CMP Slurry Blending Process Optimization and Cost Improvements using Real-time Concentration Monitoring

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Abstract - The approach of this study is to install a real-time concentration monitoring system in a slurry blending system with the main intention of replacing a complex auto-titration system for cost reduction as well as a process optimization tool to further improve and streamline the chemical mechanical planarization (CMP) slurry blending process. A Swagelok CR-288 Concentration Analyzer was installed in a Kinetics Slurry Blending System to monitor in real-time the peroxide concentration of the slurry batch from the initial stage of slurry mixing to the final step of supplying the qualified slurry to the CMP polishers. The integration of the real-time concentration analyzer in the slurry blending system contributed to a 60% cost reduction in the equipment cost of ownership (COO) as well as the optimization of the blending process parameters.

Keywords: Chemical Mechanical Planarization (CMP), Cost of Ownership (COO), Index of refraction (IoR)

I. INTRODUCTION

A. Background

Effective CMP slurry management is essential to meet interconnect challenges; improper slurry handling can adversely affect slurry health and device yield and drive up process COO [1]. The slurry supply chain is often treated as a black box and slurry quality as a given constant. In practice, a number of influences shape the properties of the CMP slurry during production, delivery, storage, mixing and supplying steps [2].

The multi-part slurries used by the semiconductor industry often require mixing or dilution before use. Oxide polishing slurries are commonly purchased in concentrated form and diluted with water on-site to minimize shipping and labor costs, while some multipart polishing slurries must be combined before use because of their short post mix lifetime [3].

The challenge lies in effectively monitoring the slurry health indicators such as hydrogen peroxide (H_2O_2) assay in the mixed slurry. The current method to monitor the peroxide assay in mixed slurry is through volumetric titration with the use of either an off- line or an in-line titration unit. This method is reliable but it incurs high equipment maintenance and chemical reagents cost as well as it requires a lapse time after completing the mixing sequence [4,5].

There are already numerous studies on the benefits of real-time chemical concentration analyzers/monitors for Wafer Fab chemical applications but the focus has been more on the innovation itself rather than on the tangible and intangible benefits derived from the technology especially its impact on CMP slurry handling, mixing and distribution.

B. Automatic Titration Units/Autotitrator

Hydrogen peroxide (H_2O_2) content in CMP slurries is commonly analyzed through a grab sample titration analysis. A specific amount of slurry sample is collected from a sampling loop right after the slurry blending and mixing sequence is completed. This sample is then either analyzed using an in-line titration unit - that is the equipment is directly connected to the slurry system, or by an off-line unit. Analysis of the peroxide content of the CMP slurry is commonly done through redox titration. The common titrants used in H_2O_2 concentration analysis are potassium permanganate ($KMnO_4$) and cerium sulfate ($Ce_2(SO_4)_3$). In this study, the H_2O_2 content of the slurry was determined by redox titration using cerium sulfate as the titration reagent.

$$H_2O_2 + 2Ce4^{+} \rightarrow 2H + O_2 + 2Ce^{3+} \qquad (1)$$

The grab sample titration analysis works very well however it is time consuming whereby the H_2O_2 concentration can be determined after a few minutes to an hour. Fig. 1 shows the typical analysis steps an auto-titrator performs during routine sampling.

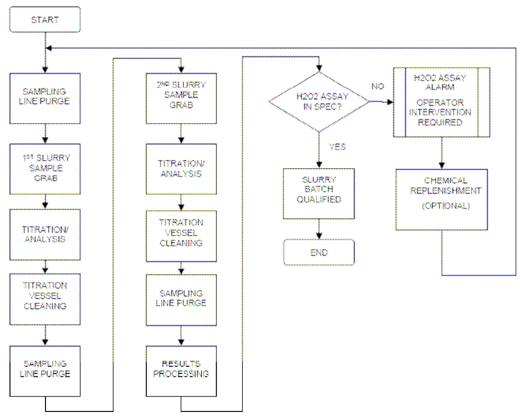

Fig. 1. An autotitrator analysis steps showing 2 sample runs and an optional chemical replenishment step.

C. Real-time Concentration Analyzer- Swagelok CR-288

CR-288's principle of operation is based on Snell's law, which gives the relationship between angles of incidence and refraction for a wave impinging on an interface between two media with different indices of refraction.

The CR288 uses an optical system to accurately measure liquid chemical concentration in real time. The principle of operation is straightforward and is based on measuring the index of refraction of the liquid in contact with the sapphire window using optical reflectivity geometry. It has a miniaturized, fully integrated optical reflectivity subsystem. Light from an LED is shone unto the sapphire window that is contact with the liquid under analysis. The light then scatters into the 128-pixel photodiode array as shown in Fig. 2.

