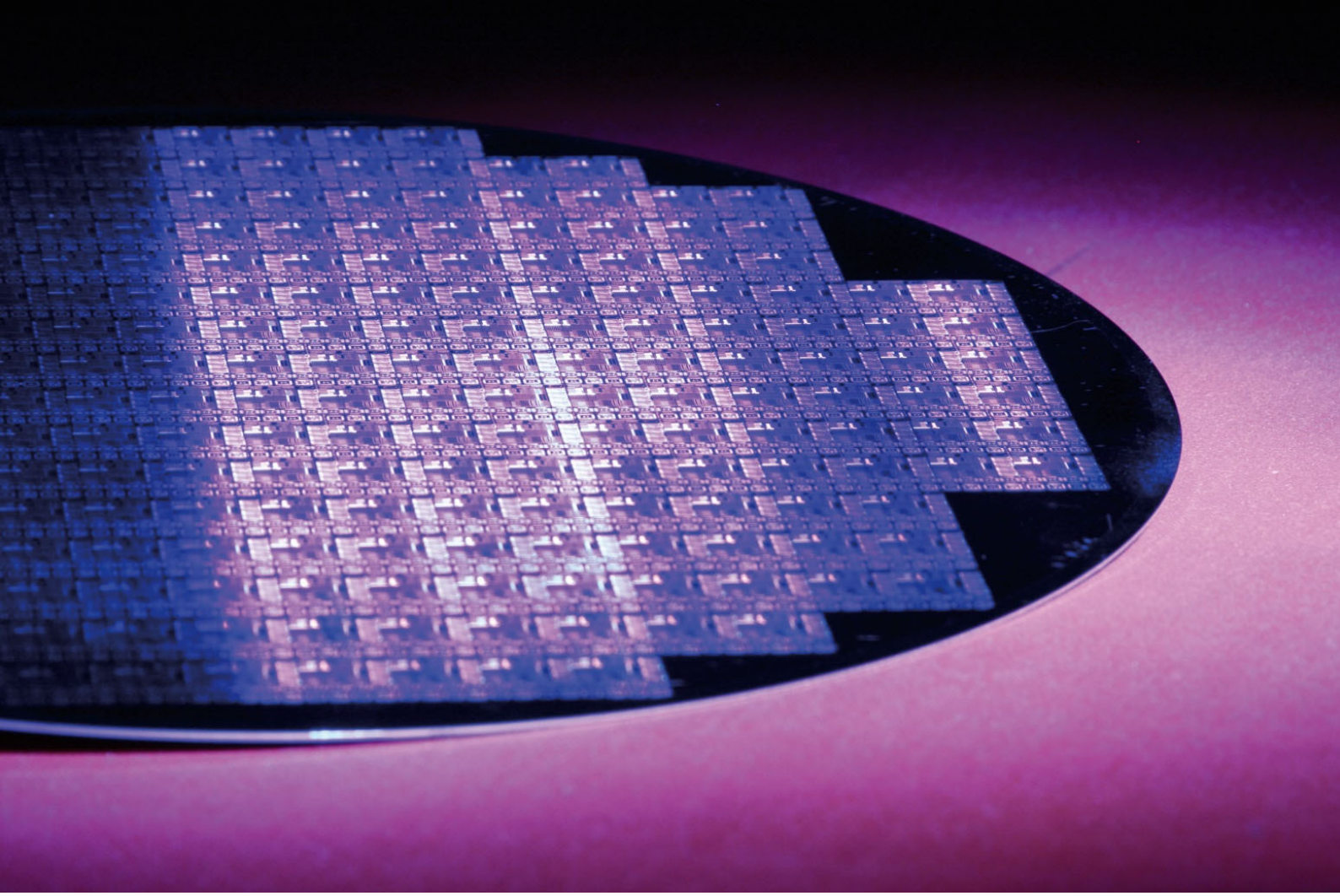

PROCESS APPLICATION NOTE 1055

Monitoring quality parameters in standard cleaning baths

Measure ammonium hydroxide, hydrogen peroxide, and hydrochloric acid simultaneously with inline analysis

Silicon semiconductor devices are manufactured on highly polished wafers. Scratches and other imperfections on the wafer could affect the performance of the final product. Therefore, surface preparation is a key step to obtain clean, mirror polished, undamaged silicon surfaces.

