CMP Slurry and Hydrogen Peroxide Concentration Monitoring in Using Multiple Online Index of Refraction Sensors

INTRODUCTION

The chemical mechanical planarization (CMP) process usually includes mixing a fixed ratio and concentration of CMP slurry (including an abrasive), hydrogen peroxide (a strong oxidizing agent) and deionized water in a slurry blending station. Constantly monitoring the concentration of hydrogen peroxide is necessary for ensuring the repeatability and efficiency of the CMP and because it degrades over time. Checking that the CMP slurry is always within specification and adjusting the mixture as necessary limits product loss.

Tools like the Entegris GV148 and SemiChem are used to continuously monitor H_2O_2 concentration, allowing for real-time adjustments to the slurry blend and ensuring that the process stays within the specified parameters. This helps prevent process drift and ensures optimal CMP performance.

GV148 PRINCIPLE OF OPERATION

The Entegris InVue GV148 liquid concentration monitor operates using Index of refraction (IoR) technology. Light from an LED is directed towards an optical window in contact with the liquid. The light reflects off the liquid, with the angle of reflection determined by the liquid's index of refraction. The reflected light is then directed to a photodiode array (PDA), where the intensity of the reflected light is measured. Small changes in light intensity, which are related to changes in the liquid's refractive index, are measured by a custom Entegris algorithm to determine concentration.

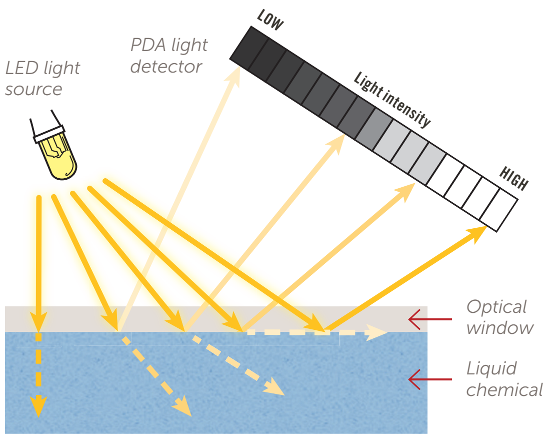

Figure 1. GV148 IoR concentration sensor.

EXPERIMENTAL

A series of experiments were performed on an Air Water Mechatronics slurry delivery systems (SDS) shown in Figure 2. The purpose of these tests was to determine if IoR concentration monitors can effectively measure slurry/ H_2O_2 concentration and to understand how fluctuations in Temp/Press/Flow impacts IoR measurements. The tests compared three commercially available IoR monitors; sensor A, the Entegris GV148, sensor B, and sensor C.

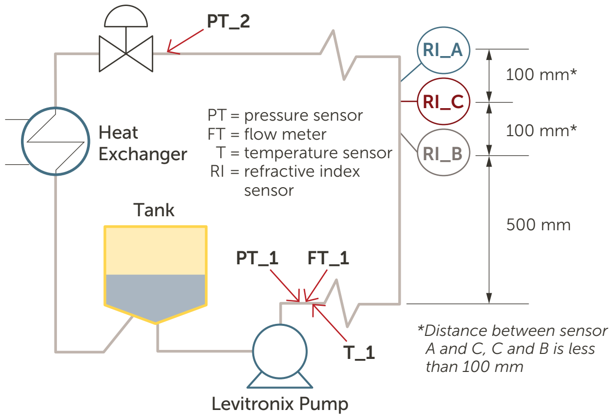

Figure 2. Test flow diagram.

The circulation conditions are shown in Figure 3.

Recirculation condition	Basic condition	Flow rate/pressure change test
Fluid volume (kg)		30
Fluid temperature (°C)	25	20 → 30
Flow rate (m/s)	0.5 / 0.7	0.5-0.7
Pressure (MPaG)	0.15	0.05~0.17

Figure 3. Test conditions.

RESULTS

Test 1: Diliuted H_2O_2

Hyrdrogen peroxide concentration was altered by adding H_2O_2 to DI water three times, starting at zero weight percent. Hyrodrogen peroxide concentration vs. time is plotted in Figure 4.

Figure 4. H_2O_2 concentration vs. time. Red = A, Entegris GV148, blue = sensor B, green = sensor C. X = control value by titration.

Test 2: Diluted Silica Slurry

Concentration of diluted silica CMP slurry was altered by adding slurry to diluted slurry. The concentration was increased from 1.85 to 2.25 % and then decreased back to 1.85% by adding DI water to diluted slurry. Results for all three sensors are shown in figures 5 and 6. Figure 7 is an expansion of the first 15 minutes in Figure 5 to show GV148 data accuracy and stability.

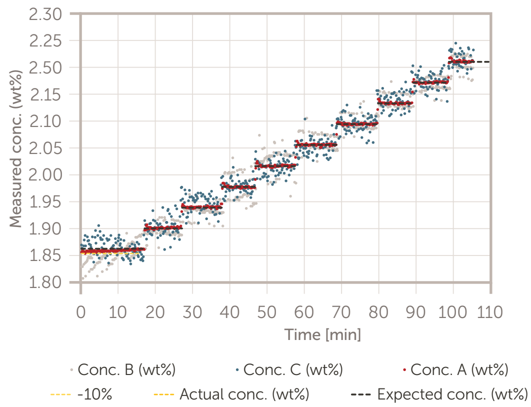

Figure 5. Increasing slurry conc. vs. time. Red = A, Entegris GV148, light gray = sensor B, blue-green = sensor C.

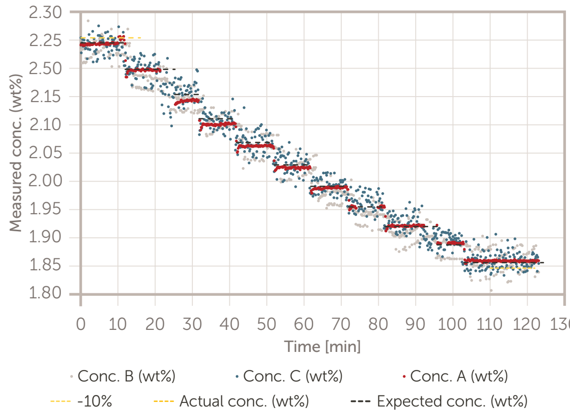

Figure 6. Decreasing slurry conc. vs. time. Red = A, Entegris GV148, light gray = sensor B, blue-green = sensor C.

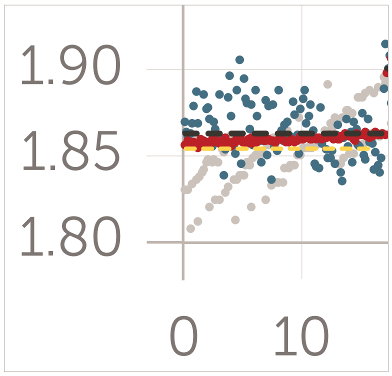

Figure 7. Entegris GV148 data in red.

Note the stability of the Entegris GV148 data compared to sensors B and C. The GV148 results are also closer to the actual/expected concentration values.

TEST RESULT SUMMARY

The three sensors were evaluated for a range of performance criteria and the Entegris GV148 was determined to be the superior product for the following reasons:

- Measurement repeatability and stability, clear IoR difference along with H_2O_2, Silica and Ceria slurry concentration changing
- Easy to use: Can create a calibration table (IoR vs. Conc.) in field, smaller body and less components, can communicate with PLC

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Corporate Headquarters
129 Concord Road
Billerica, MA 01821
USA

Customer Service
Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083