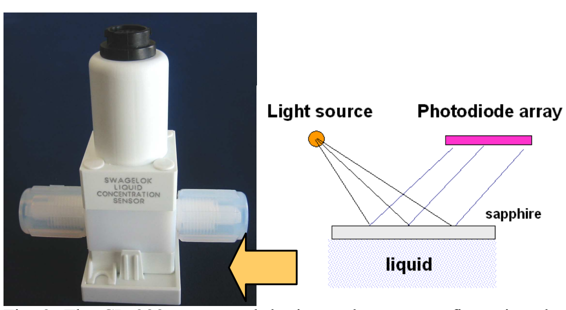

Fig. 2. The CR-288 sensor and the internal sensor configuration showing the layout of the sensor's light source, sapphire window and photodiode array in relation to the liquid.

In this way, the CR-288 measures the critical angle to high accuracy and therefore the index of refraction of the liquid. The CR-288 algorithm determines the chemical concentration from the index of refraction. The index of refraction is temperature-compensated using patented dual temperature compensation method. Furthermore, the sensor head optical geometry uses a reflection geometry coupled with optimized fluid dynamics. In this way, CR-288 can measure liquids such as slurries and optically transparent materials that cannot be measured in a transmission optical geometry. Figure 3 shows in detail how the CR-288 sensor measures a liquid's chemical concentration based on index of refraction.

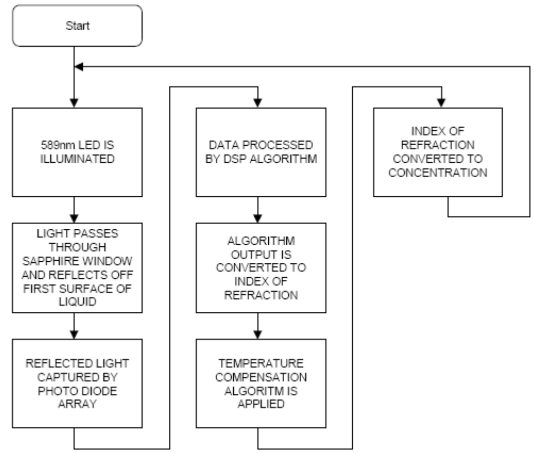

Fig. 3. A CR-288 concentration analysis flow showing a 1.2 second sample run.

The concentration monitor incorporates actual refractive measurement from the process stream to obtain a true concentration value. As a chemical solution's temperature increases, refractive index tends to decrease in a non-linear relationship. The monitor tracks this relationship to obtain the IoR@20 deg C, which is the temperature compensated concentration analysis. Common chemical solutions have a linear relationship similar to the one shown in Fig. 4, with Index of Refraction (IoR) of H_2O_2 increasing when the H_2O_2 concentration increases. The refractive measurements are standardized at 20˚C to allow baseline characterization of the relationship between changing temperatures and the IoR [6, 7].

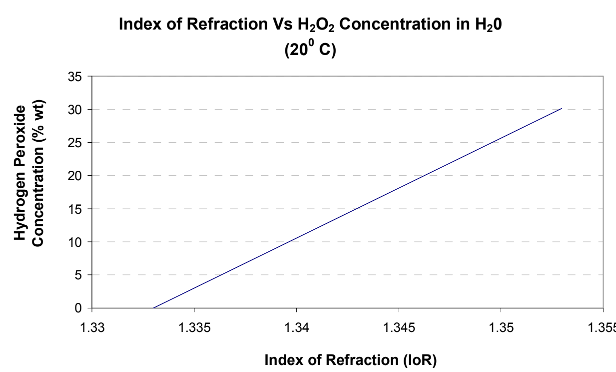

Fig. 4. Hydrogen Peroxide (H_2O_2) concentration at 20°C as a function of Index of Refraction.

For the application of CR-288 as a real-time concentration monitoring, it is essential that the analyzer is able to differentiate the IoR of the raw slurry from the IoR of the mixed slurry spiked with H_2O_2. From this difference in IoR, the H_2O_2 concentration of the slurry mixture is determined.

II. EXPERIMENTAL SECTION

A. Integration of CR-288 Concentration Analyzer into the Slurry Blend and Distribution Sequence

The Kinetics Blending and Distribution System is designed to blend and distribute colloidal and fumed slurry to medium and large CMP polisher lines. The system is composed of two blend stations working in conjunction to provide a continuous supply of slurry to multiple polishers. Fig. 5 shows the typical set-up of a slurry blending and distribution system showing the slurry supply flow from the slurry blend/distribution system to the loop filter box and then to the global loop which comprises of valve manifold boxes connected to the CMP polishers. The autotitrator is connected as an in-line metrology unit, which analyzes the hydrogen peroxide content of the slurry after the completion of the blend sequence.