Chemical cleaning is a proven method used here to remove contaminants from the wafer surface. The most common process, «RCA clean», cleans wafers via two consecutive standard solutions. The standard cleaning 1 «SC1» bath (or ammonia peroxide mixture «APM») is composed of NH_4OH and H_2O_2. The standard cleaning 2 «SC2» bath is made of HCl and H_2O_2. The key factors for efficient wafer cleaning are bath residence time and optimum chemical concentration in the cleaning baths. Rapid inline monitoring of the major SC1/SC2 bath constituents guarantees increased wafer yields while decreasing defect density.

Metrohm Process Analytics

INTRODUCTION

Efficient silicon wafer cleaning demands optimal process control to ensure an increase in product throughput without additional defects while increasing production rates and profitability at the same time.

The SC1 bath removes particles, films, and organic residue from the wafer, and forms a thin oxide layer on the surface. However, transition metal hydroxides may also remain on the wafer surface. That is when the SC2 bath becomes essential in the post chemical mechanical planarization «CMP» cleaning sequence. SC2 baths are acidic, helping to remove surface alkali and transition metals. This cleaning process leaves a thin passivation layer on the surface of the wafer to avoid future contamination.

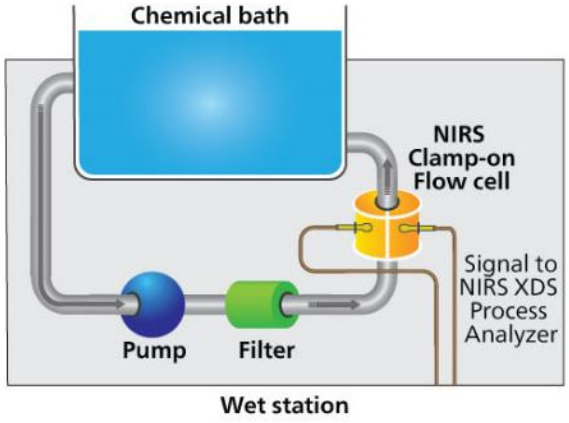

Figure 1. Inline near-infrared spectroscopy (NIRS) system configuration for cleaning bath analysis.

The smaller the semiconductor device, the more difficult it is to remove the small particles from the surface of the silicon wafer. Therefore, semiconductor manufacturers perform the standard cleaning steps in a wet bench inside a cleanroom to control the environment and avoid further contamination. This setup leaves very limited space to install an analysis system. Additionally, any chemical handling should be avoided inside the cleanroom area to increase personal and production safety, and to avoid contamination of the wafers.

A safer, more efficient, and faster way to **monitor multiple parameters simultaneously in standard cleaning baths** is via inline analysis with reagent-free near-infrared spectroscopy (NIRS). The NIRS XDS Process Analyzer by Metrohm Process Analytics enables comparison of «real-time» spectral data from the process to a reference method (e.g., titration, HPLC, IC) to create a simple, yet indispensable calibration model for the standard clean baths.

«Real-time» analysis of the baths keeps chemical concentrations within tight specifications, reducing their consumption by about 25%, saving tens of thousands of dollars of each bath component every year.

APPLICATION

Wavelength range used: **800–1300 nm**. Reference method: ion chromatography.

When cleanroom space is limited, the NIRS XDS Process Analyzer can be mounted outside the cleanroom in the subfab core facility or simply beneath the wet bench embedded in the processing unit/tool itself. The distance between the instrument and the sample points (up to 9 possible with the multiplexer option) can be hundreds of meters apart and simply interfaced to the instrument with low-dispersion optical fibers. All process baths have a circulation loop made of PFA tubing. The flow cell, designed and customized by Metrohm Process Analytics, can be clamped on to these tubes for easy installation, with no need to modify the existing setup. Just clamp the flow cell on, and start measuring.

