Safety in CMP supply systems

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1 Abstract

The CMP process is commonly associated with a high consumption of water and slurries. The slurries originate from mixing, blending and distribution plants. For these processes, 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 the optical backscattering device 1551 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 influence of high density contrasts between particles and fluids, and therefore also of the direct dosage and blending control was carried out. The measurement technique used can also be applied for wastewater treatment and segregation. The separation of components by concentration and particle type allows for a special treatment of waste components, UP water reclaim and particle reuse.

Key words: *CMP, slurry, concentration, measurement, control, mixing blending, distribution plants, yield, wastewater, treatment, segregation*

2 Introduction

The CMP process step in the semiconductor manufacturing has gained greater importance during recent technology enhancement. For the 65 nm technology it will be a key semiconductor process [1]. Quite a lot of research work has been conducted to determine the driving parameters for a successful application of the CMP process. 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. Since the number of influence parameters originating from the process is large, the slurry supply chain is often treated as a black box and/or slurry quality as given constant.

In practice, a number of influences shapes the properties of the CMP slurry during the production, delivery, storage, mixing and supplying steps. Validation of the supplier and their production process are a common means of dealing with these influences. As a consequence, this has lead to reduced quality variations in slurry production.

To reduce transportation cost the slurry is delivered at high concentrations. The lower concentration demanded by the polishing tool is then obtained by dilution. In some cases the slurry will be manufactured from dry polishing particles, only.

A distinction can be made between large systems for central slurry storage, blending and distribution, and on-site slurry handling units in close proximity to the polishing tool. Central supply systems are favorable for high slurry consumption and easy-to-handle slurry systems. On-site stations are better suited for special chemicals for which consumption is low and complex preparation modes have to be ensured.

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].

It seems quite obvious that on the way from producer to the waver the blending of slurry is a simple method to maintain slurry quality. The slurry blend ratio will be defined by volumetric or mass dosage. This dosage commonly does not take the real solid concentration into account. This may not be important for slurries with simple handling like colloidal silica. However, 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. This can be another source for concentration variation. Finally, hardware aging, e.g. of valves and pumps, can modify flow rates and dosage volumes. As a consequence, fluctuations around the demanded concentrations are very likely.

Despite being aware of all these fallacies the user commonly is forced to rely on slurry manufacturer specification, since there is usually no quality control. On the other hand, process control is a requirement if slurry properties must be guaranteed.

3 Concentration control in slurry mixing plants

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. Indirect in-line measurement methods like the determination of conductivity fail if the particle concentration varies. The sensitivity of basic measurement parameters such as pH, viscosity and oxidation-reduction potential is not large enough to determine slurry properties. On-line 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]. Consequently, a demand exists for reliable and cost saving in-line concentration measurement techniques.

A measurement device specifically designed for in process measurement was tested in an experimentation and industrial setup. The Aello 1551 is able to determine the concentration of nano scaled particles as well as the type (material) of solid. The optical backscattering measurement principle employed by the device results in the advantage of low operating costs and a reasonable purchase price. The measurement data can be exported via standard interface to any external programs. A free programmable output permits a direct process control by the sensor. By means of a serial interface the data can be transferred to monitor software. The measurement method is insensitive to gas bubbles and exhibits long-term stability [7].

First the device was tested in a laboratory setting through measurement of different slurries at various concentrations. The measurement method produced highly resolved measurement results over a broad concentration range from less than 0,5 % up to over 40 %. For specific applications, the exact range and resolution depends on the optical properties of the slurry. In this case, the system will be adjusted to the particular slurry properties.

Alumina slurry was tested first. 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 %.