The system blends slurry, DI water and up to two chemicals according to an adjustable recipe. The raw slurry is pumped from an online supply drum into the blend tank.

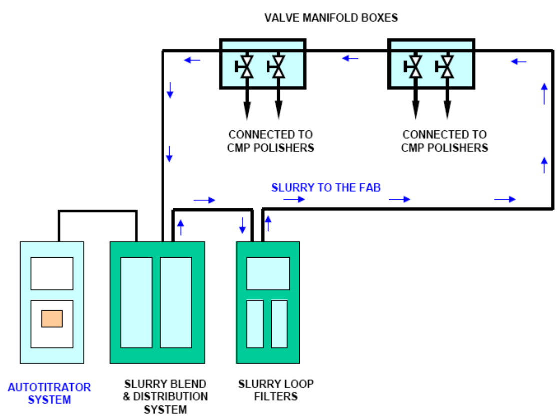

Fig. 5. Typical set-up of the CMP slurry supply loop showing the slurry blend/distribution system, loop filters, the valve manifold boxes and the autotitrator unit.

The slurry and other components are metered into the blend tank by weight and recirculated in the blend loop until the mixture is homogenous. The blend must pass quality analysis before the slurry can be distributed to the global loop as shown in Step 4: Titration Analysis of Fig. 6.

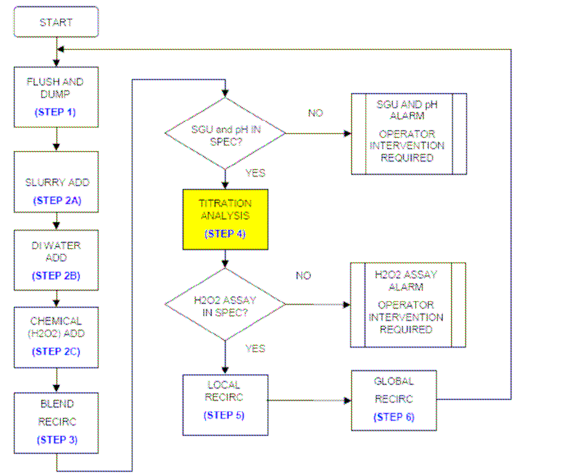

Fig. 6. The slurry blending sequence showing the different blending and qualification steps prior to supplying the slurry to the polishers.

When the recirculation and testing are complete, the slurry is available to the global loop. The moment the online blend tank weight reached the low level set point; the second blend unit begins distribution to the global loop. The low-level blend tank undergoes a cleaning cycle (flush and dump) wherein the remaining slurry is dumped to the drain and the blend tank is washed with DI water. After the cleaning cycle, a new batch will be initiated and will be on standby until the online tank reaches the low level set point [8,9].

The challenge of this study lies on how to effectively integrate the CR-288 Concentration analyzer into the existing Kinetics system by replacing the autotitrator and all its metrology functions. The integration involves the modification of the slurry blending system to align with the analyzer's output data requirements as well as retrofitting the hardware to accommodate the CR-288 probe inside the blending system. On the other hand, the application of CR-288 to determine the H_2O_2 concentration of the slurry posed a big challenge itself since the slurries tested have its own specific characteristics, which produces various interferences in the Index of Refraction results.

For the experimental runs, one slurry system blending Cu CMP Slurry E was retrofitted with a CR-288 Concentration analyzer while running in parallel with the existing autotitrator unit. Fig. 7 shows how the CR-288 Concentration analyzer was hooked up to the existing slurry blend system. The CR-288 results are archived separately using the analyzer's software database while the autotitrator results are logged into the slurry system database during the duration of the experimental run.

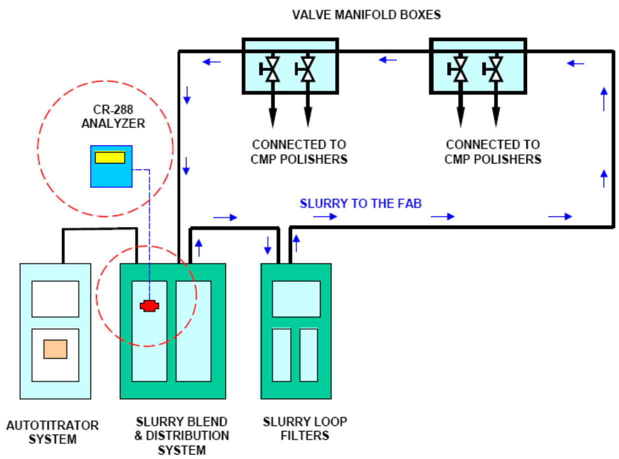

Fig. 7. Slurry system and supply loop showing the integration of the CR-288 Analyzer during the experimental runs.