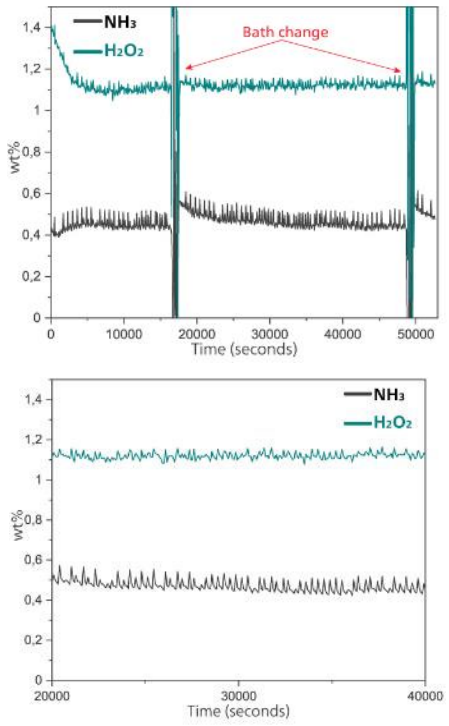

Figure 2. Trend chart of NH_3 and H_2O_2 concentrations in an SC1 bath. Note the spiking of the baths to maintain concentrations.

BENEFITS FOR NIR SPECTROSCOPY IN PROCESS

- **Increased product throughput,** reproducibility, production rates, and profitability (less wafer discarding).
- **Efficient wafer** cleaning by constantly monitoring the standard baths.
- **More savings per measurement point**, making results more cost-efficient.
- **Safer production** due to «real-time» monitoring and no exposure of operators to chemical reagents.

Figure 3. NIRS XDS Process Analyzer.

TYPICAL RANGE

Table 1. Slurry measurement parameters

Parameters		Temperature [°C]	Range [wt %]
SC1	NH_4OH	65 ± 3°C	0–1
	H_2O_2	65 ± 3°C	0–2
SC2	HCl	35 ± 3°C	0–1.5
	H_2O_2	35 ± 3°C	0–5
SC2	HCl	RT to 70°C	1–5
	H_2O_2	RT to 70°C	1–10

REMARKS

An appropriate range of samples covering the process is needed to build a calibration model. These samples will be analyzed via NIRS and via a reference method. The precision of the NIRS data is **directly correlated** to the precision of the reference method.

Other applications are available for the semiconductor industry like: copper, sulfuric acid, and chloride in acid copper baths, acidity in mixed acid etchants, hydrofluoric acid etch, ammonium hydroxide, and hydrochloric acid in standard clean baths.

CONCLUSION

NIRS analysis enables the comparison of «real-time» spectral data from the process to a primary method (e.g., titration, Karl Fischer titration, HPLC, IC) to create a simple, yet indispensable model for your process requirements. Gain more control over your production with a Metrohm Process Analytics NIRS XDS system, **capable of monitoring up to 9 process points** with the multiplexer option.

RELATED APPLICATION NOTES:

- **AN-PAN-1012** Electroless Nickel Plating; Semiconductor, PCB industry. Analysis of Nickel ion & Hypophosphite content
- **AN-PAN-1028** Monitoring Tetramethylammonium Hydroxide (TMAH) in Developer
- **AN-PAN-1054** Online monitoring of hydrogen peroxide during CMP process

Analytes:	**Acids – inorganic; Nitrogen – ammonia, ammonium; Peroxides**
Matrix:	**Etching baths/pickling baths; Other**
Method:	**Process Analysis; Spectroscopy (NIRS/Raman)**
Industry:	**Electronics & semiconductors**

Process Application Note AN-PAN-1055, Version 1 – 2020-08

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