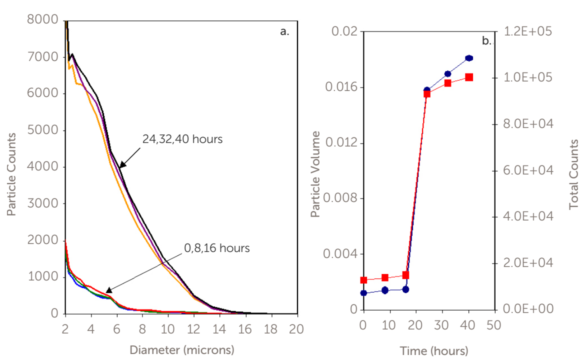

Figure 2. a. PSDs of a silica slurry after being pumped through a recirculation system for 40 hours; b. Graph of percent volume and cumulative particle counts while in recirculation system.

STRESS EFFECTS

Figure 2 contains several PSDs obtained from a silica slurry that was pumped through a recirculation system to test the pump's affect on the slurry. As can be seen, for the first 16 hours there was no noticeable change in the PSDs of the slurry. The total number of particles greater than 2 microns was steady at 10,000 and the volume percentage contributed by the tail was about 0.002%. But after 24 hours of pumping, the slurry started to show signs of breaking down. The number of particles jumped to 100,000 and the volume percentage contributed by the tail increased to 0.016%. This is an important result since it demonstrates that stress brought on by pumping can make slurry become unstable. Not only that, but this change was only on the order of hundreds of a percent so it would not have been detected by any commonly used ensemble method.

ENVIRONMENTAL EFFECTS

Figure 3 contains the PSDs obtained from a cerium oxide slurry that was subjected to three different temperatures: 30, 70 (room temperature), and 100 degrees Fahrenheit. As can be seen, the number weighted distributions at each temperature are almost identical but differences exist. The slurry sample that was cooled to 30°F aggregated, whereas the ambient and warmed samples did not. This is seen most dramatically in the volume weighted PSDs, which show that the 30°F sample has particles as large as 20 microns, and the others have no particles larger than 5 microns. This is an important result because the extremely trace amount of aggregate, produced significant wafer scratching and was of great concern to the user. And to reiterate, ensemble methods would not have detected this difference.

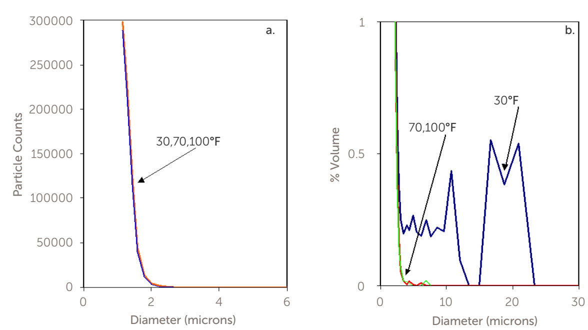

Figure 3. a. Population distributions of cerium oxide slurry at 30,70, and 100° F. b. Volume weighted PSDs of cerium oxide slurry at 30, 70, and 100° F.

CONCLUSION

Collectively, all of this data clearly demonstrates that slurries can be adversely effected by the conditions they are subjected. Whether its aging brought on by use, incorrect preparation, or being stored in inappropriate conditions, slurries will be degraded to a point where they produce unacceptable amounts of defects. Only by routine testing, of the incoming materials as well as monitoring the slurry in use, can these situations be detected before batches of wafers are ruined. The fact that CMP slurry is extensively used and will be used even more in the future, emphasizes the need for testing. This paper demonstrates clearly that SPOS has the capability to quantitatively measure the extent of aggregation and contamination of CMPs slurries which have experienced various kinds of stress.

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