In-line Refractive Index Monitoring for CMP Slurry Fault Detection

Carlo Dominic Aparece
Facilities
GlobalFoundries Fab 8
Malta, New York, USA
carlo.aparece@globalfoundries.com

Robert Johnston
Yarbrough Solutions Worldwide
Rockwall, Texas, USA
Rjohnston@yswsemi.com

Marcus Kavaljer
Vaisala Oyj
Vantaa, Finland
marcus.kavaljer@vaisala.com

ABSTRACT

Inline refractive index measurements have established themselves as the technique of choice for detecting faults in the CMP slurry blending and dispense systems of leading fabs. Refractive index, a continuous, non-sampling measurement, helps fabs identify slurry composition changes quickly. Once calibrated to a specific slurry's temperature/refractive index characteristics, refractive index measurements can determine the concentration of hydrogen peroxide in slurry with a precision to within ±0.02% by weight, for both copper and tungsten slurries. In long-term studies at this leading edge fab, measurements for low node technology CMP processes detected slurry compositions reliably for three years, with no instrument maintenance beyond routine flushing of the slurry blender tank. In data extracted over a 40-day period, repeatable measurements within the process specification were achieved for both 3% hydrogen peroxide in tungsten slurry and 1% peroxide in copper slurry. Measurements on slurries with 0.1% peroxide concentration were repeatable, but with measurement noise potentially exceeding the process limits. Regardless of the slurry concentration level, refractive index measurements can both monitor intentional slurry dilutions and detect unexpected slurry composition changes in the process. Unique with refractive index is the ability to monitor both the raw slurry and DI water dilution as well as the subsequent peroxide addition.

I. INTRODUCTION

As delivered from the supplier, CMP slurries, like most fab chemicals, usually meet very stringent specifications for composition, particle size uniformity, purity, and so forth. Still, as with other fab chemicals, arrival at the fab's loading dock is only the beginning of the story. Between incoming inspection and the process tool lie many opportunities for contamination and inaccurate blending [1].

CMP slurry is a mixture of the supplier's raw slurry formula with hydrogen peroxide and deionized water. Peroxide degrades over time, so it and water are added to the raw slurry at the fab, typically in a dedicated blender/supplier tank system. From there, a feed line transports the blended slurry to the global loop and then to the point of use.

Inaccurate dispensing or incomplete mixing at the blender tank can allow an incorrect slurry composition into the system. Stuck valves, human error, and other defects can send an incorrect mixture into the supply tank or global loop and from there into the process tool. To ensure process quality, fabs need to be able to monitor CMP slurry composition.

II. MONITORING BALANCES COST, SPEED, ACCURACY

The ideal monitoring method would provide fast, accurate measurements, made instantly available through the fab's information network. A low cost per measurement — achieved by minimizing consumables, chemical waste, and equipment costs — would further limit process risk by allowing real-time measurement.

Few technologies can meet all of these requirements. Fabs must constantly balance cost, accuracy, and other factors. For example, titration — the gold standard for measurement accuracy — is slow and expensive. It requires consumable reagents, creates a new waste stream of sampled chemicals and their reaction products, and poses a possible contamination risk due to the probe used to collect samples. Titration is also a discrete measurement. Each sample reflects a specific point in time. The larger the sampling interval, the greater the probability that excursions will occur in between samples.

Conductivity, pH, and IR spectroscopy methods can be useful in some situations, but are not universally applicable across the many compositions encountered in a semiconductor fab. Conductivity measurements require ionic solutions. Neither conductivity nor pH is precise enough for slurry composition monitoring, as several different compositions might produce the same measured value. IR spectroscopy is an optical, non-contaminating method, and can identify individual components in a solution, but it cannot easily determine their relative concentrations and is difficult to calibrate.

III. Refractive Index Gives Accurate, Non-Intrusive Measurements

First introduced in 2007, in situ refractive index measurements have become the industry standard for fast, accurate, inline slurry monitoring. At leading fabs, the incoming raw slurry must meet not only chemical composition and particle size specifications, but also an expected refractive index specification. Refractive index monitors track slurry composition throughout the fab.

In a reflective index of refraction measurement, light from a single wavelength source reflects off the interface between the liquid being measured and an optical window. A CCD camera detector identifies the borderline position at total reflection, which is the transition between light activated and non-activated pixels on the detector. This so-called "critical angle" measurement is independent of light intensity. This critical angle in turn yields the refractive index of the fluid (See Fig. 1.). Because the light does not need to pass through the fluid, this method can be used with opaque fluids and is not affected by bubbles and other flow irregularities. Overlapping refractive index values are rare: even very similar mixtures generally have unique indices.

Refractive index monitoring is continuous, collecting data once per second whenever the light source is on. As such, it provides a more accurate picture of variations in slurry composition than any discrete sampling method can.

