

Application Note AN-PAN-1054

Online monitoring of hydrogen peroxide during the CMP process

Semiconductors are the fundamental components of modern electronic goods. With the advent of the digital age and now the current «Internet of Things», smaller, faster, and more powerful processors are in constant demand for many goods and services.

To produce silicon semiconductor devices, the grown silicon ingot material has to be cut, shaped, polished, and cleaned for further processing. Polishing is a key step in obtaining high quality silicon wafers, since it achieves surface planarity for future lithographic steps.

Chemical Mechanical Planarization «CMP» is one of the main technologies used to smooth or polish the silicon wafer surface. Typically, this process consists of

mixing deionized water, CMP slurry (a colloidal silicon or alumina liquid dispersion), and hydrogen peroxide (a strong oxidizing agent) in a slurry blending station at a fixed concentration and ratio. The blended slurry mixture is pumped to the day tank for storage or to multiple polishers as part of a chemical delivery unit «CDU».

Because hydrogen peroxide degrades over time, it is necessary to constantly monitor its concentration online to ensure the CMP process is efficient and repeatable. In this manner, product loss is limited by checking that the CMP slurry is always in specification, and adjusting the mixture if necessary.

INTRODUCTION

In 2019, the Semiconductor Industry Association (SIA) claimed that the worldwide semiconductor sales had been increasing in the last couple of years, with an annual growth rate of 6.81% per year.

Pure silicon ingots, from which the wafers are cut, are incredibly expensive to create. Therefore, any unnecessary waste must be kept to a minimum during the subsequent processing steps.

In the CMP polisher, the slurry mixture comes into direct contact with a single silicon wafer, oxidizing the hard silicon surface to a softer silicon oxide layer, and increasing the **polishing efficiency**. This is kept in a precise balance with the mechanical polishing pad to

achieve optimum polishing rates for increased wafer yields.

Hydrogen peroxide is the most widely used chemical oxidizer for the CMP process, and is added to the majority of semiconductor CMP slurries. However, because of its degradation properties over time, hydrogen peroxide concentrations must be measured continuously and throughout the slurry global distribution loop to assure *in spec* slurry purity before use. This demands quick online analysis with fast response times as part of chemical replenishment and process control.

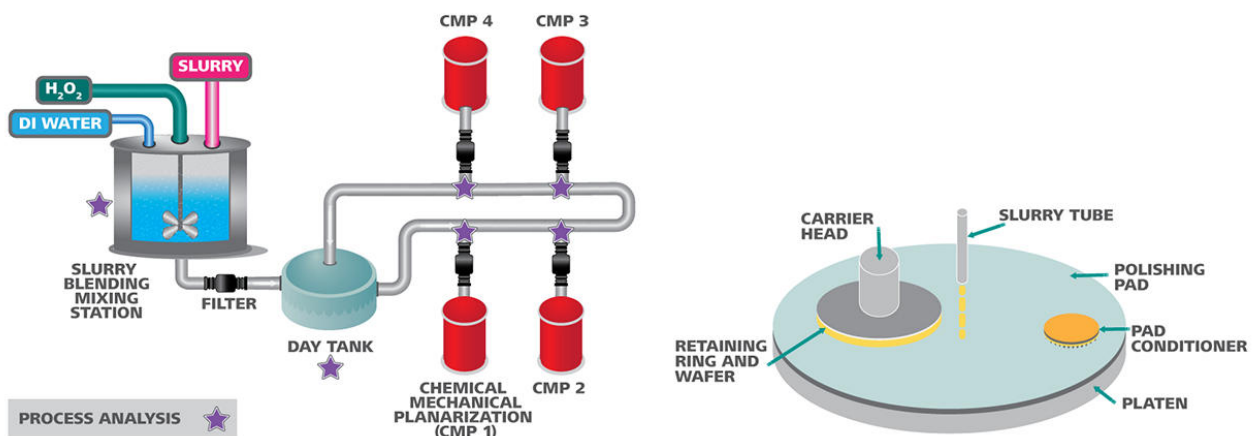


Figure 1. (Left) A typical Chemical Mechanical Planarization (CMP) process. (Right) Top view of a CMP polisher.

APPLICATION

Online monitoring of hydrogen peroxide, pH, conductivity, and temperature is possible with the **2060 Process Analyzer** from Metrohm Process Analytics. The hydrogen peroxide concentration is measured titrimetrically with cerium(IV) using a Pt-ring electrode to determine the endpoint with dynamic endpoint titration (DET). The analysis frequency is typically less than 5 minutes, ensuring timely control of the slurry mixture.

Other combinations of measurements, as well as measurement points taken from a single process stream or even multiple streams, can be realized across the Metrohm Process Analytics product portfolio. All platforms guarantee fast, accurate results continuously available for true process control.



Figure 2. 2060 Process Analyzer from Metrohm for online monitoring of hydrogen peroxide during the CMP process.

Table 1. Slurry measurement parameters

Parameters	Range
Hydrogen peroxide	0–5%
pH	2–12
Conductivity	10–10,000 $\mu\text{S}/\text{cm}$
Temperature	20–65 °C

BENEFITS FOR ONLINE TITRATION IN THE CMP PROCESS

- Enhanced wafer yields with qualified CMP slurry compositions
- Increased product throughput with less wafer defects
- Greater mixing integrity and purity in the blending station
- Enhanced control of chemical reaction rates and polishing rates of the CMP polishing process



CONCLUSION

The Metrohm Process Analytics **2060 Process Analyzer** can measure not only the concentration of hydrogen peroxide in CMP slurry, but also include pH,

conductivity and temperature measurements to give an overall health status of the produced CMP slurry mixture without delay.

REMARKS

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.

FURTHER READING

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.](#)

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CONFIGURATION



2060 Process Analyzer

The 2060 Process Analyzer is an online wet chemistry analyzer that is suitable for countless applications. This process analyzer offers a new modularity concept consisting of a central platform, which is called a «basic cabinet».

The basic cabinet consists of two parts. The upper part contains a touch screen and an industrial PC. The lower part contains the flexible wet part where the hardware for the actual analysis is housed. If the basic wet part capacity is not sufficient enough to solve an analytical challenge, then the basic cabinet can be expanded to up to four additional wet part cabinets to ensure enough space to solve even the most challenging applications. The additional cabinets can be configured in such a way that each wet part cabinet can be combined with a reagent cabinet with integrated (non-contact) level detection to increase analyzer uptime.

The 2060 process analyzer offers different wet chem techniques: titration, Karl Fischer titration, photometry, direct measurement and standard additions methods.

To meet all project requirements (or to meet all your needs) sample preconditioning systems can be provided to guarantee a robust analytical solution. We can provide any sample preconditioning system, such as cooling or heating, pressure reduction and degassing, filtration, and many more.