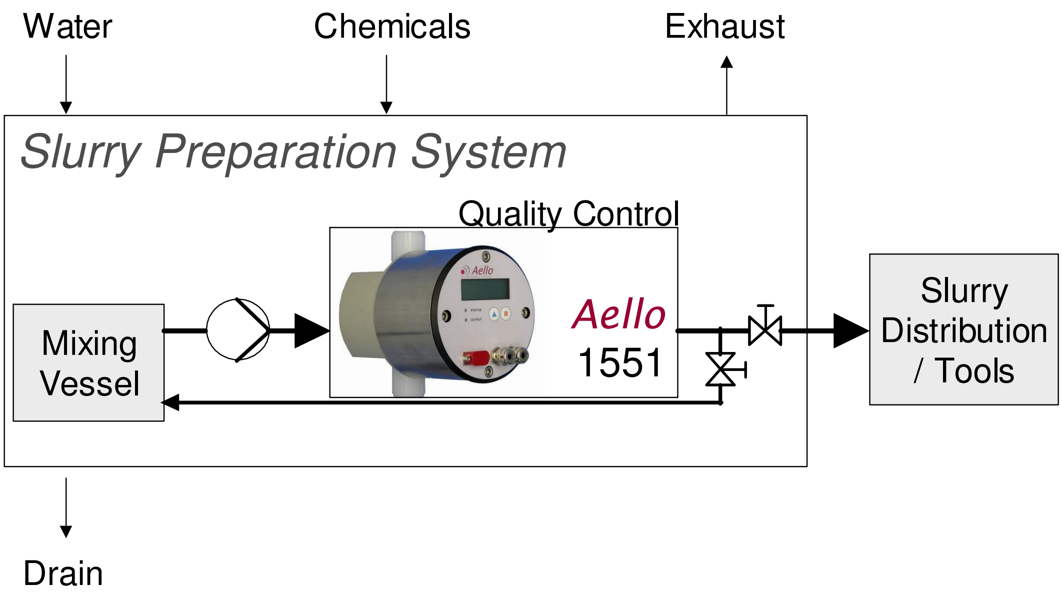

Fig. 1: Integration of concentration Aello 1551

After these very positive lab tests, the device was installed in a central slurry mixing and distribution plant as shown in figure 1. The measurement results over a time of nearly nine days are depicted in figure 2.

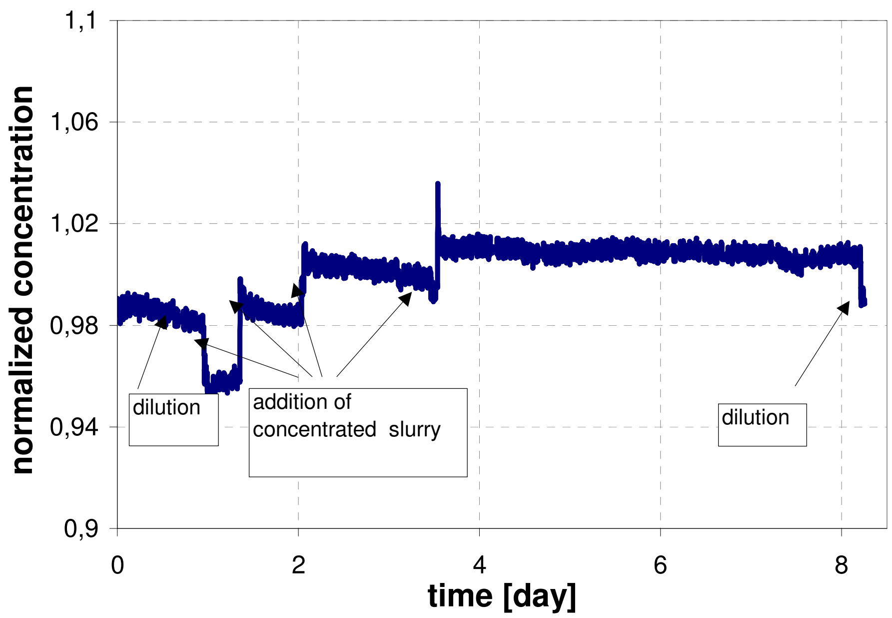

Fig. 2: Monitoring of the concentration of metal-CMP-Slurry
(values normalized to one, real mass concentration may vary between 0,1 and 30 %)

Again, 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 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. It is noteworthy that over time there appears to be a very slight trend to lower concentrations.

Long term experience of one year with the slurry mixing and distribution systems shows very low maintenances requirement. Even after months there was no need for cleaning and recalibration of the sensor probe. The device manufacturer recommends control checks of the measurement devices at intervals of 2-3 month.

For copper processing, slurries with ceria introduce a new particle material to the polishing process. The high-density contrast between particles and liquid (water 1, ceria 3) makes slurry handling more complex. The blending and distribution plants must deal with a larger tendency of settling and de-mixing of the particles. Experience in the laboratory shows that concentration variation may occur in non-optimal flow circumstances. Consequently, the measurement flow through the cell itself was redesigned to guarantee homogeneous and reproducible measurement conditions.

In a second step the measurement technique was applied. An example for such an application shows in figure 3.