Over a two-month validation period at one fab, 26 different batches of a tungsten slurry passed through the blender tank. Refractive index measurements tracked the laboratory titration reference measurement at both the upper and lower portions of the concentration range. Both the refractive index and titration measurements confirmed that all 26 batches fell within the specification (See Fig. 2.).

Refractive index monitors can be placed at multiple locations along the distribution chain, helping to prevent slurry composition failure. They are typically installed on the incoming hydrogen peroxide and raw slurry supplies, at the blending tank, and on the process feed (See Fig. 3.). In one

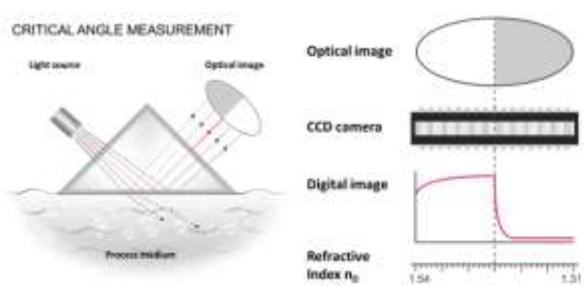

Fig . 1. The optical critical angle of total reflection yield refractive index

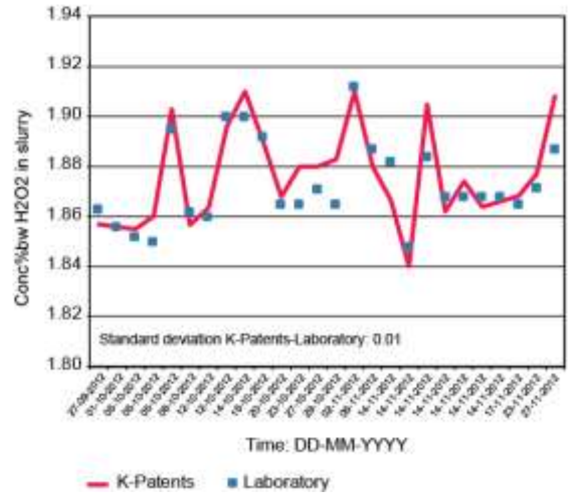

Fig.. 2. Titration vs. Refractive index measurements, two-month comparison.

situation, incorrect slurry formulation was one of the suspected cause for a CMP yield loss issue. As it turned out, though, refractive index sensors exonerated both the slurry and the dispense system. The correct formulation was being delivered to the tool. Instead, valve leakages at the CMP tool level were responsible for the problem. As this example shows, refractive index measurements can both monitor intentional slurry dilution and detect unintended dilutions (See Fig. 4.).

Successfully implementing this measurement in a fab setting requires attention to a number of subtle details. For example, the optical window must be able to tolerate the temperatures and chemical compositions of interest, and must itself have a compatible refractive index. A standard range sapphire prism allows measurements over a refractive index (n_d) range from 1.32 to 1.53, while a low range sapphire prism covers indices between 1.26 and 1.47 to accommodate 10:1 or 100:1 dilute HF. The instruments discussed in this paper combine optics by K-Patents with calibration systems and hardware integration by Yarbrough Solutions Worldwide.

While the sensor window itself is the only interface in contact with the fluid and can be isolated from pressure and temperature variations, temperature variations do affect the properties of the fluid being measured. Refractive index varies non-linearly with concentration and temperature. Published refractive index values are usually measured at 20°C, while actual in-fab chemical supply temperatures tend to vary seasonally.

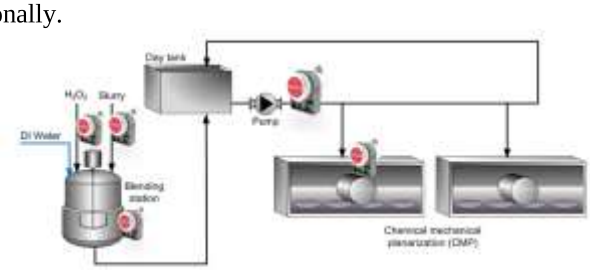

Fig. 3. Refractive index monitoring locations.

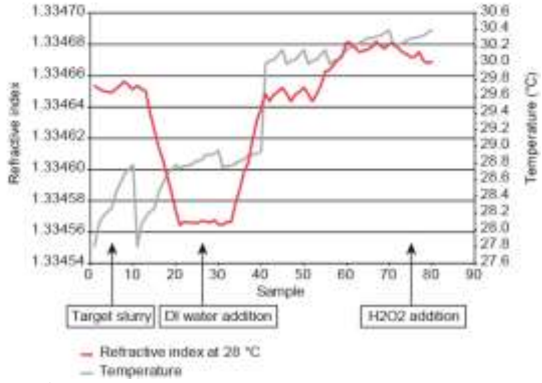

Fig. 4. Refractive index detects slurry dilutions. First, DI water was added to reach the lowest acceptable concentration, and then H2O2 was added until the highest acceptable peroxide concentration for the process was reached.

