In-line size and concentration monitoring of CMP slurries

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The CMP process is commonly associated with a high consumption of water and slurries. For processes of slurry preparation, quality control, control of slurry aging and plant parts wearout are very poor. As a consequence, only a small number of measurement options are available. Results of in-line concentration measurements with an optical device are presented for commonly used Silica, Alumina and Ceria slurries. Issues of accuracy, reproducibility and detection of drifts are discussed. A detailed study of the density influence between particles and fluids using another device to monitor the particle size in ceria slurry was caring out. Finally experiences using this technique for wastewater treatment and segregation will be discussed.

Keywords : control, concentration, size, slurry-blending, distribution plants

1. INTRODUCTION

The CMP process step in the semiconductor manufacturing has gained greater importance during recent technology enhancement [1]. When focusing on the polishing process itself, questions of productivity, yield enhancement, uniformity, removable rate, selectivity, defect reduction, reduction of machine downtimes, etc. commonly arise. The slurry supply chain is often treated as a black box and/or slurry quality as given constant. In practice, a number of influences shape the properties of the CMP slurry during the production, delivery, storage, mixing and supplying steps. The validated slurry coming from the producer can undergo some modification on the way to the waver. A slurry "story" will be written. The temperature during slurry transport is mentioned as parameter influencing the particle stability [2, 3]. Other sources emphasize effects due to the re-circulation period prior to consumption of the slurry. The pressure variation in the supplying system can support formation of particle agglomerates [4, 5]. The blending of slurry is another source for variations in slurry quality. The slurry blend ratio will be defined by volumetric or mass dosage. This dosage does not take the real solid concentration into account. This may not be important for slurries with simple handling. Already for tungsten polishing steps the slurry reactions are significantly more complex. Furthermore, high-density contrasts between particles and fluid, as it is the case for ceria, support gravitational settlement of the abrasive particles. Additionally, inhomogeneities in the slurry concentration may occur. If the slurry is sensitive to shear forces (>100 turnovers before slurry consumption in fab operation are common) the tendency to agglomeration will be heightened. Finally, hardware aging, e.g. of valves and pumps, can modify flow rates and dosage volumes. As a consequence, fluctuations around the demanded qualities are very likely.

2. TESTED MEASUREMENT DEVICES

The in-line concentration measurement of nanoscaled particles is not as simple as it may appear at first sight. Most of the commercially available off-line laboratory measurement methods cannot be applied to in-line control applications. On-line concentration measurement techniques used for other materials, such as high frequency ultrasound are no longer sensitive for particle sizes of below 200 nm. Density based measurement (Coriolis) systems overreact if bubbles are present in the pipe [6,7]. An interesting alternative represents three devices from Aello (1551 – concentration monitor, 1560 size monitor, 5000 wastewater segregation tool). The device setup is focused on in-line detection of changes in the slurry behavior. Only one relative value will give by the instruments (concentration or size index). The devices are for fix installation in the slurry supply plants.

This pragmatic approach results in the advantage of low operating costs and a reasonable purchase price. The Aello 1551 is able to determine the concentration of nano scaled particles as well as the type (material) of of solid. The optical backscattering measurement principle employed by the device can applied from ~ 0,05 – 20 weight-%. For applications in wastewater the Aello 5000 was tested. The measurement principle is similar to 1551, but it is caring out contact free (maintenance free). Also a sieve and a switch is integrated for automatic wastewater segregation. The size measurement in the Aello 1560 uses spectral extinction technology. It will be evaluate a ratio of extinction of different wavelengths strongly. This extinction ratio depends on the size of the particles. It is advantageous that the ratio is independent from the concentration of the particles. A size index will de detection between ~ 90 nm - 2 µm.

The exact ranges and resolutions of the devices depend on the optical properties of the slurry [7]. In this case, the system will be adjusted to the particular slurry properties. The light source and the detector are accommodated in a flow through sensor. The measurement methods are insensitive to gas bubbles and exhibits long-term stability [8]. The devices were tested first in a laboratory setting through measurement of different slurries. After that the devices were fixed installed for or after polishing tools (e.g. fig. 1).

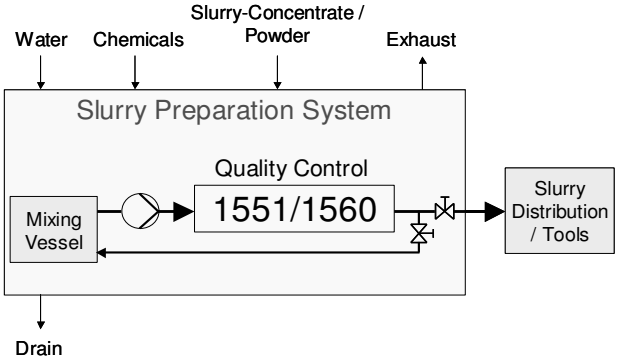
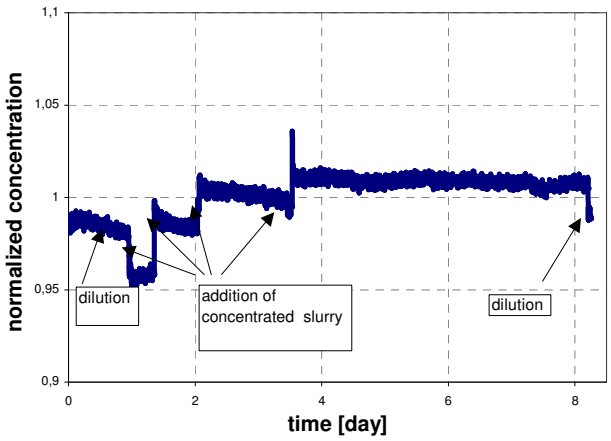


