SLURRY DELIVERY SYSTEMS DEFINING INCOMING CMP SLURRY DENSITY AND ACHIEVING TARGET PROCESS CONCENTRATION DRIVEN BY INLINE METROLOGY UNIFY CMP ENGINEERS

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INTRODUCTION

The chemical mechanical planarization CMP community has found a need for defining new industry standards for stakeholders with respect to reporting technical properties and functionality of CMP related materials and consumables in typical process conditions. CMP slurry density is one of the critical objectives. In serving complex CMP slurry feed to point-of-use, the presented slurry delivery system has validated inline refractive index unit RIU liquid measurements in combination with other proprietary measurement techniques in defining and achieving target set-points of both slurry density and H_2O_2 concentration.

Simultaneously semiconductor fabs set increasingly tight upper and lower control limits UCL and LCL on slurry density and H_2O_2 additions. The technology of defining incoming slurry density and achieving target ultrapure water UPW dilutions is enabled by a closed-loop real-time control and liquid monitoring in the slurry delivery system SDS. Data from the process characterization of the industry relevant slurry shows the system meets a slurry-UPW density dilution target in a +/-0.000025 g/cm3 control limit range. Addition of H_2O_2 to a multi-constituent solution presented a consistent blending to within +/-0.004%wt of target.

Process characterization and metrology selection is an essential step in the achievement of real-time process control and monitoring in CMP, post CMP clean, and other blending and distribution applications. To eliminate the need for offline instruments, a characterized defined process of inline metrology devices is employed to auto control the blend in achieving required tight accuracy and precision.

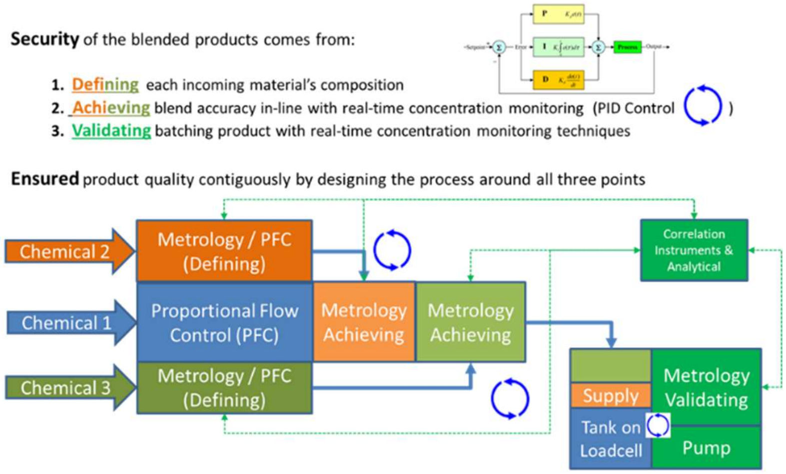
BACKGROUND

Driving industry standards among CMP experts has been undertaken by programs hosted by SEMI being a central industry collaboration platform. SEMI C96 was one of the standards presenting a test method for determining density of CMP slurries in offline conditions. The motivation was to set a common practice in preparing a slurry sample under test, accommodating for measurement conditions and report the results with given significant measurement figures. The standard was devoted specifically to CMP slurry users, integrated device manufacturers IDMs, slurry suppliers, metrology manufacturers and original equipment manufacturers OEMs [1]. The entailing challenge was to design a slurry delivery system used in R&D and high-volume manufacturing HVM eliminating the need for offline or sampling measurement methods and instead use a concert of inline instrumentation to achieve real-time results that have been proven to meet or exceed the levels seen by many offline methods.

TOWARDS CLOSED-LOOP REAL-TIME CONTROL

In previous studies it has been established that RIU measurements correlate with slurry density g/cm3 in colloidal silica slurries with a smaller limit of detection LOD compared to conventional inline u-tube densitometers [2]. RIU describes physical properties of fluids and is detected with an optical instrument yielding an output for both incoming slurry density and concentration of subsequent H_2O_2 additions. The optical concentration measurement is based on Snell’s law and critical angle of total reflection. Light is transmitted from a light emitting diode to the interface between the optical window and the liquid. With the concentration of the liquid, defined angles are reflected creating light and shadow interface images on the digital camera. The interface of the light activated pixels is converted to RIU and concentration values [3].

Inline RIU sensor metrology has been successfully employed in scale-up processes during characterization of tungsten, copper, and interlayer dielectric ILD oxide slurries in offline test benches to R&D and HVM slurry delivery systems. RIU is proven to give an instant signal in the event a slurry tote has been left unmixed with stratified fluid for longer periods than recommended for the specific slurry type. Slight composition shifts from slurry manufacturer's lot-to-lot, potentially inferred by incomplete onsite mixing can be identified in the incoming raw slurry stream and corrected for in the SDS presented in this study. In addition, target H2O2 concentration levels are specified and validated by RIU prior to releasing the blend to the supply loop.