The sensor must measure and compensate for these temperature variations in order to accurately determine composition of a chemical.

IV. Handling Different Slurry Types and Process Conditions

Each slurry's refractive index will have unique temperature dependence. Each blender tank will have unique hydraulic characteristics. While rough calibration data files are available for commonly used slurries, fine tuning is done in situ at the fab. A slurry of known composition is ramped through a variety of temperature changes and the refractive index measured. This data is then used to calibrate the refractive index for that slurry. (See Fig. 5.) Once the dependence of the slurry's refractive index on temperature is known, the instrument can correct for thermal fluctuations [2]. Fig. 6. illustrates a gradual ramp to the target concentration of 1% hydrogen peroxide by weight, with the measurement remaining stable as the temperature changes. In the event that a sensor needs to be replaced, calibration files from the existing sensor can be used to initialize the replacement. While converting refractive index to weight percent is straightforward for common chemicals, this conversion may not be possible for proprietary compounds. Instead, the refractive index of a known good solution can be treated as a control limit. Deviations from this value indicate a deviation from the ideal bath composition.

Slurries for tungsten and copper removal will have different absolute refractive index values, but generally see similar trends as the water and peroxide concentrations change. Similarly, the particle distribution of raw slurry will affect its refractive index, but adding water and peroxide will change the value in the same way. In one example, Drum 1 of a slurry had a refractive index of 1.3350, while the refractive index of Drum 2 was 1.3352. In both cases, the target concentration of hydrogen peroxide in deionized water and slurry was 2.00%. And in both cases, the same change in refractive index was measured, even though the absolute refractive index values differed (See Fig. 7.).

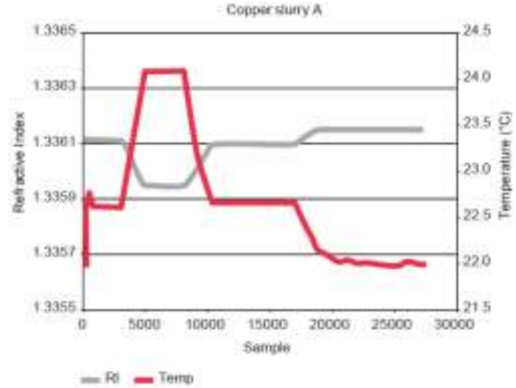

Fig. 5. Temperature dependence of refractive index.

Slurries for higher technology nodes tend to be more likely to accumulate on sensor windows, piping, and other surfaces. This problem is reduced for lower technology node slurries, which tend to depend more on chemical and less on mechanical action for material removal [3].

V. Measurement Extensibility and Stability

As more advanced process nodes require tighter control over CMP removal rate, the concentrations of some slurry components are going down. The refractive index measurement gives a precision and smallest detectable change of ±0.02% by weight of peroxide in slurry, while the process specifies the acceptable concentration window, typically 5-10% relative of the target peroxide concentration. This means that, for a target peroxide concentration of 1% ± 0.05% by weight, the instrument gives a reliable measurement over the full process window. If the target concentration is 0.07% ± 0.005% by weight, however, signal noise may give results that lie outside the process window (See Fig. 8.). It would therefore be advisable to check anomalous results by titration before taking corrective action. Regardless of the measurement precision limitations at very low peroxide levels, the instrument will still instantly detect unexpected slurry composition changes in the process as discussed in Fig. 4.

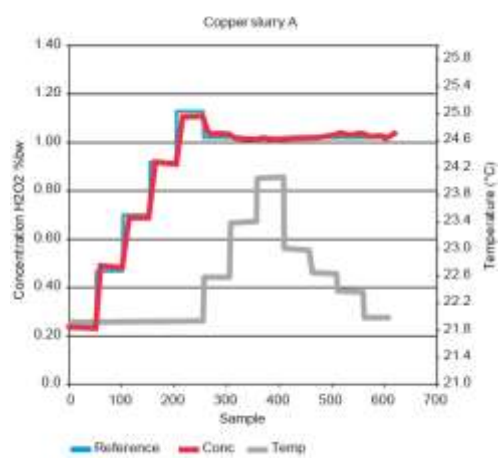

Fig. 6. Stability of temperature-corrected refractive index measurement as peroxide concentration ramps.

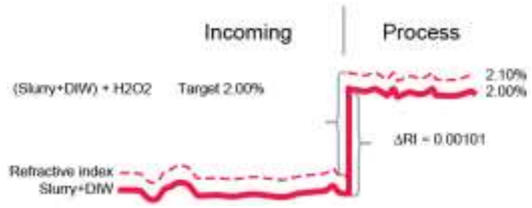

Fig. 7. Change of refractive index with water and hydrogen peroxide conditions is constant, even as raw slurry composition varies.