Fig. 1. Integration of measurement devices Aello

3. Results

3.1. Concentration control in blending plants

The concentration measurement was tested in Alumina slurry in laboratory. An accuracy of < 0,3 % was found. In general, it can be assumed that the device reports values with a relative error precision of < 1 %. Fix installed in the blending plant, the measurement results reproduce the high concentration resolution found in the lab experiences. Even very small changes can be detected. The diagram in figure 2 a) depicts the effect of subsequent addition of ultra pure water and highly concentrated slurry. The changes in concentration can be seen as expected. Additionally, the time for homogenization in the loop can be inferred from the graphs. The high-density contrast between particles and liquid using Ceria slurries (water 1, ceria 3) makes slurry handling more complex. The blending and distribution plants must deal with a larger tendency of settling and demixing of the particles. Experience shows that concentration variation may occur in non-optimal flow circumstances. An example for such an application shows in figure 2 b). It can be seen that over the first five days of measurement a drift in concentration occurs. The concentration changes about 5 %. The simple correlation between dosage and concentration as observed in figure 2a) was not found. The particle system's reaction is much more complex, and the mixing plant sports amore sophisticated design. Later in the study, better slurry homogenization and the direct control of the mixing process by the measurement device reduced the concentration variation to +- 1% (day 6-20).
1551/1560 in slurry blending plants



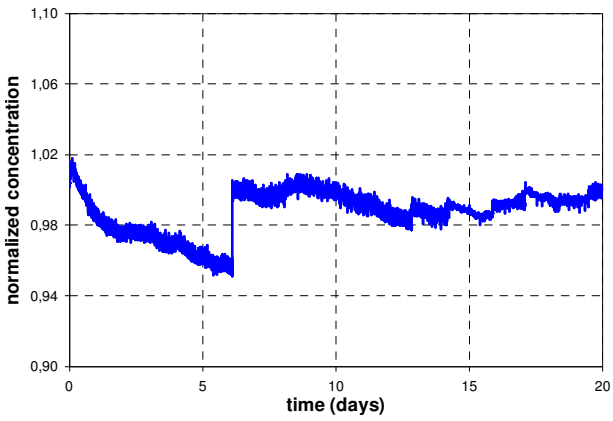


Fig. 2. In-line concentration monitoring of a) Tungsten b) Ceria CMP-Slurry (values normalized to one, real mass concentration may vary between 0,1 and 20 %)

3.2. Size monitoring in particle disintegration units

In CMP-slurries also modifications in the particle size may occur. Worst cases are big agglomerates, which provoke scratches on the waver. But especially in particles systems preparing from dry powder or with high-density contrast agglomeration is probable. For particle disintegration it will be very helpful obtaining measured size information.

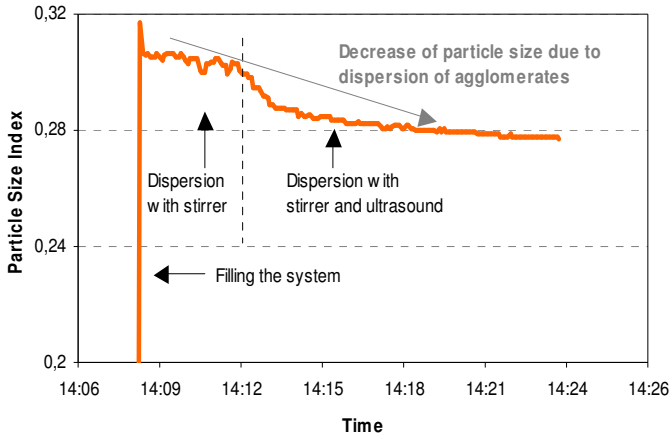


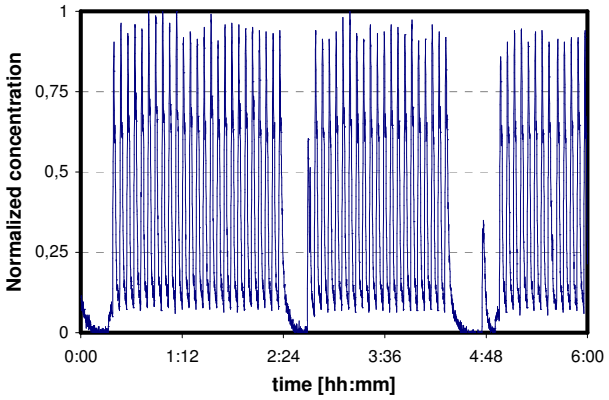
Fig. 3. Monitoring of size variation (index) by agglomerate disintegration in a Ceria slurry

The 1560 device was applied in Ceria (setup as fig. 1).