Security of the blended products comes from:

1. **Defining** each incoming material's composition
2. **Achieving** blend accuracy in-line with real-time concentration monitoring (PID Control)
3. **Validating** batching product with real-time concentration monitoring techniques

Ensured product quality contiguously by designing the process around all three points

Fig.1 Patented metrology driven closed-loop control

Fig.2 Integration of KxS Technologies inline refractive index monitor

DFS blend and distribution technology is designed for characterizing CMP slurries by employing a defined system process. The system is naturally purposed for defining incoming chemical by using metrology measures for detecting variation in the chemical feed and adjusting for the shifts in lot-to-lot feeds to maintain the blend consistency and achieve the target chemistry concentration within the tightest requirements. Inline metrology approaches in the incoming supply, blend and distribution processes provide the ability to define, achieve and validate precise chemical volume in the supply loops in real time. Automatic closed-loop adjustment for any degradation, shift or other changes in a component within the chemical mixtures ensures that the chemistry remain constant to the tightest required values.

In the characterization studies the goal was to find and detail the limits of all analytical and process instrumentation. Profiling was conducted in distinct phases to characterize all relevant portions of the mixing process including:

- Defining the incoming solutions and diluting them to various concentrations
- Achieving the same target CMP slurry density and H2O2 concentration repeatably
- And lastly, letting the system provide to the points-of-use in an automatic mode of operation unhindered

EXPERIMENTAL AND RESULTS

The experimental study included profiling the density of different CMP slurry production lots as delivered by the slurry manufacturer by using both analytical offline methods and inline metrology within the system's process pathways. The test sequence was conducted in three (3) phases. In phase one (1) the incoming slurry density was defined and diluted at five (5) dilution setpoints.

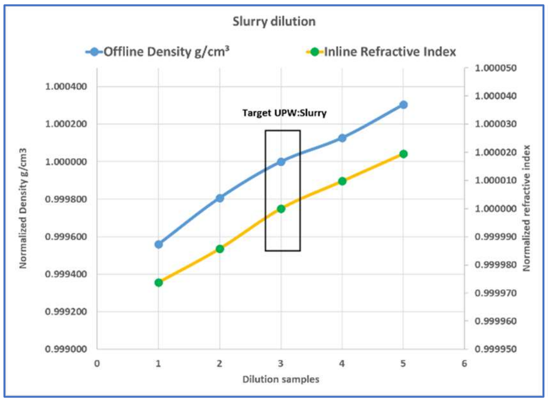


Fig.3 Defining and profiling UPW dilution ratios of an industry relevant slurry

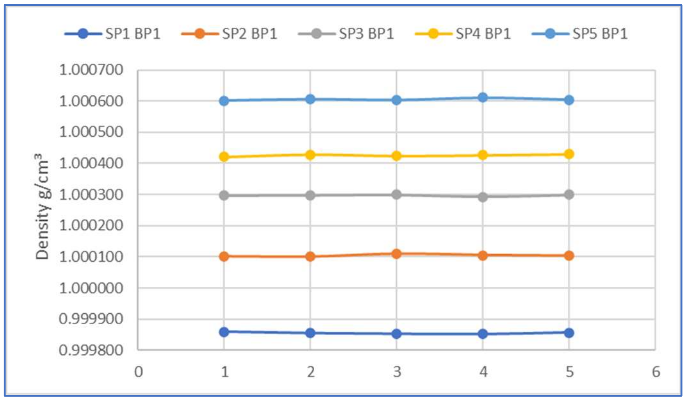


Fig.4 Repeatability study of each slurry density setpoint

In Fig.3 the five (5) slurry dilution samples were measured with an offline densitometer (upper blue line) following the set requirements in SEMI standard C96. Simultaneously within the system the inline RIU monitor detected the corresponding slurry density dilutions (lower yellow line). In Fig.4 slurry lot 1 was repeated five (5) times for each setpoint. The same exercise was performed for slurry lot 2. The data in this phase of defining and profiling incoming solutions is used as base line for teaching the mixing system to achieve chosen slurry dilution setpoints.

In phase two (2) the goal was to achieve the same slurry-UPW dilution five (5) times while changing slurry lots to ensure that the system could see and adjust by using its proprietary closed-loop real-time control. Samples 1-5 in Fig.5 represent slurry lot #1, and samples 6-10 slurry lot #2. Each offline density point represents the average of six (6) measurements.