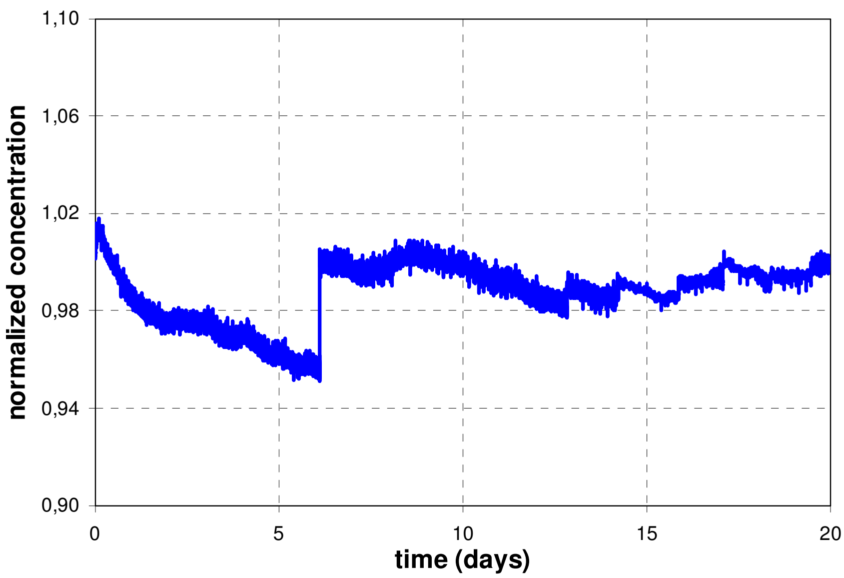

Fig. 3: Measurement of ceria-CMP-slurry
(values normalized to one, real mass concentration may vary between 0,1 and 30 %)

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 2 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).

Additionally, long-term experiences with ceria slurries show very low maintenance requirements of the measurement device (such as those already mentioned for the tungsten application).The integration with guaranteed hydrodynamic homogeneous flow condition is of great importance.

4 Wastewater treatment

Recently, questions of environment protection and water reclaim have become gained greater importance. Governmental environmental protection agencies ask for more and better waste treatment [8]. Removal of harmful contaminants from the waste streams becomes a requirement. This implies that abrasive particles and hazardous chemicals need to be removed and the pH must be brought to neutral levels. Specialized facilities for the treatment are demanded. Consequently, new ways for solid handling and wastewater segregation for fab-drain systems will have to be considered.

Capital and operating costs are the driving forces for water reclaim. 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. Therefore, the reuse (reclaim) of decontaminated water (low or no solid content) and the external use of the solid (e.g. reclaim of silica for the concrete industry) can be of economical interest. The selective and precise treatment of the different waste components is of great importance. The rinse water can be easily reused. However, since the slurry in the waver polishing step will be diluted with UP water by a factor of 60, the polishing step often delivers UP water with no or only very littler contamination.

Therefore, integrated slurry control and segregation system for the waste streams will gain in importance.

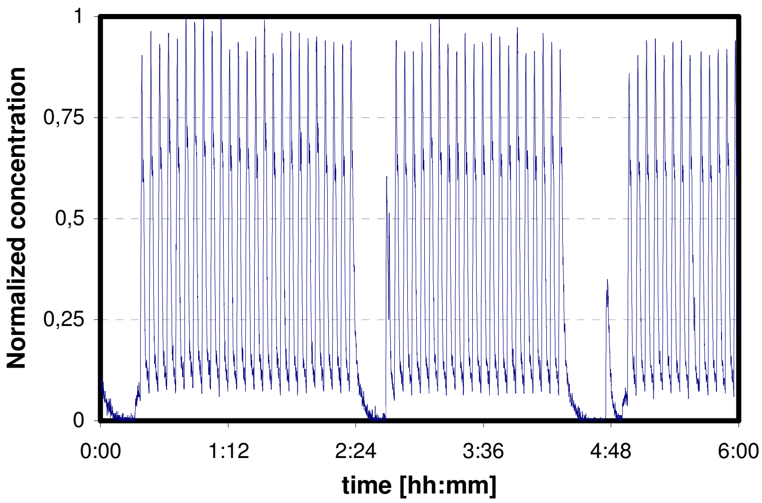

Fig. 4: Use for wastewater at a polisher of the Chemical Mechanical Polishing
(values normalized to one, real mass concentration may vary between 0,1 and 30 %)

The concentration measurement device used for the slurry supply can also be applied in wastewater treatment. Figure 4 gives an impression of the silica particle content after the polishing step. As mention above, the concentration is very low when compared to the original ~10 to 30 % applied to the waver (in figure 4 the concentration is normalized to 1).

The polishing cycle of a single wafer polishing is reproduced with great exactness. Additionally, the time for a change the wafer carrier is shown in the diagram 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 unpredictably due to blockage by polishing particles.