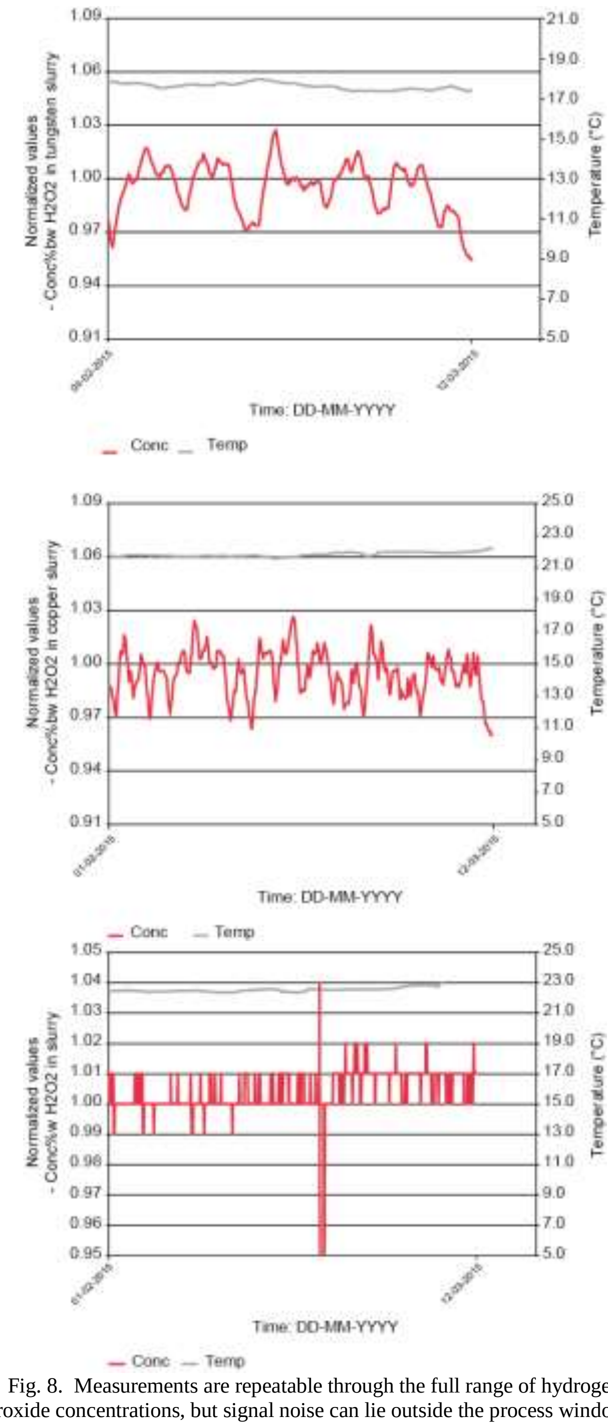

Fig. 8. Measurements are repeatable through the full range of hydrogen peroxide concentrations, but signal noise can lie outside the process window for very low concentrations.

Measurement stability is an important consideration for any fab instrument, particularly one used for continuous monitoring. The instrument discussed in this paper is unique in that it provides a drift-free measurement signal, due to hardcoded instrument parameters. At this leading edge fab only false positive alarms have been encountered; slurry scaling of the sensor window would give a false negative alarm.

At this leading edge fab, data for a copper slurry with a target peroxide concentration of 1% by weight was extracted over 40 days through a number of tanks, remaining within process limits. Measurements of an 0.1% concentration slurry were repeatable, but measurement noise fell outside process limits. Measurements of slurries for lower technology node CMP processes remained stable for three years, with no instrument maintenance beyond routine flushing of the slurry blender tank.

VI. Summary and Conclusion

Though refractive index measurements can be applied to a wide variety of chemicals of interest to semiconductor fabs, they are particularly well-suited to CMP applications because of their tolerance for opaque mixtures and their ability to give useful information regardless of inconsistencies in the raw slurry material. As these results show, refractive index measurements are stable enough for long-term, ongoing monitoring of fab slurry dispense systems. Though they do face challenges as process tolerances shrink, refractive index measurements can continue to support even the most advanced process chemistries.

References

[1] Thomas Moseman, “In-situ Chemical Monitoring in Semiconductor Fabrication Chemical Supplies”, 2006

[2] Marcus Kavaljer, Marja Kivenheimo, “Best Practice Guide on K-Patents Semicon Refractometer PR-33-S Field Adjustment for CMP Slurries”, 2013

[3] V.Balan, C. Euvard, A. Seignard, Clement Chaffard, Emmanuel Gourvest, Sebastien Gaillard, M. Rivoire, *Tungsten CMP Process Investigation for FDSOI 14nm and beyond*, International Conference on Planarization/CMP Technology, 2013, p.17-21