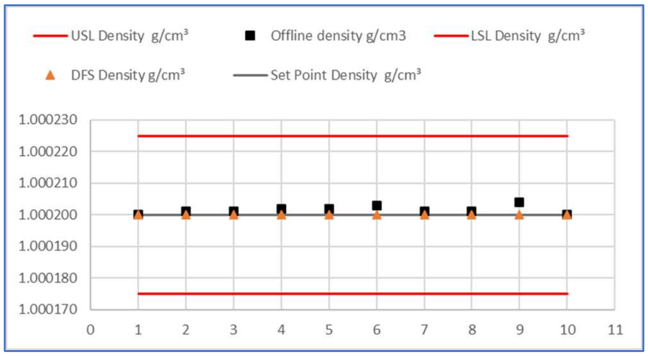


Fig.5 Achieving the target slurry density

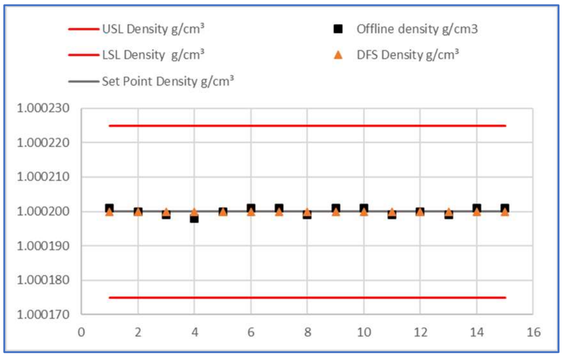
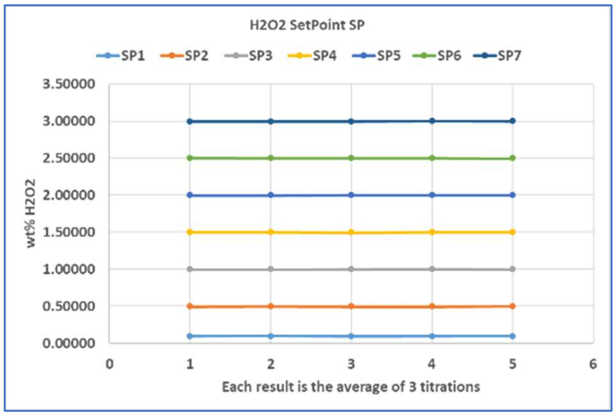


Fig. 6 System automatic mode in full operation

Phase three (3) in the study represents data when the mixing system was in full operation and unhindered automatic mode (Fig. 6). The same exercise was repeated with other slurry-UPW dilutions to prove the robustness of the system autocorrecting back to target level as seen in data points 11-15. Data shows the system meets an extreme accuracy within upper and lower control limits of +/- 0.000025 g/cm3. The precision having the purpose to operate within +/- 0.000015 g/ml (corrected to 20C) on the same solution and have consistent blend to within +/-0.000045 g/ml (corrected to 20C) over time.

Lastly, as many CMP slurry applications have the addition of oxidizing agents, the SDS diluted the slurry to achieve center point density and then H2O2 added to seven (7) concentrations setpoints to demonstrate the accuracy. The blend cycle was repeated five (5) times for each setpoint to demonstrate the same high precision capabilities within a multi-constituent solution. Each sample was verified by analytical titration values which are plotted against the same system targeted concentration values. Against the analytical results, the system's calculated concentration demonstrated an accuracy within 0.004%wt in the range 0.10-3.00%wt H2O2 in slurry under test and a precision within 0.0027%wt of respective repeated setpoint blend.



wt% H2O2	SP1	SP2	SP3
AVE	**0.10008**	**0.50015**	**1.00009**
STDEV	**0.00010**	**0.00005**	**0.00004**
Max	**0.10021**	**0.50023**	**1.00015**
Min	**0.09995**	**0.50011**	**1.00004**
(Range) Span	**0.00026**	**0.00012**	**0.00011**
Slurry BP1 Setpoint =	0.955 Ratio	0.955 Ratio	0.955 Ratio
Wt% H2O2 BP2 Setpoint =	0.100000	0.500000	1.000297
Number of samples	**SP1**	**SP2**	**SP3**
1	0.10005	0.50011	1.00007
2	0.10021	0.50018	1.00004
3	0.09995	0.50012	1.00011
4	0.10006	0.50011	1.00015
5	0.10014	0.50023	1.00009

Fig.7 System achieving accurate and precise H2O2 addition within a concentration setpoint range

CONCLUSION

Process characterization and metrology selection used within the method of process validation is an essential step in the achievement of real-time process control and monitoring of CMP, post CMP clean and other blending and distribution applications. Impacts such as those caused by changes or shifts in incoming slurries or chemicals batch-to-batch can be mitigated by using the DFS platforms, which allow users to develop optimal formulations and control parameters around any part of the chemical formulation. This gives the freedom to optimize onsite, in real-time, and maintain the proprietary process secrets as internal intellectual property IP. Offering a benefit of in-house development, while enabling a user chemical source independence or incoming supplier coordination for product development joint efforts. The DFS system gives its user clients the keys to real-time HVM metrology driven process control and monitoring.

References

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[2] Vazquez Bengochea, L.; Sampurno, Y.; Kavaljer, M.; Johnston, R.; Philipossian, A. Characterization of CMP Slurries Using Densitometry and Refractive Index Measurements. *Micromachines* **2018**, *9*, 542. https://doi.org/10.3390/mi9110542 (2022-08-31)

[3] KxS Technologies inline refractive index monitor, https://kxstechnologies.com/en/dcm10/ (2022-08-31)

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