Chemical reactions occurring in the waste pipe pose a great safety risk. Reactions can occur if different CMP slurries are used in succession. Different iso-electrical behavior of the particles can be sufficient to induce sediment formation. Chemical reaction thus can lead to pipe blocking and unplanned tool shutdowns.

Picture 5 illustrates what may happen in case of chemical particle interaction. The pipe shown was destroyed during a high pressure-cleaning attempt.

Fig. 5: Blocked waste water pipe after high pressure cleaning behind the polisher

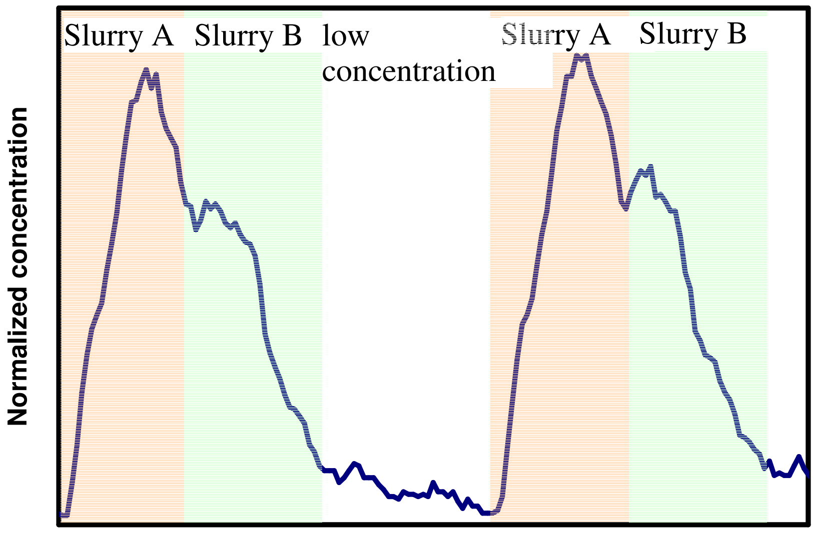

Fig. 6: wastewater usage at a polisher of the Chemical Mechanical Polishing (values normalized to one, real mass concentration may vary between 0,1 and 30 %)

Not only the detection of particle concentration but also the particle type as well as the separation of different types of slurries can increase the availability of the waste treatment infrastructure and prevent uncontrolled machine failures. The Aello 5000 can serve as a possible tool for such a separation of different wastewater contents after the polishing process. The common features are shown in figure 7.

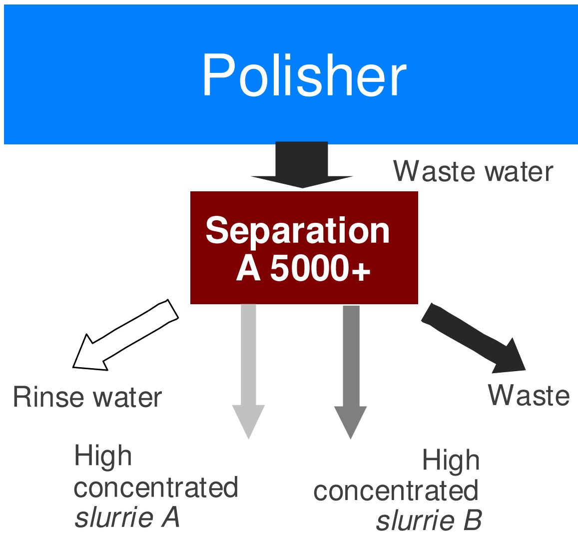

Fig. 7: Tool for CMP content separation for the wastewater of a polisher

Tests of the device in tungsten applications completed successfully. Maintenance requirements are posed by the integrated sieve. This part has to be emptied from broken waver fragments after some months of employment.

5 Summary

The slurry concentration can be an overlooked effect influencing the polishing success of the CMP-process. Concentration 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 device is suitable for measuring over a broad concentration range. It determines concentration and the slurry particle type by an optical measurement method.

Highly detailed concentration results were presented for alumina as well as ceria. Also very good experiences were obtained with slurries containing other particles e.g. silica. The variation in the slurry mixing and delivery plants was detected in the different applications.

The evaluation of the measuring systems for metal CMP slurry and CMP wastewater characterization shows high-resolution concentration values for different customer applications. Installation in CMP mixing plants of semiconductor manufacturers in the US and Europe 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"* University of Technology 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, Munich (2001)
[7] U. Feller, J. Altmann: "*Monitoring of CMP-Slurries and CMP-Wastewater"*
proceedings 12th CMP-user meeting, Munich (2004)
[8] B. Jenkins et. al: "*The many options for managing CMP wastewater*"
Solid State Technology, July (2004)

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