Fig. 3 shows the influence of the different kind of slurry preparation of the mean particle size (index) in the slurry loop. Only stirring and pumping was for this slurry not enough to destroy agglomerates. High power ultrasound shows a strong effect.

3.3. Sewage treatment

Governmental environmental protection agencies ask for more and better waste treatment [9]. The demand for the reduction of CoO (cost of ownership) in all departments also applies to waste reclaim. Even right now, the waste treatment and the prices for the waste disposal for some regions offer large business opportunities. Integrated control and segregation systems for the waste streams will gain in importance. The concentration measurement device 1551 can also be applied in wastewater treatment. Figure 4a) gives an impression of the silica particle content after the polishing step. The concentration is very low when compared to the original ~10 to 30 % applied to the waver, in the waste ~ 0,01 - 1 %. The polishing cycle of a single wafer polishing is reproduced with great exactness. The time for a change the wafer carrier is shown by extended intervals of lower particle content in the water originating from the tool. The distinction between pure water and the targeted treatment of higher solid content is possible. As experience shows, this effect becomes increasingly important since UF filter modules behave non-predictably due to blockage by polishing particles.



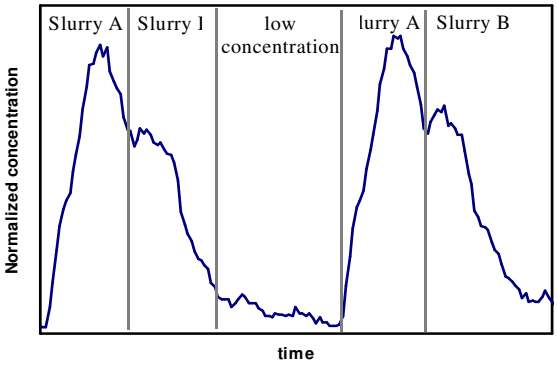


Fig. 4. a) Solid content of wastewater after a polisher and b) distinction between two kinds of solids (values normalized to one, real mass concentration may vary between 0,01 and 10 %)

Chemical reactions occurring in the waste pipe pose a great safety risk of pipe blocking and unplanned tool shutdowns. Reactions can occur if several CMP slurries are used in succession. Different iso-electrical behavior of the particles can be sufficient to induce sediment formation. Spectral backscattering permits to detect also the type of particles. This can be used to separate waste streams and prevent uncontrolled machine failures. Tests of a modified device (5000) after the polishing process with integrated wastewater switch in tungsten applications were completed successfully (see fig. 4b). Only the integrated sieve has to be emptied from broken waver fragments after some months of employment.

4. SUMMERY

The slurry concentration and particle size can be an overlooked effect influencing the polishing success of the CMP-process. Concentration and size variations stemming from the slurry mixing or supplying system are usually not monitored and therefore not controlled. The in-line concentration measurement of slurries with high particle loads needs special measurement instruments. The used Aello 1551/1560/5000 devices are suitable for measuring over a broad concentration and size range. They determine a concentration and size index as well as the particle type by using optical measurement methods. Highly detailed results of measured values were presented for Silica, Alumina and Ceria. Variation in the slurry mixing and delivery plants was detected in the different applications. The tested devices are strong in in-line use. These devices are helpful complement to high-resolution slurry characterization methods suitable in laboratories. Installation in CMP mixing plants of semiconductor manufacturers in the Europe, US and Taiwan has demonstrated long-term stability and low maintenance demands (service-free for months). Tool owners and media personnel report increased product reliability (less rejected slurry) and higher process yields.

REFERENCES

[1] G. Grover: *"Challenges and advances for CMP consumables"*
Solid State Technology, April (2004)

[2] T. Kuntzsch, *Erfassung und Beeinflussung des Zustandes von Poliersuspensionen für das chemisch-mechanische Polieren (CMP) in der Halbleiterbauelementfertigung,* TU Dresden, thesis, January (2004)

[3] B. Johl, R. K Singh, *Optimum process performance through better CMP slurry management*, Solid State Technology, Nov (2004)

[4] St. Kealler, *Performance and reliability improvements or CMP slurry dispense systems,* proceedings 6th CMP-user meeting, Munich (2001)

[5] P.S. McMillan, *Using liquid flow controllers to enhance CMP tool performance,* Micro, January (2001)

[6] F. Hinze, J. Altmann, *In-line particle sensors for process control,* proceedings 6th CMP-user meeting (2001)

[7] www.AELLO.biz

[8] U. Feller, J. Altmann, *Monitoring of CMP-Slurries and CMP-Wastewater,* proceedings 12th CMP-user meeting, Munich (2004)

[9] B. Jenkins et. Al, *The many options for managing CMP wastewater*, Solid State Technology, July (2004)