This set-up will enable the CR-288 probe to capture the same slurry sample stream that is going to the autotitrator' sampling loop during each scheduled analysis. The experimental runs will be conducted based on the sampling schedule as described in Table 1.

TABLE 1
CR-288 CONCENTRATION ANALYZER AND AUTOTITRATOR SAMPLING PLAN [a]

Analyzers used	Autotitrator	CR-288
Blend tank sampling frequency	Every 3 hours	Real-time monitoring
Day tank sampling frequency [b]	Every 3 hours	Real-time monitoring

[a] Experimental run duration is for 3 months with the system running 24 hours operation.
[b] The blend tank will act as a day tank once the station switches to global supply – system is supplying slurry to the CMP process.

III. RESULTS AND DISCUSSION

The titration results for the H_2O_2 concentration of Cu CMP Slurry E as analyzed by the slurry system's autotitrator and the CR-288 Concentration Analyzer were compared using the Analysis of Variance (ANOVA) using the Tukey-Kramer test for means comparison with the use of SAS-JMP software. The ANOVA test results showed that the CR-288 concentration analyzer is comparable to the autotitrator's performance. Sample to sample variation from the CR-288 analyzer were also better as compared to the autotitrator sample results as seen by the quantile plots of the two titration results as seen in Fig. 8.

With the availability of real-time chemical concentration data, it was seen that majority of the existing blend system set points were not optimized and unnecessary titration samplings were being performed. Through real-time monitoring, blend recirc timings – that is the time required for the slurry to recirculate in the loop before it is ready for sampling – was reduced.

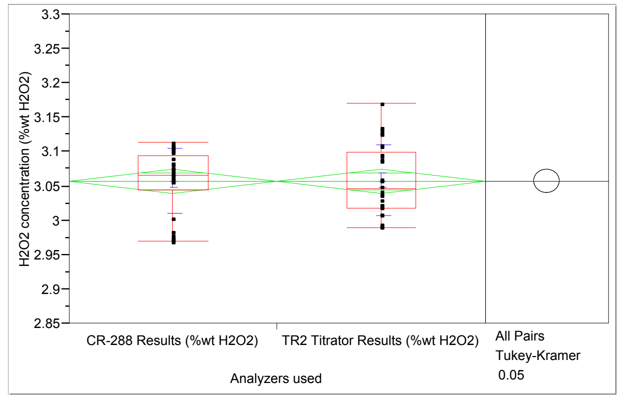

Fig. 8. Analysis of Variance (ANOVA) for the CR-288 Analyzer and TR2 Autotitrator Results for the H_2O_2 concentration of Cu CMP slurry E showed that the CR-288 results means are comparable to the TR2 Autotitrator results.

The old set-up with the in-line autotitrator takes up to 1 hour before a slurry mixture is accepted as a qualified slurry blend. Optimizing the blend recirc timings will eliminate unnecessary lapse time between slurry blending steps and at the same time shortening the qualification step in determining the CMP slurry's H_2O_2 concentration without sacrificing slurry quality. Figures 9 and 10 shows the optimized slurry blending steps reduced from 6 to 5 steps only.

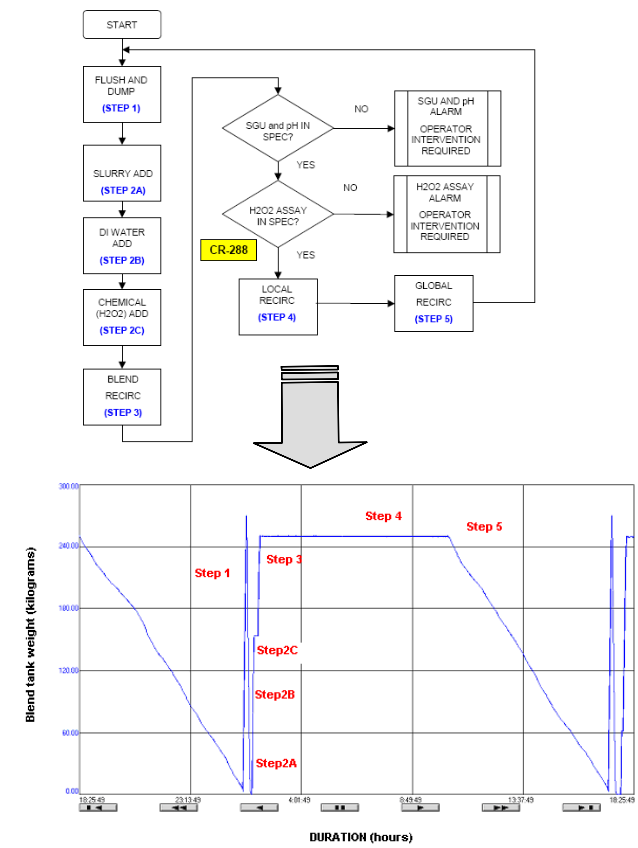

Fig. 9. The blend tank weight trending showing the revised slurry blending and distribution flow with the titration analysis step eliminated upon the CR-288 integration.

The CR-288's digital display unit (DDU) was connected to the slurry systems network making the concentration trend and data available in the existing Facilities Control Room. An added feature is the capability to counter check the sensor's performance during each DI water rinse cycle (between Steps 1 and Step 2A) since DI water always give a unique reference value for index of refraction.

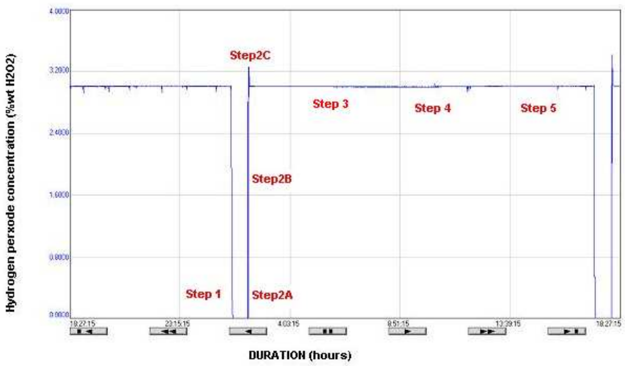

Fig. 10. A real-time H_2O_2 concentration trend measured by the CR-288 analyzer. The dip in the concentration trend after Step 1 – Flush and dump is due to the cleaning cycle – it is an added feature to countercheck for any sensor drift.

The integration of the CR-288 concentration analyzer into the slurry blend system eliminates the use of the autotitrator unit resulting to the subsequent 60% reduction in the slurry system's COO as detailed in Table 2.

TABLE 2
BENEFITS GAINED FROM THE CR-288 ANALYZER INTEGRATION

Equipment capability and requirements	**Autotitrator**	**CR-288 Concentration Analyzer**
Slurry blend qualification duration	Typically 1 hour	< 1 minute
Concentration monitoring	Analysis done at set time intervals only	Real-time trending
Preventive maintenance cost	Fortnightly PM activity	No PM required, probe is cleansed every DI water rinse cycle
Spares and consumables	Chemical reagents required for analysis, Regular replacements of analytical probes, motors and valves	None required
Calibration	Quarterly calibration Requires calibration standards	Auto-check every DI water rinse cycle, probe performance is monitored thru trending
Space requirements	1.5 square meters (m^2)	0.05 square meters (m^2) - Mounted on top of the blend system

IV. CONCLUSION AND RECOMMENDATIONS

Engineering improvements covering mainly on the innovative integration of the analyzer as well as process optimization performed on the system contributed to a 60% reduction in the equipment cost of ownership (COO). The slurry blending process was simplified by replacing a complex and cumbersome titration analysis method with a simple, cost-effective and more efficient real-time metrology. Real-time concentration monitoring enhanced the quality control aspect of the slurry blending process as well as introducing as a pro-active approach to CMP slurry health monitoring.

Topics for future work would be the monitoring of CMP slurries used in the manufacture of devices at or below the 90-nm technologies considering the much tighter requirement for oxidizer concentration, which is currently on the borderline of the concentration sensor detection limits. Another challenge is the integration of the CR-288 analyzer into other slurry blend system to test out its ease of integration.

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Srinath Rajan is a lead engineer at Kinetics, Singapore. His work as a Senior Controls Engineer at Kinetics has mainly focused in providing solutions to the control system for Slurry Dispense Systems, Chemical Dispense Systems and Monitoring Systems. He has carried out projects in the applications including Bio-pharmaceutical and Semiconductor industries. He has been working at Kinetics, Singapore, providing Solutions to Slurry Dispense Control System and Chemical Dispense Control Systems for the semiconductor industry segment for over 5 years. He received his Bachelor of Engineering degree in Electronics and Communications from University of Mysore, India.